Biomaterials Journal
http://www.biomatj.com
Online ISSN: 2812-5045

Type of the Paper (Review Article)

Hardness and Nano-indentation

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Abstract: The assessment of the mechanical properties of dental materials is vital for developing successful materials used in dentistry. These include hardness which has been used to predict the wear resistance of a material against applied forces such as occlusal loading. Hardness tests has taken many forms to accommodate the different materials' natures and behaviors. Efforts have been made to understand the relationship between hardness and other physical properties such as yield strength and elastic modulus. With the development of nano-based dental materials, it became important to establish indentation tests on the nanometer scale, known as nanoindentation tests. Nanoindentation is one of the most precise ways of determining and comparing the mechanical properties of restorative materials.

Keywords: *Hardness; Nanoindentation; Nanomaterials; Mechanical properties*

Citation: *Mona El-Deeb.* ***Hardness and Nano-indentation.*** *Biomat. J., 1 (9),1 – 7 (2022)*

https://doi.org/10.5281/znodo.5829408

Received: 22 August 2022
Accepted: 30 Septebmer 2022
Published: 30 Septebme 2022

Owing to its importance, hardness plays a major role in determining the clinical performance and durability of dental materials. For several years, researchers have shown interest to understand the relationships between hardness and other fundamental properties of materials.

Since the development of new indenters and instruments that allow indentations to be routinely performed on submicron scales, known as nanoindentation, there has been extensive research to apply such novel method for different applications and mechanical property measurements at surfaces.

Hardness

Hardness is one of the most characteristic properties of materials. According to materials science and engineering dictionary, ***hardness*** is defined as the measure of a material's resistance to deformation by surface indentation or by abrasion [1].

The surface behavior of a dental restorative matterial, as well as its bulk properties, affect the material's success during function. Surface properties determine the ability to be polished, the retention of that smooth surface, the resistance to scratching due to an opposing cusp. Besides, these characteristics are not easily modelled by a simple axial loading test [2] .

Measurement of hardness:

To obtain data about **surface hardness**, a number of techniques are available. **Indentation hardness** is determined by applying an indenter of specified geometry to the surface of the sample under a pre-determined load and, from a measurement of the width of the indentation (d) or its depth (t), its area is calculated (Figure 1). From such data, hardness is calculated, expressed in stress units. It corresponds to the stress that the material could just support at equilibrium without further deformation, that is to say, a kind of yield point [2]. Basically, the softer the material, the larger and deeper the indentation, and the lower the hardness number [3].

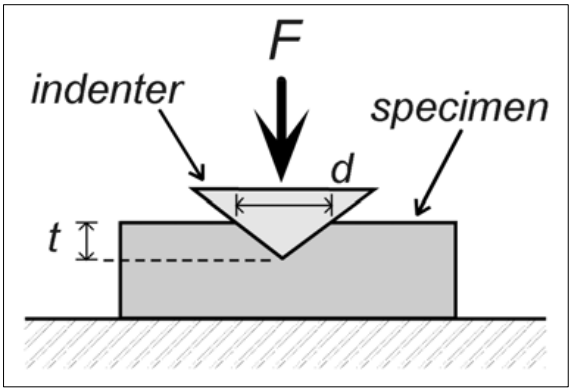

Figure 1 Generalized principle of an indentation hardness test. The size of the indentation, *d*, or the depth, *t*, is determined for a given load, *F* [2].

It is, of course, necessary for such a test that the indenter is rather harder than the test specimen, otherwise it would deform and invalidate the test, or even be destroyed [2]. Diamonds and sapphires have low coefficients of friction against metals and, therefore, are useful as indenters [4].

We may understand the nature of these tests by considering what happens as the indenter is lowered slowly into the specimen:

- Initially, there will be point contact. This corresponds to an infinite stress and the substrate deforms plastically.
- There is now a finite area of contact, but the stress is above the yield point. The indenter thus sinks steadily into the specimen causing further deformation.
- Only when the area of contact has increased to the point where the actual stress is just identical to the yield stress will the movement stop (Figure 2).

Thus, we can see that this is a kind of strength test, but in a reversed manner. Instead of increasing the load or strain of a specimen of predetermined cross-sectional area until yield occurs, in a hardness test the *area* over which a fixed load is applied is allowed to vary until equilibrium is achieved [2].

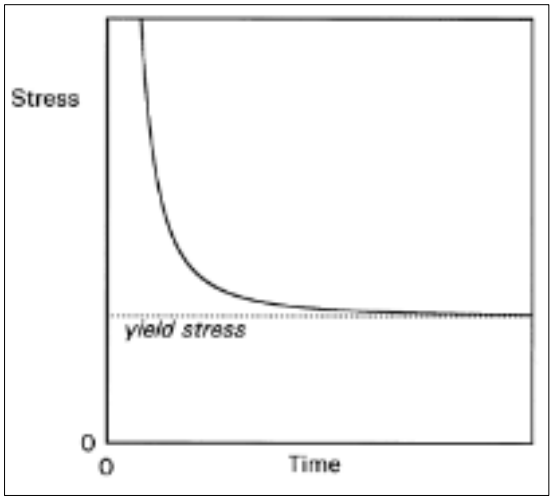

Figure 2 Diagram showing the variation in stress beneath a hardness indenter as it sinks into the test substrate [2].

The relation between hardness and other mechanical properties:

It is reasonable that a general association is found between hardness value and the **yield stress** of a material. Several relationships were suggested, including the following empirical equation, which is widely used in literature [5]:

$$H = c\,\sigma_y$$

Where H represents hardness of the material, and σ_y is the yield strength, while c typically lies between 1 and 3, depending on whether the material has a collapsible structure (porous, network, cellular) or can be deformed at almost constant volume (for most materials $c \approx 3$). In porous or cellular materials, collapse of the porosity or cells may accommodate some or all of the deformation.

Other publications reported the relationship between the Vickers hardness value and the ultimate tensile strength (UTS) according to the relation [5]:

$$H_V \approx 3\,\sigma_{UTS}$$

To further improve the understanding of these relations above, *Zhang et al* examined the relationship between Vickers hardness, yield strength, and ultimate tensile strength (UTS) for different kinds of materials. It was observed that materials with different indentation morphologies always exhibit different relationship between hardness and strength [5].

The morphology of the resultant indentations could be classified into three types: “sink-in”, “pile-up” and “crack” (Figure 3).

- Coarse-grained materials with good ductility always exhibit high work-hardening ability. Materials near the indenter (part A in Figure 3(a)) are hardened by the applied plastic deformation. Then, the hardened part A could compress the materials around it (part B in Figure 3(a)), and induce its plastic deformation. Once the part B is hardened, it could also induce the plastic deformation near it. Therefore, the plastic

deformation could be uniformly extended in a relatively large range, which is related to the slip bands area in the surface, as shown in Figure 3(d). The hardness of materials with "sink-in" morphology only represents a hardened state of material.

- In pre-deformed materials through work hardening, the plastic deformation induced by hardness test always concentrates in a relatively small area near indentation. The materials near indentation are continuously hardened but cannot induce the plastic deformation far away from the indentation (Figure 3(b) and (e)), which will lead to "pile-up" morphology, shown as a small bulge of material at the periphery of indentation. The piled-up materials near the indentation could be hardened to UTS much easier due to the lack of ductility and concentration of plastic deformation. The hardness of these materials could represent the intrinsic property related to UTS.
- With further decreasing the plastic deformation ability, the materials become much brittle and the extrusion behaviors under the penetration of indenter are greatly limited. In this case, the normal stress (indicated as σ in Figure 3(f)) near the corner becomes more important, which will induce the local cracking around the indentation, leading to "crack" morphology. This type of material has no work hardening ability under tensile stress, the hardness is related to the fracture strength of material [5].

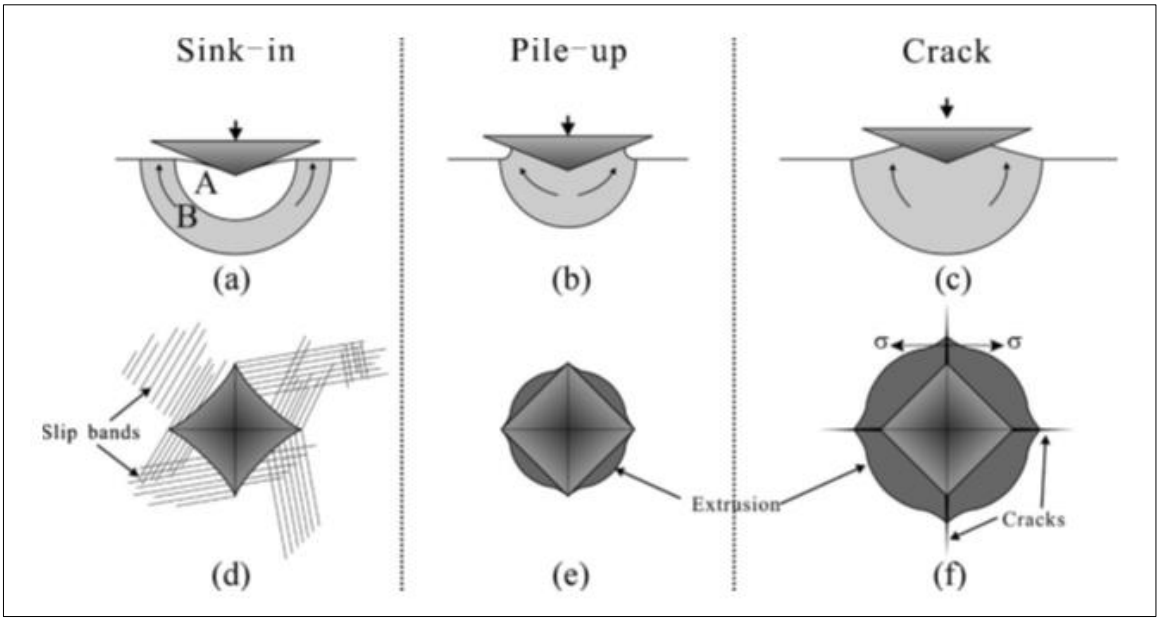

Figure 3. Schematic illustration of three types of indentation morphologies [5].

A good understanding of the relationship between the hardness and tensile properties of materials is very important for several reasons:

1. First, reliable hardness–strength correlations allow for rapid overall mechanical property evaluations using fast and inexpensive hardness testing instead of elaborate tensile testing. Also, no machining is required, except for surface preparation of the sample [4].
2. Second, several new materials are produced in small scale, these materials have insufficient volume to perform extensive tensile testing, hardness testing is often the only choice.

3. Third, in contrast to tensile tests, the hardness of a material can be measured nondestructively in situ on fully assembled components and devices, thus allowing for structural integrity tests in service, for example, in elevated temperature applications [5].

The most commonly used indentation methods for hardness measurements:

Generally, in macrohardness measurements (Brinell and Rockwell tests) the load applied is ≈ 2 N (or 1 kg), while in microhardness tests (Vickers and Knoop tests) a load < 2 N is applied [6].

- ***Brinell hardness number (BHN)***

The indenter for the Brinell hardness test is a small, hardened steel ball that is forced into the surface of a material under a specified load, leaving a round dent in the material, and hardness is determined by measuring the diameter of the dent.

- ***Vickers hardness number (VHN)* or (*diamond pyramid hardness test*)**

The Vickers hardness indenter is a pyramid-shaped diamond with a square base. Hardness is determined from the diagonals of the square-shaped indentation and taking the average of the two dimensions.

- ***Knoop hardness number (KHN)***

The indenter is also shaped like a diamond, but one diagonal is much longer than the other. Only the long diagonal is measured to determine the KHN.

- ***Rockwell hardness***

The Rockwell hardness test is used primarily to determine the hardness of steels. It uses different types of hardened steel balls or diamond cones and different loads. Each combination forms a specific Rockwell scale, suitable for materials of different hardness ranges.

- ***Shore A durometer***

The Shore A hardness test is used to measure the hardness of rubbers and soft plastics. The Shore A scale is between 0 and 100 units, with complete penetration of the material by the indenter yielding a value of 0, and a value of 100 indicates no penetration yielding [7].

Nano-Indentation

To accurately measure the properties of materials with microstructural constituents, smaller than the dimensions of the indenter, it is necessary to create indentations of a smaller size scale and to spatially control the location of the indentations. These *nano-indentation* techniques are able to apply loads in the range of pico-Newtons or nano-Newtons, resulting in indentations ≈1 μm in size [8]. Characterization of microstructural features is possible, such as grain boundary regions, filler phases (microphases), coatings, or filler/matrix interface [9].

A diamond indenter (Berkovich indenter) is usually used, in the form of a sharp pyramid with an equilateral triangular base [10]. During a typical nanoindentation test, force and displacement are continuously recorded as the indenter tip is pressed into the test material's surface. Information is derived from the penetration of the indenter on loading and also from the elastic recovery of the specimen on unloading (Figure 4) [11].

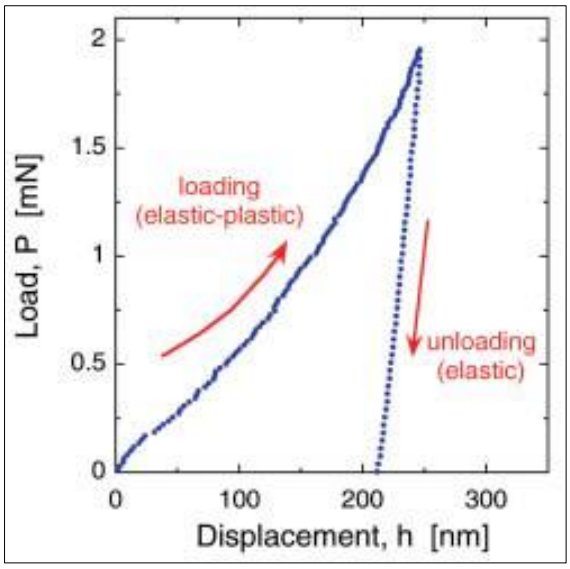

Figure 4. Example of a typical load-displacement curve (P-h curve) obtained during nanoindentation of an elastic-plastic material [11].

Advantages of nanoindentation:

1. Nanoindentation technique is not only used to measure hardness of micron-sized phases, it is also useful for measuring elastic modulus, as well as yield strength and fracture toughness for brittle materials [8].
2. With appropriate add-ons and software applications (such as atomic force microscope), it is possible to calculate substantial properties such as recovery rate and yield strength, which are difficult to determine with conventional methods [12]
3. Nanoindentation allows accurate control of the indentation force as well as accurate measurement of the indentation depth [10].
4. Indentation depth is continuously monitored, eliminating the need to image the indentation to compute mechanical properties [8].
5. It is relatively nondestructive, and the specimen preparation is less time consuming [12].
6. Allows the simultaneous comparison and evaluation of different variables and properties of different materials [12].

However, some problems may arise with nanoindentation:

1. Time-dependent responses may lead to inaccuracy in interpretation of load-displacement traces. This can be obvious in the case of soft, polymeric or biological materials [10].
2. Also, at micro and nano scales, indentation size effects have a major influence on test results; that is, different sizes of indenter may lead to different results [10].
3. Although nanoindentation techniques may be useful for characterizing homogenous materials, they are inappropriate for determining *bulk* properties of biphasic materials such as dental resin-composites. In this situation, the measurements obtained may reflect only the mechanical properties of the fillers or matrix not the combined material [10].

Conclusions

Hardness test should not be taken to mean more than a relative, comparative test. Because of the complexity of the stress pattern, no hardness value can be directly related to other fundamental mechanical properties, such as the yield stress. Obviously, there is some correlation, but not necessarily linear, and certainly not a scale factor. Any correlation with a mechanical property should be experimentally validated. Similarly, comparisons between different hardness scales are meaningful only through experimental verification [2,6].

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