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Mechanical and Surface Properties of Ceramic Materials [Review Article]

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Abstract

Dental ceramic materials can provide aesthetic realism. Ceramics cause regular & diffuse light transmission and both specular and diffuse light reflection. Therefore, it has the potential to reproduce the depth of translucency, depth of color and surface texture of natural teeth. The following reviews highlight the mechanical and surface properties of the ceramic materials.

Keywords;Ceramic;Mechanical properties;Surface properties.

Introduction:

Ceramic materials are inorganic and non-metallic materials. Most ceramics are compounds between metallic and non-metallic elements for which the interatomic bonds are either totally ionic or predominantly ionic but having some covalent characters. Most dental ceramics are compounds of oxygen with metals or semimetals that have some properties of both metals and non-metals. But all ceramic products are non-metallic in nature [1,2].

Dental ceramic materials can provide aesthetic realism. Ceramics cause regular & diffuse light transmission and both specular and diffuse light reflection. Therefore, it has the potential to reproduce the depth of translucency, depth of color and surface texture of natural teeth. Moreover, dental ceramics have superior biocompatibility, high stability in the oral cavity, high chemical inertness, temperature resistance, high wear resistance, high corrosion resistance, low density when compared to metals and coefficient of thermal expansion and contraction near to that of natural teeth [3-5].

In the past, ceramic materials had limited applicability because of their brittle nature. Their principal drawback was catastrophic fracture in a brittle manner with very little energy absorption. To overcome this point, different toughening and strengthening mechanisms were introduced by using new composites, other multiphase ceramics with useful toughness and applying different toughening and strengthening methods [1,5].

Mechanical Properties:

Brittle fracture of ceramics:

Both crystalline and noncrystalline ceramics always fracture before any plastic deformation can

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occur in response to an applied tensile load. The unstable fracture of ceramics starts from critical flaws. This phenomenon can be explained by "**The weakest point**" theory which states that fracture always propagates from the largest flaw favorably oriented to the tensile stress [1,6].

The brittle fracture follows the formation and propagation of cracks through the cross section of material in a direction perpendicular to the applied load. Crack growth in crystalline ceramics may be:

• **Trans-granular crack growth**: through the grains (cracks propagate along specific crystallographic planes of high atomic density).

• **Intergranular crack growth**: along the grain boundaries [1].

The measured fracture strength of most ceramic materials is substantially lower than that predicted by interatomic bonding force theory. This could be explained by the presence of different flaws in the material that act as stress raisers (the amplitude of the tensile stress is amplified with the absence of an opposing mechanism that can slow down or divert this damaging action as plastic deformation due to the nature of bond present in ceramics which ranges between ionic and covalent bonds) [1]

Stress raisers could be:

Microscopic defects as minute surface cracks, interior cracks or grain corners.

Macroscopic defects as voids, inclusions, sharp corners, scratches or notches.

According to Griffith energy balance theory, there are two conditions necessary for crack growth:

i. The bonds at the crack tip must be stressed to the point of failure. The stress at the crack tip is a function of the stress concentration factor, which depends on the ratio of its radius of curvature to its length.

ii. For an increment of crack extension, the amount of strain energy released must be greater than or equal to that required for the surface energy of the two new crack faces [17].

The degree of amplification of stress depends on:

- Crack length
- Radius of curvature of the crack tip.

The maximum tensile stress could be calculated from this equation:

$$\sigma_m = 2\sigma_0 \left(\frac{a}{\rho_t}\right)^{1/2}$$

Where, σ_m = maximum tensile stress
σ_0 = magnitude of the applied tensile stress
ρ_t = radius of curvature of the crack tip
a = the length of the surface crack or half of the length of the internal crack [1].

The distribution of flaws, crack size, shape and orientation differ from one sample to another (i.e.: its resistance to crack propagation is statistically distributed according to the flow size distribution). Additionally, properties of dental ceramics vary widely, depending on the nature, amount, particle size distribution of crystalline phase and porosity present (ceramic microstructure). Furthermore, the mechanical strength of ceramic materials is greatly affected by their processing and fabrication methods. Besides, the test methodology variability is high, making comparison of materials difficult [6,7].

From the previously mentioned, we can conclude some factors that can affect the mechanical resistance of ceramics:

- Presence of cracks.
- Crack size, geometry and orientation.
- Nature of bonds in ceramics.
- Microstructure of ceramics.
- Processing and fabrication method of ceramics.
- The methodology of the test used for measuring the mechanical properties.

Fracture mechanics

The concept of fracture mechanics is applied to brittle materials to provide sound parameters for ceramic characterization due to the high variability of defects' distribution [7].

Fracture mechanics is the science that allows scientists to analyze the influence of flaw/stress

interaction on the probability of crack propagation through brittle solids. It allows quantification of the relationships between material properties, stress level, the presence of crack-producing flaws and crack propagation mechanism [2,7].

Fracture toughness

It was found that when a brittle material was subjected to tensile stresses, specific crack shapes in certain locations were associated with greatly increased stress levels. Therefore, determining the ability of a material containing a crack to withstand applied load is crucial. **The fracture toughness (KIc)** of a material represents the resistance of a material to rapid crack propagation in a stress field. I subscript for *KIc* denotes mode I crack displacement [1].

$$KIc = Y\,\sigma\sqrt{\pi a}$$

K_{Ic} = *Fracture toughness.*

Y = Dimensionless parameter or function that depends on both crack and specimen sizes and geometries as well as on the manner of load application as shown in figure (1).

σ = *Applied tensile load*

a = Length of a surface crack, or half of the length of an internal crack.

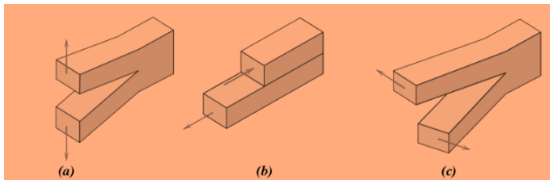


Figure1: Schematic diagram showing modes of crack surface displacement (a) mode I, tensile mode (b) mode II sliding mode (c) mode III tearing mode.

Many factors might affect the fracture toughness of a material, the most influential of which are **temperature**, **strain rate**, and **microstructure**. The magnitude of *KIc* decreases with increasing strain rate and decreasing temperature [1].

The difference between strength of the material and fracture toughness is that strength is dependent on the size of the initiating crack present in the particular sample. Meanwhile, the fracture toughness of a material is generally independent of the size of pre-existing cracks. It is an inherent property of the material to resist rapid crack propagation [2] .

c. Tests used to measure fracture toughness:

i. Single-edge-notch beam test (SENB):

- In this test a starter notch is formed in the specimen.
- The notch tip width should be less than twice the grain size of the tested material.
- The load application and the setup of this test is similar to the four-point loading test.
- The fracture toughness is estimated from:
  - Maximum load
  - Dimensions of the specimen
  - Dimensions of the notch
- Disadvantages of the SENB test:
  - The difficulty of the specimen and notch preparation that needs special equipment.
  - Specimen size is relatively large and the crack size is larger than the real flaws in the material. [8].

ii. Small crack tests:

- Indentation fracture test:
- In this test a series of cracks are introduced by Vickers indenter.
- By viewing the indentation site, the cracks appear to originate from the corners of indentation.
- The indenter load should only produce 4 cracks originating from the four corners of the indenter.
- No crack chipping or branching should occur.

Equation used to measure fracture toughness (K_{Ic}):

$$K_{Ic} = 0.016 \left(\frac{E}{H_v}\right)^{\frac{1}{2}} \left(\frac{P_v}{C^{3/2}}\right)$$

E = Young's modulus P_v = Indentation load

H_v = Hardness C = Crack length

Advantages of Indentation Fracture test:

- Simple technique
- Requires few specimens
- No standard test specimens are needed

Only flat polished surface is required (less than 1mm 2).

- Saves time and cost.
- Disadvantages of Indentation Fracture test:

- The scatter in the obtained values that may reach up to 40%.

This could be due to the crack production and subcritical crack growth associated with the residual stresses present in the indentation field which makes the accurate measurement of crack length difficult [8].

Indentation strength test:

This test is performed on two steps:

First step is the production of a flaw using microhardness indenter whether Vickers or Knoop.

Second step is the application of tensile stresses on indented surface of the specimen.

Fracture toughness (K_{Ic}) is calculated from the equation:

$$K_{Ic} = 0.59 \left(\frac{E}{H}\right)^{\frac{1}{8}} \left(\sigma_f P^{\frac{1}{3}}\right)^{\frac{3}{4}}$$

E= Elastic modulus H= Hardness

σ_f= fracture strength P= Indentation load

Fracture toughness is dependent on:

- Elastic modulus
- Indentation load
- Hardness
- Fracture strength

Advantages of Indentation strength test:

- It showed good agreement with conventional mechanics tests as SENB test.
- Reproducible results have been obtained.
- Fractography of ceramics

It is the analysis of the fractured surfaces. It involves the examination of crack propagation path and microscopic features of the fractured surface. A failure analysis focuses on determination of the location, type and source of the crack initiating flaw. [1].

Fractographic investigation could be done using:

- Magnifying glass
- Low power stereo binocular optical microscope in conjugation with light source.
- Scanning electron microscope.
- Transmission electron microscope [9].

After nucleation and during crack propagation, crack accelerates until a critical velocity is reached. By reaching this velocity the crack starts to branch. Successive repetition of branching may occur till a family of cracks is produced.

The site of nucleation can be traced to the point where a set of cracks converge.

The rate of crack acceleration and the degree of branching are directly proportional with the stress level.

During propagation the crack interacts with microstructure, stress & generated elastic waves:

- Producing distinctive features on the fracture surface.
- Providing important information on the origin of the crack initiation.
- Providing also important information on the source of the crack producing defect.

Three major features are present on the fracture surface:

- Smooth mirror region:
- Formed by slow growth of crack during initial failure.
- Surface is flat and smooth.
- The outer perimeter is roughly circular with the crack origin in the center.
- For glass ceramics the mirror region is extremely flat and highly reflective. While for polycrystalline this region is rougher and has granular texture. (1,8)

Mist region:

- It is a faint annular region just outside the mirror region.
- For polycrystalline it is often not detected.
- As the crack lengthens both strain energy and kinetic energy increase leading to an increase in the velocity of the crack till reaching terminal crack velocity. At which the velocity will not increase furthermore, instead additional microcracks start to form at the tip of the crack. These microcracks have no enough energy to propagate leaving visible perturbations on the fracture surface.

Hackle region:

- It lies beyond the mist region.
- It has rougher texture.
- It is composed of set of striations radiating away from the crack source in the direction of crack propagation, as shown in figure (2).

From the mirror region radius, we can detect different information on the material. As the greater the acceleration rate, the sooner the crack reaches its critical velocity and the smaller the mirror radius and vice versa. [1,8]

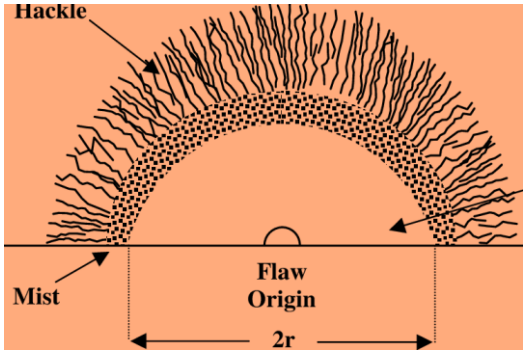


Figure 2: Schematic diagram that shows typical features observed on the fracture surface of a brittle ceramic.

1) Edge toughness:

In brittle materials, cutting operations frequently result in excessive edge chipping. This occurs during milling of ceramic blocks. Thus, in grinding operations, parameters should be adjusted to avoid large chip sizes for an adequate final surface finish. Chipping of veneering ceramics while in function is another serious problem, clinical longevity researches have shown that chipping is a major cause of failure.

Edge toughness is used to obtain information about the edges of materials rather than the bulk or surface characteristics. It is calculated through edge chipping test.

Edge Chipping Test is used to evaluate the resistance of brittle materials to flaking near an edge.

The specimen used for edge chip fracture test is a rectangular block.

Chips are formed by advancing an indenter into a material near an edge.

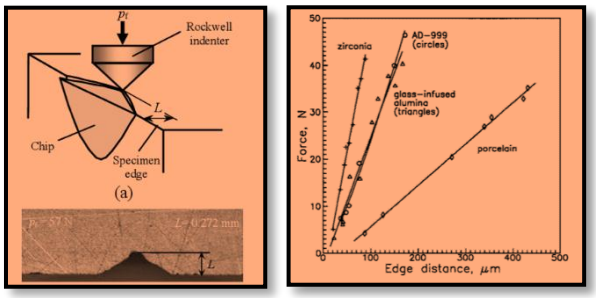
Indenters include Rockwell, Knoop and Vickers pyramidal indenters.

The force required for chip formation (F) at distance(d) from the edge is plotted.

$T_e = \Delta F / \Delta d$

Edge toughness (Te) is defined as the slope of the line representing the relation between the force necessary to cause edge chipping and the distance from the test specimen edge at which the load is applied[15,16], as represented in figure (3).

Figure 3: Edge Chipping Test and relation between



applied force and edge chipping.

4) Stress-strain behavior

a. Flexural strength

The stress–strain behavior of brittle ceramics is not usually obtained by tensile test for three reasons:

- First, it is difficult to prepare and test specimens having the required geometry.
- Second, it is difficult to grip brittle materials without fracturing them.
- Third, ceramics fail after about 0.1% strain, which necessitates that tensile specimens be perfectly aligned to avoid the presence of bending stresses, which are not easily calculated.

Accordingly, more suitable bending tests are most frequently used for strength measurement of dental ceramics. These include uniaxial bending tests (three-point, and four-point bending tests) and bi-axial bending tests [1].

i) Uniaxial bending tests:

- Three-point loading test:

A beam is supported at each end with load applied in the middle till fracture occurs.

$$\sigma_{3p} = \frac{3FL}{2bd^2}$$

σ_{3p} = Flexural strength of the three-point test (MPa)
F = Force (N) L= Span length (mm)
b = Width of the beam (mm)
d = Thickness of the beam (mm)

According to the DIN EN 843-1 cross head speed should be 0.5 to 1.0 mm/min so that the fracture occurs 5 & 15 sec.

For this test the principal stress on the lower surface is tensile and it usually causes cracks originating from this site leading to catastrophic failure.

Advantages:

- The test design is uncomplicated.
- Sample shape is relatively simple.

Disadvantages:

- Small volume of the specimen is exposed to the max. tensile stress.
- Random distribution of flaws within the material, lowers the probability of finding large sized flaws in material with small volume, thus showing higher strength.
- The specimens are very sensitive to edge fracture, so manual chamfering of the edges will improve the reproducibility of the test.
- The results can vary greatly depending on specimen size, span to depth ratio, fabrication method, type and duration of loading.

The surface polish is an important factor that can influence the results. So, to improve the accuracy of the results specimens should be highly polished, s shown in figure (4) [8].

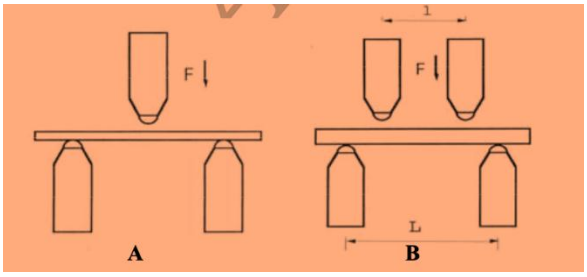


Figure 4: Schematic representation of three-point loading test (A) and four-point loading test (B).

Four-point loading test:

- The aim of this design was to apply load over a relatively wider area.
- The bar is placed over a mounting jig with two round supporting rods. The loading is done with two round chisels till fracture occurs.

$$\sigma_{4pt} = \frac{3F(L-l)}{2wh^2}$$

σ_{4pt} = Flexural strength of the specimen.

L = Span length (mm)

w = bar width (mm)

$l = Distance\ between\ loading\ chisels\ h$ = *bar height (mm)*

Advantages:

- Stress distribution has been improved.

Disadvantages:

- Highly sensitive to edge fracture, surface finish and sample dimensions [8].

Biaxial bending tests:

The biaxial strength testing has some advantages compared to the uniaxial testing:

- simple specimen preparation, no chamfering or roundation is required to the edges.
- Absence of tensile loaded edges. The load is applied centrally away from the edges.
- The multiaxial loading condition does not discriminate cracks in particular orientation.
- The biaxial tests are less sensitive to surface imperfections resulting from specimen preparation [10].

Different configurations of biaxial strength test are present:

Piston on three ball (Recommended by the ISO 6872:2008).

- Ball on ring
- Ring on ring
- Ball on three balls.

In piston on three balls test a disc shaped specimen supported by 3 steel balls with a diameter between 2.5 &6.5 mm and positioned 120° apart on a supported circle with a diameter between 10 and 12 mm.

The specimen is placed concentrically on these supports and the load is applied with a flat punch (diameter 1.4 ± 0.2mm) at the center of the specimen [10].

Elastic Behavior

The elastic stress–strain behavior for ceramic materials using these flexure tests is similar to the tensile test results for metals. A linear relationship exists between stress and strain. The slope in the elastic region is the modulus of elasticity. The range of moduli of elasticity for ceramic materials is between about 70 and 500 Gpa. It is slightly higher than that of metals [1].

Fracture resistance

- Flexural strength tests don't mimic the clinical situations; thus, the obtained results are partially the results of simple geometric shapes.
- In fracture tests specimens are fabricated in the anatomic configuration of teeth. This can be very useful in identifying the material's behavior.
- The anatomic specimen could be designed in the form of bridges, crowns or inlays.
- Then, specimens are loaded till failure occurs
- Examination of the failed glass ceramic is done to reveal the cause of failure.

Disadvantage:

Failure loads are usually very high compared to the range of loads reported regarding failure while in function [8].

Fracture statistics

When using bending test to measure the strength of a ceramic material, an average strength value must be calculated based on the number of tested samples. This is because of the wide distribution of flaws within the material that results in a considerable variation between samples.

This is different from metals which exhibit normal distribution of strength values. Ceramics exhibit an asymmetrical distribution curve which starts at a low strength value, increases gradually to a maximum value and then decreases sharply in the higher strength range. Therefore, for a ceramic restoration, a finite probability exists for specimens to fail near zero values.

The Weibull modulus is a material-specific parameter, which describes the flaw size distribution. The higher the Weibull modulus, the more consistent the material is. Indicating that defects or flaws are evenly distributed throughout the entire volume.

For determining the Weibull modulus, it is necessary to load a large number of samples(30-50) at a constant stress rate up to the fracture, using flexural tests.

The large value of Weibull modulus (≥20) ensures fewer fatal flaws, a smaller error in strength estimation, and greater clinical reliability. [8]

Surface Properties

Hardness

Accurate hardness measurement is difficult to gain due to brittleness and high susceptibility to cracking.

Hardness of ceramics is measured using Vickers or knoop test, which have pyramidal indenters. Spherical indenters produce severe cracking. Vickers is widely used for measuring hardness of ceramics, as shown in figure (5).

For both Vickers and Knoop tests, hardness number decreases with increasing load or indentation size till reaching a constant hardness plateau that is independent of load. The hardness number at this plateau varies from one ceramic to another.

An ideal test is designed to use sufficiently large loads that lie near the plateau, yet, it does not introduce excessive cracking.

To ensure standardization, for each specimen, three or more readings are taken and then averaged [18].

Advantages:

- The test design is uncomplicated.
- Sample shape is relatively simple [1].

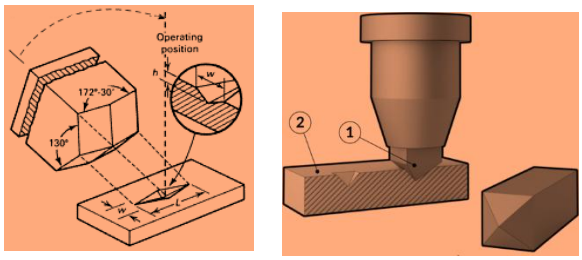


Figure 5: Schematic representation of Knoop indenter and Vickers indenter.

Martens Hardness & Indentation modulus:
Specimens are cut into disks (10×2 mm) with a low-speed diamond saw under constant water cooling. Martens Hardness & Indentation modulus are assessed with a universal hardness testing machine.

The diamond indenter of the machine is pressed vertically into the polished surface of the specimens with a load of 9.8 N for 20 seconds. All specimens are tested 6 times and mean values are calculated with the following equations:

For Hardness:

$$HM = \frac{F}{A_S(h)}$$

HM =Martens Hardness (N/mm^2)

F =applied force (N) AS (h) is the surface area of the indenter at distance h from the tip (mm2)[21].

For Indentation modulus:

It quantifies the elastic response of a material subjected to the action of a concentrated load in a single point. The relationship between the applied stress and displacements is no longer linear. Thus, the indentation modulus represents a close estimation of EIT is the elastic indentation modulus of the indenter (kN/mm^2)

$$E_{IT} = (1 - v_S^2)\left(\frac{\sqrt[2]{A_p(h_c)}}{\sqrt{\pi}\,S} - \frac{(1-v_i^2)}{E_i}\right)^{-1}$$

EIT is the elastic indentation modulus of the indenter (kN/mm2)

vs is the Poisson ratio of the specimen

vi is the Poisson ratio of the indenter

S is the contact stiffness

Ap is the projected contact area [20,21].

Wear:

Several attempts have been made to relate the hardness of dental materials to their abrasiveness and wear resistance, but recent studies have demonstrated other factors that influence the wear properties of a ceramic, such as microstructure, porosity, crystal size, surface roughness, and environment [11].

a. Two body wear resistance test:

- Dual axis chewing simulator is used.
- Cylindrical specimens are embedded in acrylic resin to be subjected to a two-body wear test.
- As antagonists, standard cusps (from yttria stabilized tetragonal zirconia polycrystalline are most commonly used) with a slight conical shape and a round tip.
- Cusps are embedded in autopolymerizing acrylic resin using custom-made copper specimen holders.
- Followed by application of the masticatory cycles.

Quantitative surface analysis could be done using a CAD/CAM three- dimensional contact scanner.

From each worn surface, a three-dimensional mesh is obtained.

The three-dimensional mesh is imported to a software. Moreover, the height of each zirconia cusp was measured before and after each test using a digital caliper. The height difference between the pre-test and post-test measurements of each cusp is recorded as the antagonist wear (mm) [13].

Disadvantage:

This test measures the volume of material lost but does not reveal mechanisms of wear.

a.Single pass sliding tests

Single pass sliding technique can also characterize modes of surface failure [12].

Ball on plate test:

- It is done in ball cratering machine.
- A ball (specimen) slides against the ZrO_2 disk.
- After testing, the samples are rinsed with deionized water and dried with ambient air. The diameter of the circular wear crater is measured by an optical microscope.

The wear is in the form of scratches parallel to the ball sliding direction. The scratches are isolated from each other and their number increases as the number of wear cycles of the test increases, as shown in figure (6) [14].

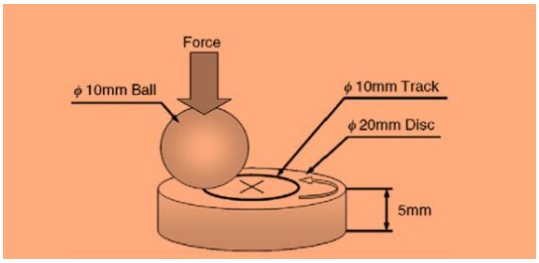


Figure 6: Schematic representation Ball on plate test.

Pin on plate test:

- This test simulates the masticatory motion. It is carried out as a simplified representative test for tooth-to-restorative material contact with the pin representing the tooth and the plate representing the

restorative material (zirconia). Artificial saliva could be added to simulate the oral condition.

- A reciprocating pin-on-plate Bruker-UMT-2 tribometer was used to evaluate the wear characteristics of tooth/zirconia tribopairs.
- The samples were fixed into an acrylic device, which is installed on the tribometer. This device is also used as bath for the artificial saliva solution.

The plates were cleaned ultrasonically in distilled water before and after the tests. The amount of wear for both pins and samples was measured by weight loss. Before and after testing the pins and the zirconia plates were placed in oven for 2 days at 35°C to have the same level of dehydration during weighting, as shown in figure (7) [19].

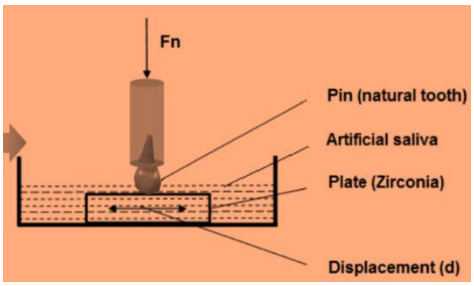


Figure 7: Schematic representation Pin on plate test.

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