

## Compression

The strength of ceramic materials measured in compression is much larger than that measured in tension? As a result the tensile strength is generally cited as the critical parameter for purposes of design. However, there are a number of applications including biaxially prestressed engine bearings, armor, and bioceramic components for bone replacement, where the main loading mode is in compression. The successful utilization of ceramic components in these applications requires reliable estimates of compressive strength.

Compressive failure is thought to involve the combination of damage in the form of microcracks and microvoids, this damage is thought to be generated by localized microplasticity arising from twinning and slip.

Failure occurs by structural collapse when the density of the damage zone reaches a critical size. Because this size appears to depend only upon intrinsic material characteristics, the compressive strength is generally thought to be independent of specimen size. For the case of uniaxial

testing, the compressive stress  $\sigma_c$  is given simply by the applied load  $P$  divided by the cross-sectional area  $A$ . However, the high stresses required for failure under compressive loading have created a number of problems in the design of test fixtures and specimen geometries required for successful compression strength measurement. In particular, problems related to improper alignment and load block stress concentrations can lead to the generation of tensile stresses in the specimen sufficient to cause failure. Therefore, the measured compressive strength will underestimate the true value. The possible error sources are:

- (1) load block/specimen size mismatch,
- (2) load block/specimen agreement mismatch,
- (3) surface irregularities, and
- (4) unusual loading.

**Testing Machines.** The most common testing machines are universal testers, which test materials in tension, compression, or bending. Their primary function is to create the stress strain curve. Testing machines are either electromechanical or hydraulic. The principal difference is the method by which the load is applied. Electromechanical machines are based on a variable-speed electric motor; a gear reduction system; and one, two, or four screws that move the crosshead up or down. This motion loads the specimen in tension or compression. Crosshead speeds can be changed by changing the speed of the motor.

Hydraulic testing machines are based on either a single or dual-acting piston that moves the crosshead up or down. However, most static hydraulic testing machines have a single acting piston or ram. In a manually operated machine, the operator adjusts the orifice of a pressure-compensated needle valve to control the rate of loading. In a closed-loop hydraulic servo system, the needle valve is replaced by an electrically operated servo valve for accurate control.

### Factors effect on ceramic strength

#### 1- Processing and Surface Flaws

The flaws in ceramics can be either internal or surface flaws generated during processing or surface flaws introduced later, during machining or service.

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##### Pores

Pores are usually quite deleterious to the strength of ceramics not only because they reduce the cross-sectional area over which the load is applied, but more importantly because they act as stress concentrators.

Typically the strength and porosity have been related by the following empirical relationship:

$$\sigma_p = \sigma^0 e^{-BP}$$

where  $P$ ,  $\sigma_p$ , and  $\sigma^0$  are, respectively, the volume fraction porosity and the strength of the specimen with and without porosity;  $B$  is a constant that depends on the distribution and morphology of the pores.

## 2 Inclusions

Impurities in the starting powders can react with the matrix and form inclusions that can have different mechanical and thermal properties from the original matrix.

Consequently, as a result of the mismatch in the thermal expansion coefficients of the matrix and the inclusions large residual stresses can develop as the part is cooled from the processing temperature.

## 3 Agglomerates and large grains

The rapid densification of regions containing fine particles (agglomerates) can induce stresses within the surrounding compact. Voids and cracks usually tend to form around agglomerates. These voids form as a result of the rapid and large differential shrinkage of the agglomerates during the early stages of sintering. Since these agglomerates form during the fabrication of the green bodies, care must be taken at that stage to avoid them.

## 4 Surface flaws

Surface flaws can be introduced in a ceramic as a result of high-temperature grain boundary grooving, post fabrication machining operations, or accidental damage to the surface during use, among others. During grinding, polishing, or machining, the grinding particles act as indenters that introduce flaws into the surface. These crack scan propagate through a grain along cleavage planes or along the grain boundaries, as shown in Figure 10.

Grain boundary cracks Cleavage cracks within grain

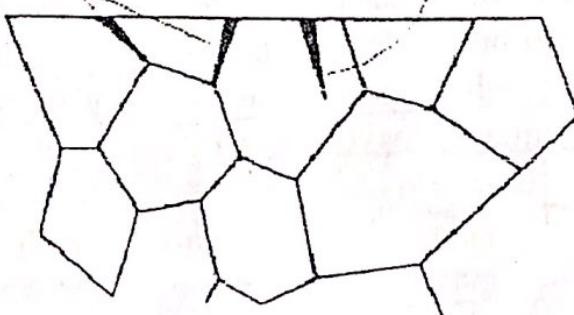


Figure 10 Schematic of cleavage and grain boundary cracks that can form on the surface of ceramics as a result of machining.

## 2- Effect of Grain Size on Strength

Typically, the strength of ceramics shows an inverse relationship to the average grain size  $G$ . A schematic of the dependence is shown in Figure 11, where the fracture strength is plotted versus  $G^{-1/2}$ .

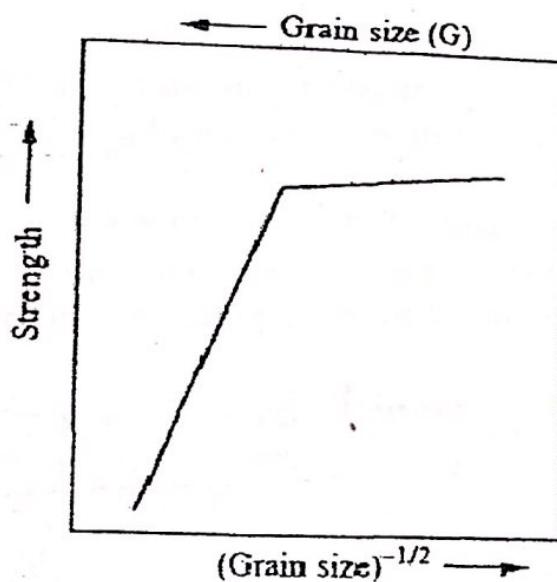


Figure 11 Schematic relationship between grain size and strength for a number of ceramics.

## 3- Effect of Compressive Surface Residual Stresses

The introduction of surface compressive layers can strengthen ceramics and is a well established technique for glasses. The underlying principle is to introduce a state of compressive surface residual stress, the presence of which would inhibit failure from surface flaws since these compressive stresses would have to be overcome before a surface crack could propagate. These compressive stresses have also been shown to enhance thermal shock resistance and contact damage resistance.

## 4- Effect of Temperature on Strength

The effect of temperature on the strength of ceramics depends on many factors, the most important of which is whether the atmosphere in which the testing is being carried out heals or heals or worsens surface flaws in the material. In general, when a ceramic is exposed to a corrosive atmosphere at elevated temperatures, one of two scenarios is possible:

- (1) A protective, usually oxide, layer forms on the surface, which tends to blunt and partially heal preexisting flaws and can result in an increase in the strength.
- (2) The atmosphere attacks the surface, either forming pits on the surface or simply etching the surface away at selective areas; in either case, a drop in strength is observed. [For ceramics containing glassy grain boundary phases, at high enough temperatures the drop in strength is usually related to the softening of these phases.]

## Elastic constants

Some parameters that describe the mechanical behavior of materials are constants, like Young's modulus E. Some, like hardness, are not. Hardness depends on how the material was tested.

These are four constants that are most common

1- E—Young's modulus (also referred to as the elastic modulus) is a material constant defined by Eq. 1 for a linear elastic material under uniaxial tensile or compressive stress. It is therefore the slope of a ( $\sigma$ - $\epsilon$ ) curve where only elastic deformation occurs.

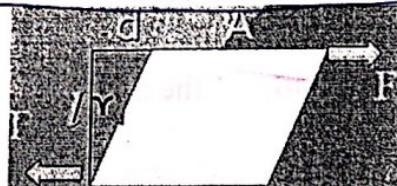
$$\sigma = E \epsilon$$

2. v—Poisson's ratio is the ratio of lateral strain to longitudinal strain for a given material subjected to uniform longitudinal stresses within the proportional limit. The term is found in certain equations associated with strength of materials.

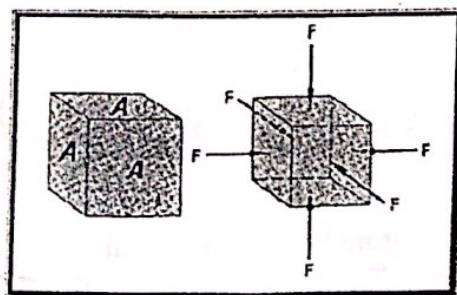
$$v = -\epsilon_T / \epsilon_L$$

3.  $\mu$ —Shear modulus is the ratio of shear stress to shear strain within the proportional limit.

$$\mu = \tau / \gamma$$



4. B—Bulk modulus is the ratio of stress to strain for hydrostatic compression. Sometimes an applied stress F/A results in decrease of volume. In such cases, there is a bulk modulus B of elasticity. The bulk modulus is negative because of decrease in V



$$B = \frac{\text{Volume stress}}{\text{Volume strain}} = \frac{-F/A}{\Delta V/V}$$

$$B = -P(\Delta V/V)$$

Although these constants are related directly to bonding forces between atoms, in real ceramics they are affected by microstructure, e.g., porosity and the presence of second phases. Because strain is dimensionless, elastic moduli have the same dimensions as those of stress: force per unit area ( $N/m^2$ ) or (Pa) and the relationship among them are:

$$E = 2\mu(1+v); \quad E = 3B(1-2v)$$

### Measurement of Young's modulus

Young's modulus are determined from static and dynamic tests. In static measurements such as the classical tensile or compressive test, a uniaxial stress is exerted on the material, and the elastic modulus is calculated from as the slope of the stress-strain curve at the origin. But this is not generally a good way to measure the modulus. For one thing,  $\Delta L$  may be too small to measure with precision. And, for another, if anything else contributes to the strain, like creep or deflection of the testing machine itself, then it will lead to an incorrect value for  $E$  - and these spurious strains can be serious.

A better way of measuring  $E$  is by Dynamic methods, it is nondestructive allowing repeated testing of the same sample. These include bar resonance methods by impulse excitation of vibration then measure the natural frequency of vibration of a round rod of the material, simply supported at its ends (Fig. 8) and heavily loaded by a mass  $M$  at the middle (so that we may neglect the mass of the rod itself). The frequency of oscillation of the rod,  $f$  cycles per second (or hertz), is given by

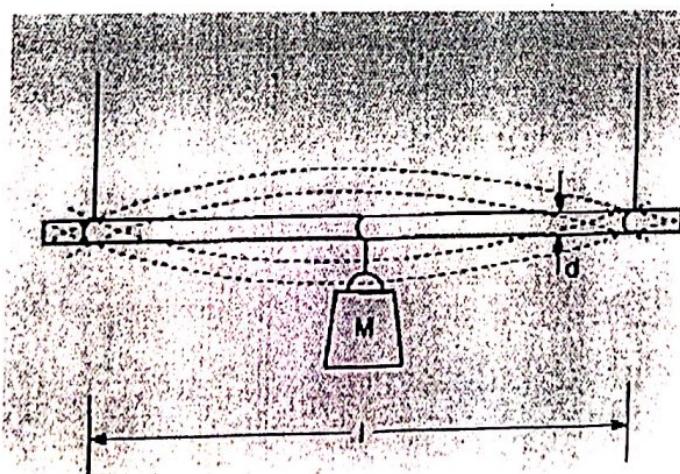


Figure 8: vibrating bar with a central mass,  $M$ .

$$f = \frac{1}{2\pi} \left[ \frac{3\pi E d^4}{4I^3 M} \right]^{\frac{1}{2}}$$

where L is the distance between the supports and d is the diameter of the rod. From this,

$$E = \frac{16\pi M I^2 f^2}{dL}$$

The second dynamic method is the sonic pulse technique, the dynamic Young modulus is determined by measuring the sound velocity in the sample. The basic principle of this method is that the velocity of an ultrasonic wave through a material is related to its density and elastic properties. The velocity of longitudinal waves,  $v_l$ , depends on Young's modulus and the density,  $\rho$ :

$$v_l = \sqrt{\frac{E}{\rho}}$$

$v_l$  is measured by 'striking' one end of a bar of the material (by gluing a piezoelectric crystal there and applying a charge-difference to the crystal surfaces) and measuring the time sound takes to reach the other end (by attaching a second piezoelectric crystal there) as shown in figure 12. Most moduli are measured by one of these last two methods.

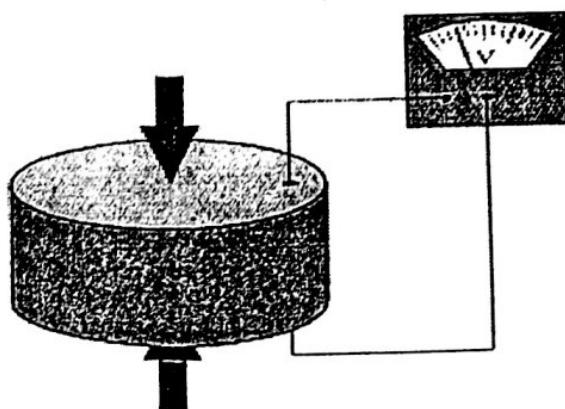


Figure 12

## Flexural strength tests of ceramics

To avoid the high expense and difficulties of performing tensile tests on ceramics, tensile strength is often determined by the bend test. The main advantage of the bend test, other than its lower cost, is that we use simple sample geometries. The specimens have either a rectangular or cylindrical geometry.

Brittle Materials, including ceramics, are tested by Flexure Test (Transverse Beam Test, Bending Test). There are two standard Flexure Test methods:

### 3-point Flexure Test

### 4-point Flexure Test

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**flexural strength** — a measure of the ultimate strength of a specified beam in bending.

## Flexural strength calculation

### 3-point Flexure Test

In this test a specimen with round, rectangular or flat cross-section is placed on two parallel supporting pins. The loading force is applied in the middle by means loading pin. The supporting and loading pins are mounted in a way, allowing their free rotation about:

- • axis parallel to the pin axis;
- • axis parallel to the specimen axis.

This configuration provides uniform loading of the specimen and prevents friction between the specimen and the supporting pins.

### 4-point Flexure Test

In this test the loading force is applied by means of two loading pins with a distance between them equal to a half of the distance between the supporting pins. The four-point bend test is preferred because an extended region with constant bending moment exists between the inner rollers.

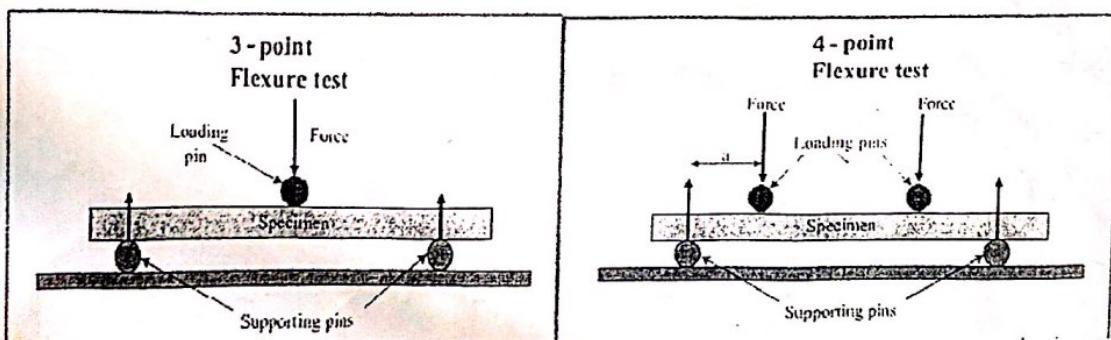


Figure 12 Types of bending test: a) 3-point bending. b) 4-point bending

## Flexural strength calculation

As a result of the loading, the specimen bends, causing formation of tension in its convex side and compression stress in the concave side. The cross head speed in flexural test normally varies within the range 0.004-0.4 inch/min (0.1-10 mm/min). Speeds 1 mm/min and 0.1 in/min (2.54 mm/min) are mostly used in the tests. The maximum stress and corresponding maximum strain are calculated for every load value. The results are then plotted in the stress-strain diagram.

**Modulus of Rupture (Flexural Strength)** is the stress of the extreme fiber of a specimen at its failure in the Flexure Test. Flexural Strength is calculated by the formula:

$$\sigma = 3LF/(2bd^2) \text{ in 3-point test of rectangular specimen}$$

$$\sigma = 3Fa/(bd^2) \text{ in 4-point test of rectangular specimen}$$

$$\sigma = 16Fa/(\pi D^3) = 2Fa/(\pi r^3) \text{ in 4-point test of round specimen}$$

Where

L – specimen length;

F – total force applied to the specimen by two loading pins;

b – specimen width;

d – specimen thickness;

r – specimen section radius;

a - distance between the supporting and loading pins;

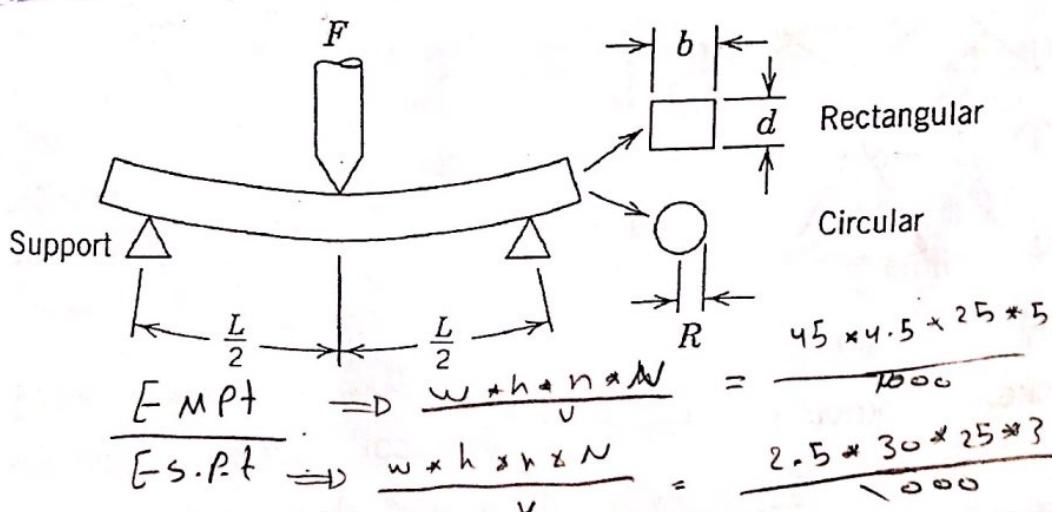
D – section diameter of round specimen.

We can't use the standard tensile test, but we still need elastic modulus and fracture strength.

**Solution: bend test.**

Most appropriate for bars, rods, plates, and wafers.

Possible cross sections



## Hardness Test of Ceramic materials

Hardness is a measure of a material's 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.

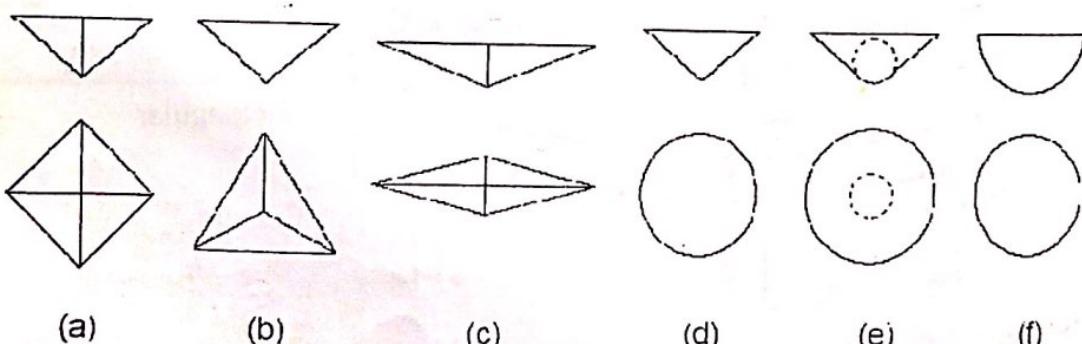
Hardness value helps to characterize resistance to abrasives or wear, resistance to plastic deformation, modulus of elasticity, yield strength, ductility, and fracture toughness. It is necessary for cutting tools, wear and abrasion-resistant parts, prosthetic hip joint balls and sockets, optical lens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

indentation test (IP (ج), Low)

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  $\leq 1$  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

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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 indentation 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:

$$HK = \frac{\text{load (kgf)}}{\text{impression area (mm}^2\text{)}} \\ = P/C_p L^2$$

where P equals the load, C<sub>p</sub> 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 described 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<sup>2</sup>. 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.

- ① 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

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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:

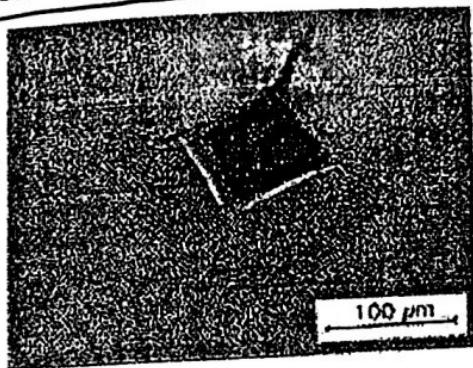
$$HV = 1.854 * F/D^2$$

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.

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لينة فتح الصدمة  
ليس متوفراً



### 3 Berkovich test

مختبر نادر لجوانب

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).

$$H = \frac{1569.7P}{d^2}$$

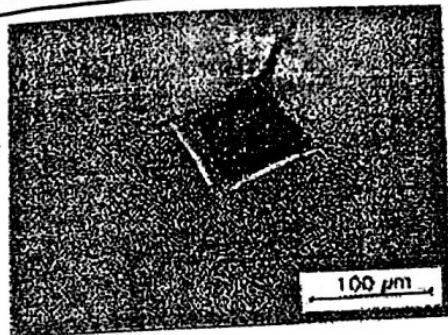
where  $P$  = test force in gf,  $d$  = diagonal of indentation in  $\mu\text{m}$ , and a triangular pyramid indenter with an angle of  $115^\circ$  is used.



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بيان مقدمة  
بيان نوبه وعذر



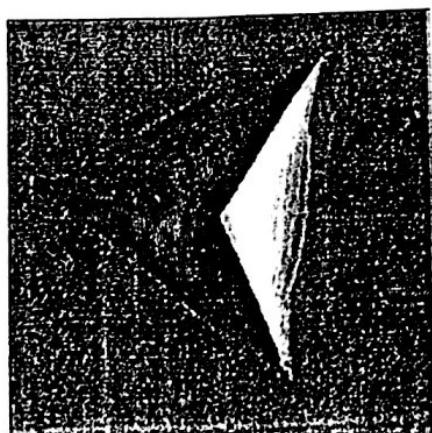
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## Important points in hardness testing of advanced technical ceramic

- 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 Mounting

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 rock or move during indentation. if necessary , it may be mounted in mounting resin for micro hardness tests. Specimen

## 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  $3 \mu\text{m}$  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.

## Relation of Hardness and Fracture Toughness

**Fracture Toughness** is ability of material to resist fracture when a crack is present. The general factors, affecting the fracture toughness of a material are: temperature, strain rate, presence of structure defects and presence of stress concentration (notch) on the specimen surface.

**Stress-intensity Factor (K)** is a quantitative parameter of fracture toughness determining a maximum value of stress which may be applied to a specimen containing a crack (notch) of a certain length,  $K_{IC}$  - stress-intensity factor, measured in  $\text{MPa}^* \text{m}^{1/2}$ ;

The use of the Vickers hardness indentations to measure fracture toughness ( $K_{IC}$ ) has become quite popular. Indentation hardness is a measurement of the size of an indentation made by a diamond pyramid-shaped indenter of specified size and shape pressed into a polished surface by a known load. Among a variety of indenter geometries used in hardness testing, the Vickers indenter is one in the most widespread use. Cracks associated with Vickers or Knoop hardness impressions are widely used as artificial defects of "known" size for the fracture toughness ( $K_{IC}$ ) measurement of ceramics. The quantitative relations between the surface crack length of indentation cracks and the fracture toughness. Interest in this method stems from its simplicity and the small volume of material required to conduct  $K_{IC}$  measurements. Vickers indentation is implanted onto a flat ceramic surface and cracks develop around the indentation with their lengths in inverse proportion to the toughness of the material. By measuring crack lengths, it is possible to estimate  $K_{IC}$ .

The conventional procedure of hardness testing consists of applying a fixed load on a diamond indenter and measuring, with the help of a microscopy, the dimension of the resultant indentation on the surface of the test material after unloading. The cracks length is inversely proportion to the material toughness; therefore  $K_{IC}$  may be estimated by measuring the cracks length. Toughness measured by the crack-length method is dependent on the elastic modulus of the material, microindentation hardness and crack length, and the applied load

$$K_{IC} = 0.016 \left( \frac{E}{H} \right)^{1/2} \times \frac{F}{c^{3/2}}$$

where  $F$  is the load in Newtons,  $c$  is the crack length from the center of the indent to the crack tip in meters,  $E$  is the Young's modulus in GPa and  $H$  is the Vickers hardness in GPa.

