Hardness Test of Ceramic materials

Hardness is a measure of a materials resistance to penetration by a hard indenter of defined geometry and loaded in prescribed manner, it is one of the most frequently measured properties of a ceramic

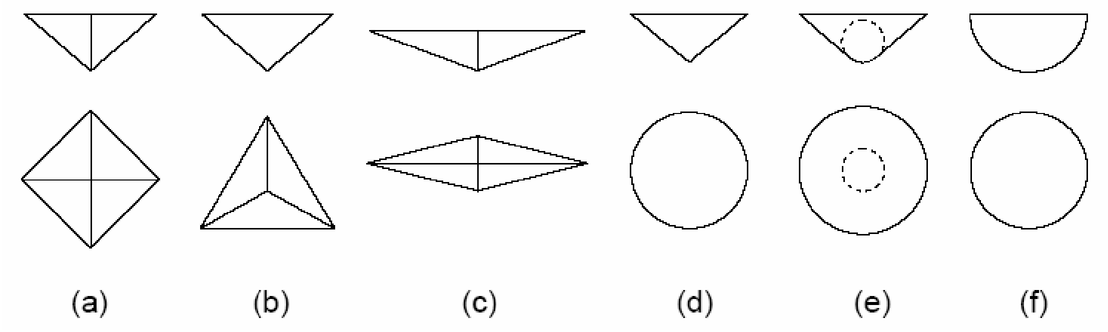
Hardness value helps to characterize resistance to abrasives or wear, resistance to plastic deformation, modulus of elasticity, yield strength, ductility, and fracture toughness. It is necessary for cutting tools, wear and abrasion-resistant parts, prosthetic hip joint balls and sockets, optical lens glasses, ballistic armor, molds and dies, valves, and seals.

The hardness of a ceramic is defined by its chemical composition, including porosity, grain size, and grain-boundary phases. There are many different hardness tests and each gives a different number.

The Methods and Process of Hardness Testing

There are multiple measurements of hardness, including scratch hardness, indentation hardness, and rebound hardness. Each type of measurement is based on an individual measurement scale, however, conversion between scales is possible for practical purposes.

Indentation tests are a common method of testing the hardness of a ceramic material. Indentation is a straight forward test of penetrating a given material with an indenter under a pre-defined indentation load, then measuring the resulting indentation. Indenters come in a variety of different shapes and sizes as in figure 15 , and the load can be set for nano, micro, or macro indentation ranges, so as to specify the range of mechanical properties that will be tested. The general concept behind the measurement is very similar: the harder the testing material is, the smaller the indentation will be



Vickers Berkovich Knoop Conical Rockwell Spherical

Figure 15 a variety of different indenter's shapes and sizes

Static indentation hardness tests such as Brinell, Rockwell, Vickers, Knoop and Berkovich are frequently used methods for determining hardness. The basic concept utilized in all of these tests is that a fixed force is applied to an indenter in order to determine the resistance of the material to penetration. If the material is hard, a relatively small or shallow indentation will result, whereas if the material is soft, a fairly large or deep indentation will result.

These tests are often classified in one of two ways: either by the extent of the test force applied or the measurement method used. A ‘‘macro’’ test refers to a test where a load >1 kg is applied; similarly ‘‘micro’’ refers to a test where a load of ≤ 1 kg of force is applied. Additionally, some instruments are capable of conducting tests with loads as light as 0.01 g and are commonly referred to as ultralight or nanoindentation testers. Rockwell and Brinell testers fall into the macro category, whereas Knoop testers are used for microindentation tests. Vickers testers are employed for both macro and microindentation tests. In the case of more brittle materials, such as ceramics, using too heavy a load can result in cracking of the specimen, evident at the corners of the indents, as well as chipping of the material around the indentation perimeter

Ceramic hardness is usually tested using either the Vickers or Knoop method, most often using diamond indenters, For research purposes, Vickers, Knoop, and Berkovich (triangular pyramid) indenters are customary; Rockwell and Brinell indenters are rarely suitable for ceramics research. Other methods, such as the Vickers hardness test and Rockwell scales, can also be used to determine hardness, but are known to cause more damage to the testing material than the Knoop method.

1- Knoop Test

In the Knoop test, a rhombic-based sharp diamond indenter like a long pyramid is used on a polished surface under a predetermined load for a predetermined length of time (an example of both is a 500 g load held for 10 seconds). The indention is then measured under a microscope and the Knoop hardness (HK) is calculated. The formula used to determine the Knoop hardness of a material is as follows:

HK = load (kgf)/impression area (mm^2)

$= P/C_pL^2$

where P equals the load, C_p equals the correction factor related to the shape of the indenter (generally 0.070279), and L equals the long diagonal of the indentation. The test descript in detail in ASTM standard E 384, Microhardness of Materials

The majority of oxide ceramics tested have a Knoop hardness of 1000 to 1500 kgf/mm^2. Dense ceramics are often measured for hardness by the Knoop hardness test, a method of microindentation optimized for brittle materials or thin coatings such as ceramic. The Knoop hardness test is most practical for the purpose of ceramic coating tests, as only a small indentation is required to evaluate and measure a material's hardness. The Knoop method has a few disadvantages; particularly worth noting is the optical microscope resolution limits are potentially serious for Knoop indentations due to the slender tapered tip. The amount of time required to apply the indenter may also be considered a drawback of this test.

A major advantage of the Knoop indentation over Vickers for ceramics is that larger indentation loads may be applied without cracking. Experience with a wide range of ceramics has proven that the Knoop indentations are far less likely to crack, Knoop indentations are about 2.8 times longer and are shallower than Vickers indentations made at the same load. With longer indentations, the accuracy and the precision of the length measurements are superior. Even if the sides of the indentation are displaced or cracked, a credible diagonal length reading and hardness estimate may be made.

2- Vickers test

The Vickers hardness test method uses a square-based diamond pyramid indenter to penetrate the testing material. In the Vickers test, the load is applied smoothly, without impact, and held in place for 10 or 15 seconds. The physical quality of the indenter and the accuracy of the applied load must be controlled to get the correct results. After the load is removed, the two impression diagonals are measured and then averaged, HV is calculated using the following formula:

$$HV = 1.854*F/D^2$$

where F is the measurement of the applied load in kg and D the average diagonal in mm. The square pyramidal indenter creates smaller, deeper impressions that are more likely to crack than Knoop indentations. The original Vickers testers were developed for test loads of 1 to 120 kgf, ASTM standard E 384, Microhardness of Materials, covers Vickers hardness;

The Vickers four-sided indenter is known to crack brittle materials, in which case the Knoop method is likely preferred. It may be necessary to limit the applied force in Vickers teste to a level where a minimum of cracking occurs. It is also worth noting that a direct comparison between Vickers and Knoop hardness numbers is not possible due to the differences in indentation method.

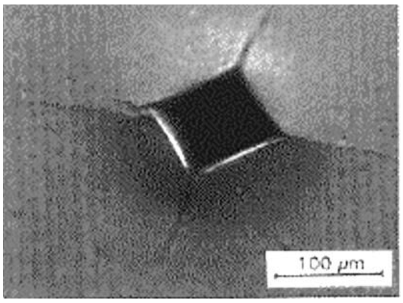


Fig. 16

3- Berkovich test

The Berkovich tip is a nearly flat, three-sided pyramid with a sharp point used to make indentations to test the hardness of materials greater than 100 nanometers thick. It is generally used in small-scale indentation studies and has the advantage that the edges of the pyramid are more easily constructed to meet at a single point. Similar to the Knoop method, nanoindentation requires the placement of an indenter tip, such as the Berkovich indenter in this case, resulting in the measurement of the indentation created by the added pressure of a defined load. In this scenario, hardness (or H) is equal to the max load (or P_{max}) over A_r (or the residual indentation area).

$H = 1569.7P/d^2$

where P = test force in gf, d = diagonal of indentation in µm, and a triangular pyramid indenter with an angle of 115° is used.

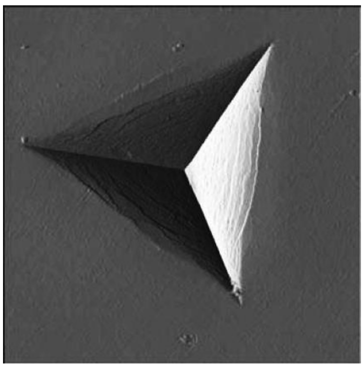


Fig. 17

Important points in hardness testing of advanced technical ceramic

Ceramic tend to show a marked load dependence of hardness number. It is critically important that the test load is appended to each test result, and that no attempt is made to compare results at different test loads in order to make a choice of material or to test to a specification.

At low indentation loads in Vickers and Knoop test, the small size of the indentations means that measurement errors can be large resulted from the load dependence of hardness and from measurement uncertainty due to the small indentation size.

At higher loads, cracking and spalling become problems; in some cases, they make credible measurement impossible. The indentation size effect, in which hardness decreases with increasing indentation load, is found with both Knoop and Vickers hardness. A constant hardness is reached at loads from 5 N to 100 N, depending on the ceramic.

Cracking compounds the difficulty in estimating where a tip ends, and all too often makes reading the indentation size difficult. Furthermore, because hardness is inversely proportional to the square of the diagonal length of the indentation, any error in length measurement is doubled. Therefore, it is crucial that the diagonal length be measured carefully, especially for ceramics in which the indentation size is small and the percentage error may be larger

Many ceramics contain porosity which may be distributed uniformly or unevenly, hardness test will tend to compact the pores in the immediate vicinity of the area of contact of the indenter, giving a lower hardness than for an area which is pore free. Care should be taken that the positioning of the indenter for hardness measurements is random, and not selective, although clearly large obvious pores need to be avoided.

Specimen Preparation

Prior to conducting the test, a specimen will typically undergo a certain level of preparation.

- For all tests the test-piece shall have parallel flat faces so that it does not move during indentation. if necessary, it may be mounted in mounting resin for micro hardness tests.

- The indenter should be perpendicular to specimen at the point of contact, Place the indent in the center of the measuring field, because lens image quality is best in the center.

- The surface quality of ceramic test-pieces may affect the results, for a shallow indentation, a rough surface finish will cause a high level of variation in the readings microindentation tests, such as Vickers and Knoop, rough polishing to a finish of 3 µm or better is recommended.

- Spacing of indents is important because indenting produces plastic deformation and a strain field around the indent, the material surrounding the impression is disturbed and possibly work-hardened. For this reason, a minimum spacing requirement between indentations can be found for each type of hardness test in a corresponding standard. The spacing is specified in terms of indentation diameters, rather than units such as micrometers, to account for the greater amount of cold working that often occurs in soft materials that produce larger indentations. If indentations are too closely spaced, the hardness values can become erratic.

- Time should be held constant from test to test
- Specimen thickness must be at least 2.5 times the Vickers diagonal length. Because the Knoop indent is shallower than the Vickers at the same load, somewhat thinner specimens can be tested.