

Review

# ***In Vitro* Testing Methods For The Evaluation of The Mechanical Properties of Composite Resins**

*Kompozit Rezinlerin Mekanik Özelliklerinin Değerlendirilmesine Yönelik In Vitro Test Yöntemleri*

Naz Bayar<sup>1</sup> , Merve Nezir<sup>2</sup> , Suat Özcan<sup>3</sup> 

## ABSTRACT

The selection of the right composite resin restorative material for clinical applications can be difficult, due to the wide range of available options. The mechanical properties of these materials have a significant impact on their longevity in the oral environment. Results from laboratory experiments that analyze the effects of compositional modifications can assist clinicians in making a more informed decision about the choice of the most appropriate material. This review examines the testing methods used to evaluate the mechanical properties of composite resin restorative materials.

**Keywords:** Composite resin; Mechanical properties; Testing methods.

## ÖZET

Klinik uygulamalarda, doğru kompozit rezin restoratif materyalin seçimi mevcut seçeneklerin fazla sayıda olması nedeniyle zor olabilmektedir. Bu materyallerin mekanik özellikleri, ağız ortamındaki ömürleri üzerinde önemli bir etkiye sahiptir. Materyal bileşimindeki modifikasyonların etkilerini analiz etmek için uygulanan laboratuvar deneylerinin performansı, klinisyenlerin en uygun materyal hakkında daha bilinçli karar vermelerine yardımcı olabilmektedir. Bu derlemede kompozit rezin restoratif materyallerin mekanik özelliklerinin değerlendirilmesinde kullanılan test yöntemleri incelenmektedir.

**Anahtar Kelimeler:** Kompozit rezin; Mekanik özellikler; Test yöntemleri.

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İletişim: Dt. Naz Bayar.

Gazi University, Faculty of Dentistry, Department of Restorative Dentistry, Ankara, Turkey.

E-mail: [208549401@gazi.edu.tr](mailto:208549401@gazi.edu.tr)

<sup>1</sup> Dt., Gazi University, Faculty of Dentistry, Department of Restorative Dentistry, Ankara, Turkey.

<sup>2</sup> Research Assistant Dt., Gazi University, Faculty of Dentistry, Department of Restorative Dentistry, Ankara, Turkey.

<sup>3</sup> Prof. Dr., Gazi University, Faculty of Dentistry, Department of Restorative Dentistry, Ankara, Turkey.

## INTRODUCTION

The impact of the human oral microenvironment on dental biomaterials used in restorative dentistry is significant. To fulfill the requirements of specific applications, dental materials must have properties similar to those of the natural tooth structure.

Evaluation of dental composites by mechanical testing is an essential step toward understanding their functional performance and esthetic qualities in the dynamic environment of the oral cavity. A material's mechanical behavior is determined by its response to forces or loads, and this determines its suitability for a specific application.<sup>1</sup> Mechanical testing and characterization follow standards established by international institutions such as the American Dental Association (ADA), British Standards, or Federation Dentaire Internationale.<sup>2</sup>

This review provides a comprehensive account of the mechanical and optical behavior of dental materials and assists in the selection of materials for successful, long-lasting dental restorations.

### 1. Flexural strength

Flexural strength, also known as the modulus of rupture, is an important measure of a material's resistance to deformation or breakage when subjected to bending or flexural stress. Optimizing flexural strength is critical to the long-term durability of dental restorations. Flexure testing can be performed in three ways: four-point flexure testing, biaxial flexure testing, and three-point flexure testing.

#### 1.1. Transverse bending (four-point and three-point) flexure tests

Transverse bending, a typical testing method for dental composites, provides a variety of approaches that depend on loading supports, load applicators, specimen geometry, and specimen preparation processes.<sup>3</sup>

The four-point flexure test, frequently used by researchers such as O'Brien,<sup>4</sup> involves the preparation of a rectangular sample supported at two ends, with force applied at two points in the middle. The values obtained using this method yield a typical decrease of 30-40% compared to those obtained from a three-point flexure test.<sup>5</sup> The

load applicator in four-point bending tests consists of two points spaced a certain distance apart. This design allows stress to be concentrated over a more extensive area of the beam. The design ensures that beam failure is confined to a specific region, which is essential for the accurate application of beam equations.<sup>6</sup> In the three-point method, beam failure may not occur directly under the applied load, which can result in inaccurate outcomes. The available evidence suggests that strengths tend to be greater in 3-point bending. However, it is worth noting that both tests can be carried out in similar ways on specimens with the same dimensions.<sup>7</sup> The location of the maximum bending moment and the maximum axial stresses are the primary differences between the three-point and four-point methods. In three-point loading, the maximum stress occurs directly below the loading point, while in four-point loading, it is spread out over the area between the loading points. This creates a stress gradient that exposes only a small area to a high tensile stress, making the measurement susceptible to flaws in the edge or surface of the sample. Despite the seemingly simple setup, misalignment and testing errors can easily compromise the results.<sup>7</sup>

#### 1.2. Biaxial flexure test

The biaxial flexure test involves the preparation of disk-shaped specimens. These specimens are then subjected to force from the center, resulting in counter-directional forces from the outer sides.<sup>5</sup> This approach is suitable for brittle materials and involves the use of various load configurations, such as ball-on-ring, piston-on-three-balls, ring-on-ring, and ball-on-three-balls.<sup>8,9</sup> Experimental data from previous studies demonstrated that only the ball-on-ring and ball-on-three-balls loading procedures could reliably measure the flexural strength of brittle dental materials. Uncertain fracture stresses have been observed with the other methods, which have led to inaccurate results. Careful determination of specimen dimensions and the ball-loading surface is essential for reliability, particularly considering the impact of thickness on deflection and stress distribution.<sup>8</sup> The biaxial test is commonly used for brittle materials that exhibit minimal deflection and shows potential as a method for evaluating dental materials in simulating intraoral behavior.<sup>10</sup>

## 2. Hardness

Hardness is a measurement of a material's resistance to localized deformation. It is not an inherent property but rather a product of a specific measurement method. This involves pressing a defined-shaped indenter into the surface of the material at a particular load and time, and then measuring the size or depth of the indentation once the force has been removed.

The simplicity of the hardness test has made it a common technique for characterizing resin composites. Traditional hardness testing procedures, such as those of Brinell,<sup>11</sup> Knoop,<sup>11</sup> Rockwell,<sup>12</sup> and Vicker,<sup>13</sup> involve the application of a hard indenter to the material, creating an indentation that persists after the indenter is removed.

### 2.1. Vicker's hardness test (diamond pyramid hardness test)

The Vicker's hardness test is performed using a diamond indenter that has a pyramidal shape with a square base and an angle of 136° between opposing faces. The test material is subjected to a force that varies from 1 gram to 100 kilograms for a period of 10 to 15 seconds.<sup>14</sup> A microscope is used to measure the two diagonals of the resulting indentation on the surface of the material after the load is removed.<sup>14</sup>

The Vicker's hardness is calculated by dividing the applied load by the square of the area of the indentation. It is important to include the Vicker's hardness number, test load, and dwell time in the report. The Vickers test is a widely used method for evaluating the hardness of hard metals, and it is often applied to dental materials, including dental gold alloys and dental tissues. It is also used by researchers to evaluate the degree of polymerization.<sup>13</sup> This method has several advantages, including the durability of the diamond tip, its ability to adhere to surfaces of varying hardness, and the accuracy of the measurements.<sup>15</sup>

### 2.2. Brinell hardness test

The Brinell hardness test is one of the oldest tests of its kind. It involves applying a specific load of 123 N to a polished surface of a material using a hard steel or tungsten carbide ball with a diameter

ranging from 1.6 mm to 10 mm. The ability of the material to resist penetration by the ball is reflected by the indentation left in the material. The penetrator remains in contact with the specimen for 30 seconds before it is removed, and the resulting indentation is carefully measured. The average hardness value is then determined by dividing the load by the indentation area. This test is particularly useful for assessing average hardness values due to its relatively large indentation area. However, Brinell testing may be less effective in evaluating smaller and more localized areas. The primary source of inaccuracy in Brinell testing, as with other indentation methods, is in the measurement of the indentation, making the method relatively subjective and operator-dependent. For standardized indentation measurement, computerized optical Brinell scopes with image analysis capabilities are available. It is critical to specify the load and the dwell time. Although Brinell hardness tests are not widely used in testing resin composites, they are important in certain applications.<sup>11</sup>

### 2.3. Knoop hardness test

The Knoop hardness test was designed as a microscopic alternative to the Vicker's test. It is especially well suited for evaluating thin plastic or metal sheets and fragile materials. The test uses a maximum applied load of 3.6 kgF. The Knoop test has the advantage of being highly adaptable to a broad scale of hardness by setting the test load. However, it requires a carefully refined and flat surface, resulting in a lengthy test procedure and time-consuming measurements.<sup>11</sup> This test utilizes a diamond indenter with a rhombic-based pyramid shape that applies a pre-determined test load for a defined duration. The duration of the initial load should not exceed 10 seconds, and the load should be maintained for a period of between 10 and 15 seconds. The Knoop hardness value is calculated by dividing the force applied during the test by the area of the indent. The Knoop indenter measures only the long axis and provides superior measurement accuracy when compared with the Vicker's indenter. This technique is particularly beneficial when the indentations are close to or at the edge of the specimen.<sup>13</sup>

### 2.4. Rockwell hardness test

The Rockwell hardness test is a method of quantify-

ing the permanent depth of indentation caused by the application of force by an indenter, such as a 120° diamond cone with a 0.2 mm radius spherical tip or a ball indenter. This technique involves the application of force in two steps. First, an initial test load of 3 kg, called the preload, is applied to the specimen for a maximum of 3 seconds. The indentation depth is measured and recorded. This method reduces the impact of surface texture by utilizing a low initial load as a reference point for surface texture and avoiding errors attributable to surface imperfections. A primary load is then added to achieve the required total test load, which is maintained for a specified dwelling time to allow for elastic recovery. The final position is evaluated relative to the preload position to determine the variation in indentation depth when the primary load is released. The Rockwell test is a suitable method for measuring the hardness of viscoelastic materials as it provides a direct measure of hardness. However, this method has several drawbacks, including the need for an initial weight measurement, the considerable time involved, and the possibility of losing track of the deformation after the weight is lifted.<sup>12</sup>

### 2.5. Barcol test

The Barcol resin depth test method directly measures hardness with a spring-loaded indenter that has a 1 mm diameter: it is used for the measurement of the degree of cement polymerization. To determine the degree of polymerization of the resin composite, samples of varying thicknesses ranging from 0.5 to 6.0 mm or more are prepared with 0.5 mm increments. Once the sample is prepared, the top surface is polymerized by exposing it to light. The depth of polymerization can be determined by comparing the Barcol test readings on the top and bottom surfaces and finding the thickest layer for which the bottom reading stays within 10% of the top reading.<sup>11</sup>

### 2.6. Shore A hardness test

A different method is needed to measure the hardness of soft materials compared to metals or polymers. The test avoids the issue of deformation caused by the indenter's tendency to return to its original depth due to the elasticity of the material under test. The Shore A hardness test is used to determine the hardness of rubber and soft plastics. The results of the test are presented on a scale of 0 to

100, where 100 indicates no surface penetration of the material and 0 indicates complete penetration.<sup>12</sup>

## 3. Surface roughness

Achieving polished surfaces on dental restorations is crucial for both oral comfort and cosmetic considerations. The primary goal of finishing and polishing procedures is to ensure that restorations have a proper shape, are properly aligned, and have a smooth surface that promotes the maintenance of healthy gingival margins. Researchers have found that composite resin restorations with polished surfaces accumulate less plaque.<sup>16</sup> Additionally, research suggests that a smooth surface can aid in preventing gum complications, discoloration, patient discomfort, and secondary caries.<sup>17</sup> Surface roughness is measured using instruments equipped with either optical or mechanical sensors. The measurements employ both qualitative and quantitative techniques. Qualitative techniques include scanning electron microscopy (SEM), while quantitative methods involve surface profile analysis using profilometers.<sup>18</sup> Furthermore, the Atomic Force Microscope (AFM), a relatively recent breakthrough in the field, offers an alternative approach for measuring surface roughness.<sup>19</sup>

### 3.1. Profilometers

#### 3.1.1. Optical profilometers

Surface topography refers to the three-dimensional features of a surface that can be analyzed and displayed using optical profilometers.<sup>17</sup> These devices provide a non-contact approach to obtaining 3D measurements of a surface. By using optical beams for scanning, precise measurements can be made at exact distances between reference points on the surface. Optical profilometers are known for their exceptional resolution, allowing measurements down to a few nanometers over a large area of 100 square meters. Their ability to provide comprehensive surface analysis and facilitate the understanding of the inherent properties of different materials and surfaces makes them an optimal tool.<sup>20</sup>

#### 3.1.2. Mechanic profilometers

Designed specifically for two-dimensional measurements, mechanical profilometers work by systematically scanning the surface with a diamond tip that

maintains a constant linear distance from the sample surface. These profilometers use a diamond tip to scan sensors horizontally with a resolution of between 20 and 50  $\mu\text{m}$ . To reduce the effect of surface grooves on the readings, it is advisable to take measurements from multiple angles.<sup>20</sup> Mechanical profilometers can store values using either digital or analog hardware and software. The sensitivity of the technique is at the level of 0.01  $\mu\text{m}$ . In cases in which the surface roughness is minimal, the resolution of the sensor is not sufficient, requiring the use of optical measurements.<sup>21</sup>

### 3.1.3. Scanning electron microscopy (SEM)

SEM, or scanning electron microscopy, is a crucial dental tool for examining composite surface roughness. SEM uses a narrow beam of electrons, only 10 nanometers thick, to scan the surface being examined. This technique provides high-resolution images that are essential for evaluating surfaces with scratches and distortions. The method is particularly effective in capturing minute structures and anomalies that can occur on composite resin surfaces. While SEM is commonly used, it has limitations, particularly in accurately describing three-dimensional surface features and complex surface topography. However, SEM examination provides valuable insights that significantly improve our understanding of composite resin materials and optimize their performance in dental applications.<sup>17</sup>

### 3.1.4. Atomic force microscopy (AFM)

The atomic force microscope (AFM) scans the surface of the sample using an extremely thin lever or tip, typically with a diameter in the range of 40 to 60 nm. The AFM captures the detailed interaction between the sharp tip and the surface of the material, allowing for the precise delineation of nanoscale surface features. This provides a comprehensive understanding of the topography and irregularities of dental composites. The use of atomic force microscopy allows the acquisition of high-resolution images, which are critical in advancing the understanding of material properties. This, in turn, facilitates the development of dental composite formulations to achieve optimal clinical outcomes.<sup>22</sup>

## 4. Bond strength

### 4.1. Tensile tests

Tensile tests are highly effective in evaluating the mechanical performance of dental composites. However, the lack of specific standards for dental materials presents challenges. Furthermore, the tensile properties of dental materials vary depending on specimen preparation, test speed, and environmental conditions. To ensure reliable and comparable results, it is necessary to monitor and record these factors according to established guidelines. Tensile testing is particularly effective with malleable materials such as metals and alloys. It can also determine interfacial strength.<sup>23</sup>

#### 4.1.1 Macrotensile tests

Tensile tests provide a more uniform force distribution than shear testing. This yields significantly more consistent bond strength test results. In macrotensile tests, the bonded contact is perpendicular to the load direction.<sup>24</sup>

Alignment is crucial. If not properly aligned, the force will bend the specimen. Furthermore, the operator must maintain precise alignment of the tooth surface with the bonded substance. Tensile testing demands more technical precision than shear testing.<sup>24</sup>

Macrotests, which were commonly used until the mid-1990s, are efficient. A finite element analysis by Van Noort *et al.*<sup>25</sup> showed an uneven force distribution along the adhesive interface, resulting in a high rate of cohesive failures caused by excessive stress on the substrate but not at the adhesive contact. According to Sirisha *et al.*,<sup>26</sup> macrotensile tests are still able to be used to measure material adherence to dental structures, despite their fundamental limitations, because they are simple to use. However, the authors observed that macrotensile tests frequently overestimate adhesive values and therefore fail, making microtensile tests more accurate.

#### 4.1.2 Microtensile tests

Multiple studies using conventional bond strength testing methods have found that the stress distribution at the dentin-resin interface is non-uniform. Homogeneous stress creates localized stress, which can lead to fracture. To address this issue, more

advanced test systems with smaller surface areas have been developed to avoid the limitations of conventional tests.<sup>27</sup>

In 1994, Sano *et al.*<sup>28</sup> developed the microtensile bond strength test, which revolutionized the measurement of tensile bond strength, particularly on extremely thin surfaces. This technique significantly improved the accuracy of bond strength evaluation by evenly distributing stress over these small surfaces.<sup>29</sup>

Tension occurs when axial forces act in the opposite direction on a straight object. Tensile strength measures the object's resistance to this force. Microtensile bond strength test values are obtained by dividing the stress after loading by the area of the bonded surface. However, a smooth surface is necessary to achieve this bond strength measurement, and it is necessary to ensure that stress is transmitted in the desired direction.<sup>27</sup>

Many shapes can be used for specimen preparation, including sticks, dumbbells, and sand glass.<sup>29</sup> However, it is important to understand the limitations of this method. Hardness, precision, and time are all important characteristics that can cause significant difficulties. Extreme caution must be taken when cutting and setting specimens to avoid irregularities that could nullify tensile bond values.<sup>29</sup> This is undoubtedly a challenging effort, but its promise for evaluating the long-term adhesive properties of composite resins *in vivo* is evident.

#### 4.2. Push-out test

The push-out test, introduced in 1970, is a reliable method for evaluating the strength of dentin bonding.<sup>27</sup> For this test, a spherical piece of dentin is created, and a hole of the appropriate size is drilled. An adhesive system is applied, and the material is placed in the hole. A pointed tip with a diameter ratio of less than 0.85 is then inserted into the material, and pressure is applied until separation occurs. The force required for separation is carefully measured and documented. This test is more suitable for clinical applications than shear and tensile tests, since it accurately simulates the bonding of materials to dentin and verifies the effectiveness of the adhesive system.<sup>24</sup> To overcome the limitations, the push-out test, which is derived from the shear punch test, has been proposed as a more suitable approach for evaluating

the bond strength of intracanal restorative materials. However, the application of the shear punch test formula to calculate the bond strength in the push-out test presents certain challenges. Factors such as pin size, specimen thickness, and material type significantly affect the predicted shear strength. Despite the common use of the push-out test,<sup>30,31</sup> no study has systematically investigated the effect of these parameters on the predicted bond strength, and thus the limitations of the method have not been properly addressed.

#### 4.3. Shear test

Shear strength testing is a popular method for analyzing the resistance of composite resins to forces applied parallel to their surface or along an established plane. This method is preferred over tensile bond strength testing due to its simplicity. Tensile tests can be challenging to perform without creating adverse stress distribution when positioning specimens in the testing equipment. Shear tests are commonly used due to the simplicity of specimen preparation and the test technique. However, concerns have been raised about the accuracy of measurements, primarily because cohesive failure within the substrate has become more common with the use of new adhesives with higher bond strengths.<sup>31</sup>

To gain a deeper understanding and improved accuracy, we must consider the valuable perspectives of pioneers such as Della Bona<sup>32</sup> and van Noort.<sup>25</sup> Their research highlights the fundamental challenges that contribute to the limitations of shear strength bond testing, ultimately leading to more accurate ways to evaluate the performance of bonded interfaces.

##### 4.3.1 Macro-shear tests

Before being placed in the universal testing machine, the area to be bonded is prepared under appropriate conditions for the macro-shear bond strength test. The machine's applicator arm applies force to the part being tested for bond strength by attaching a single-angled nail-shaped tip, flat chisel, or wire loop. The shear bond testing method uses a knife-edge device to isolate the bond specifically to the tooth surface. The ISO standard specifies that the testing instrument's cutting edge must operate at speeds between 0.45 and 1.05 mm/min. The bond test result is determined by dividing the maximum

force by the surface area over which the bond formed. This calculation is based on the concept of applying a constant force until failure occurs at the surface where two materials are bonded using an adhesive. In macro-shear bond testing, bond failure occurs when the bond between two materials fails due to tensile tension.<sup>24</sup>

#### 4.3.2. Micro-shear tests

Shimada *et al.*<sup>33</sup> developed the micro-shear bond strength test in 2002. In this method, a maximum number of specimens is obtained from a single tooth.

The test specimens are cut into 1 mm slices perpendicular to the long axis of the tooth. The slices are then cut into 1mm square sticks. The sticks are then mechanically compressed and connected to the microshear bond tester. The rods are inserted into the microshear tester with the fracture points aligned with the composite resin and tooth contact.<sup>33</sup>

This test can be used to determine the profile of both surface and deep sublayers, either at the desired location or throughout the material, to evaluate the bondability of adhesive systems. It can also be used to investigate the effect of moist and humid environments, including oral fluids such as saliva, on the bonding of adhesive systems. The primary reason for this is the test's ability to analyze superficial regions (0.02-0.05 mm).<sup>27</sup>

### 5. Accelerated aging

Loss of physical, chemical, and mechanical properties occurs with natural aging. *In vitro* simulation using the "aging procedure" in a laboratory environment mimics the natural aging process that occurs in the oral environment. In other words, aging can be experimentally reproduced in the laboratory.

Exposure to heat and cold, moisture, the erosive effect of acidic pH, the abrasive effect of brushing, and other variables can all degrade dental restorative materials. In the laboratory, the aging process of dental restorative materials allows us to observe changes, such as the long-term progressive increase in surface roughness over a short period of time.<sup>34</sup>

#### 5.1. Storage in water

The most commonly used aging method described in the literature is storage in distilled water at room temperature or 37°C for 3-12 months. This approach is widely accepted as a reliable indicator of the stability of adhesive bonding. Long-term water storage and thermal cycling combined with bond strength testing are methods commonly used to evaluate the degeneration *in vitro* of restored teeth. Data from before and after degradation procedures can be standardized and compared with previous publications.<sup>35</sup>

#### 5.2. Thermocycling

The thermocycling approach involves alternating hot and cold water for 10,000 to 30,000 cycles. Intermittent contraction-expansion stress can cause hydrolysis of exposed collagen fibrils, resulting in fissure propagation along the adhesive contact. This allows water and pathogenic oral fluids to penetrate.<sup>36</sup>

#### 5.3. Storage in NaOCl solution

The storage procedure is carried out in a 10% NaOCl solution for 1 to 5 hours, and the non-specific proteolytic properties of hypochlorite are used to mimic aging by degrading organic resin and tooth contact components and exposed collagen fibrils.<sup>37</sup>

#### 5.4. Toothbrush simulator

Toothbrush simulators are commonly used to evaluate the effects of toothbrushing on both hard dental tissues and dental materials. It is well known that wear on these surfaces is not only caused by the act of chewing food but also by the mechanical action of toothbrushing. How and how often people brush their teeth, the stiffness of the bristles, the dexterity of the patient, and the type of toothpaste used can all affect the amount of wear. In addition, the consumption of acidic foods before brushing can contribute to the erosion of tooth surfaces, exacerbating the effects of wear.<sup>38</sup>

A common method used in aging is the use of a toothbrush simulator. This simulator consists of a toothbrush that moves in a cyclic linear movement across the surface of the material being tested, implementing an adjustable pressure force. For example, a force of 200 g, equivalent to 2 N of strength, may be applied.<sup>39</sup> The standardization of the tooth-

brush simulator test is of critical importance, with respect not only to the device itself but also to the toothbrush that is selected.

### 5.5. Jaws simulator

In 1980, DeLong and Douglas<sup>40</sup> introduced the concept of an “artificial oral environment”. This concept combined a jaw movement simulator with an artificial oral environment.

The jaw simulator included models of upper and lower teeth, sometimes with a single tooth with a filling. Servo-hydraulic motors facilitated the movement of these tooth models, with one motor controlling vertical movement and another controlling horizontal movement. Computer-controlled precision ensured accurate replication of physiological chewing movements. The simulator allowed individual adjustment of bite force and chewing cycles.<sup>41</sup>

## 6. Wear resistance

The direct effect of compositional parameters on wear resistance causes resin composites to exhibit different wear patterns. The loading force should pass smoothly through the polymer matrix to the filler particles. The size, composition, and strength of the filler-polymer matrix bond and the polymerization kinetics determine the wear properties of dental composites.<sup>42</sup>

### 6.1. Pin-on-disc tribometer

The pin-on-disc tribometer wear test is a method commonly used in various industries for evaluating the wear resistance of materials. In this method, a “PIN” body in the shape of a roller or non-rotating ball is fixed to the surface of a disc-shaped specimen. The PIN is loaded with a specified force at a specified distance from the center of the specimen. The disk is then rotated at a selected motor speed and completes a specified number of revolutions, forming a track on the sample surface.<sup>43</sup>

This test is preferred because of its simplicity, standardization, and cost efficiency. However, there have been challenges in comparing the results of different studies due to variations in parameters such as load, speed, and the number of cycles. In addition, the test device does not accurately replicate conditions in the oral cavity, which is a critical factor when study-

ing wear in dental materials.<sup>43</sup>

### 6.2. Nanoindentation

Nanoindentation wear testing has become a critical aspect of studying the physical properties of composite resins, because it allows materials to be evaluated under controlled conditions. The testing process involves applying a diamond tip with a defined shape to specimens prepared under specific conditions. Diagonal lines are measured. Based on a known force, the hardness is calculated. Modern testing methods now involve computer-controlled, real-time processes that draw load curves to evaluate hardness and use reliever curves to evaluate modulus. These adaptations of the classic indentation test allow for non-destructive testing and offer the advantage of micro- and nanoindentation scales.<sup>44</sup>

### 6.3. Scratch test and confocal laser scanning microscopy (CLSM)

This test has a long history, beginning in 1824 with the development of the Mohs scale. Over time, the scratch test has evolved and modernized, focusing on the use of diamond as the hardest material and incorporating computer-controlled measurements. The test involves applying a precisely-defined diamond tip with increasing force and linear motion to the surface of the material being tested. The force (F) that required to produce a scratch with a width of 0.01 mm is measured. Advances in microscopy, particularly confocal laser scanning microscopy (CLSM), have further enhanced the analysis of scratches. CLSM can capture three-dimensional images, allowing detailed examination of scratch boundaries, their depth, and the effects of material homogeneity.<sup>45</sup>

## 7. Fracture toughness

The resistance to crack propagation from a preexisting flaw is a characteristic of fracture toughness, which remains consistent regardless of the test method or sample geometries. The determination of fracture toughness requires a specimen with a specified flaw, through which failure is induced by crack propagation. Fracture toughness plays a significant role in characterizing dental composites, since fracture represents one of the primary modes of failure.



There are several methods available to test fracture toughness, with convenience frequently influencing the choice of methodology. Dental composites generally exhibit relatively low fracture toughness, placing them in the category of brittle materials. However, they do undergo some plastic deformation during testing, which must be taken into account to achieve accurate results.

### **7.1. Single-edge notch: 3-point bending test**

The 3-point bending test is a commonly used method for determining the fracture toughness of dental composites. The test involves loading a sharply notched beam in 3-point bending, which applies a tensile force to the pre-crack, ultimately causing fracture through the thickness of the specimen. The loading span should be about 10 times greater than the thickness of the specimen.<sup>46</sup>

Simulating a true pre-crack, a critical assumption in this test is that the tip of the notch is infinitely sharp. Creating true pre-cracks in materials such as metals by cycling specimens in tension is possible, but this method is challenging or even impossible with brittle materials. Although pre-cracks can be created in composites, it is often cost-prohibitive due to the large specimen size.<sup>47</sup>

### **7.2. Compact tension test**

The compact tension method is an experimental technique commonly used in studies of fracture mechanics. It is particularly useful for evaluating the fracture toughness of materials. In this method, a tensile force is imposed across a notch in a plate-shaped specimen. The force is typically applied through perforations in the upper and lower surfaces of the plate, resulting in the opening of the notch. The compact tension method is more complex and sophisticated than the simpler beam bending method, but it provides more extensive data. One significant advantage of this method is its ability to determine the 'R-curve behavior' of a material. This behavior refers to an increase in fracture toughness as the length of the crack increases. In other words, the material becomes tougher and more resistant to catastrophic fracture as the crack grows. This toughening phenomenon occurs due to specific energy-dissipating mechanisms that are activated within the material during loading and cracking.<sup>46,47</sup>

### **7.3. Double torsion test**

The double torsion method is a technique used to study crack propagation in plate specimens containing a groove. The specimen is placed on parallel rods, and the load is applied near one end through two-point surfaces located on opposite sides of a precisely made notch. As the load increases, the crack propagates down the groove, which is visible and measurable on the top surface. The double torsion test may be the most applicable for dental composites in which pre-cracking is difficult. One of the advantages of the double torsion method is that it allows multiple measurements to be made on the same specimen. After the crack has propagated to a certain extent, the load can be removed and a new measurement taken. This allows researchers to study the relationship between crack propagation and load in detail.<sup>48</sup> The results obtained using the double torsion method have been found to correlate well with those obtained using other methods, such as the single-edge notched beam and compact tension methods. This suggests that the double torsion method is a valid and accurate technique for studying crack propagation in dental composites.

### **7.4. Chevron-Notch test**

The Chevron-Notch test is a widely used fracture mechanics technique that involves a cylindrically shaped specimen with a chevron-shaped notch at one end. Stress is applied to the cylinder, causing the notch to open and initiating a fracture. The notch must be created during the cure process or cut into the specimen after the cure.

Studies of dental composites using the Chevron-Notch method have generally yielded higher values than other testing methods. However, this difference in results may be attributed to the use of test setups with higher-than-acceptable compliance. This increased compliance can cause additional deformation of the test system, which can affect the results.<sup>49</sup>

### **7.5. The short rod test**

Short rod fracture toughness tests are commonly used in dental composite resin research to determine the material's resistance to fracture. These tests require careful consideration of specimen geometry

and test parameters. One significant criteria is the ratio of the notch thickness to specimen diameter.

In the short rod test, it is important to consider the mode of crack propagation. Certain materials may exhibit crack-jumping, in which the load decreases with each crack propagation through the wedge-shaped ligament in the notch region. This type of crack growth is stable until the critical load is reached, at which point the crack propagates unstably, indicating the fracture toughness. Conversely, other specimens may exhibit smooth and continuous crack propagation until the critical load is reached.

The critical load is only reached after the crack has propagated a certain distance from the tip of the chevron notch, regardless of the mode of crack propagation. This can be confirmed by examining the load-displacement record of the test. Furthermore, the fracture surface will display clear regions where stable and unstable crack growth is separated.<sup>49</sup>

## 8. Compressive strength

Compressive tests are fundamental for assessing the mechanical properties of brittle materials such as porcelain, amalgam, and cement, particularly in applications involving heavy loads. These tests determine the material's compressive strength, modulus of elasticity, and failure characteristics by progressively increasing the load on a sample until it fractures or deforms. Compressive testing remains indispensable, even though materials may undergo 'barreling' under load, exposing the specimen's sides to tensile stress. The data derived from these tests are critical for understanding material behavior under stress, thereby facilitating the optimization of material formulations and enhancing their performance in practical applications.<sup>9,50</sup>

## 9. Fatigue strength

Fatigue strength is a critical parameter for assessing a material's durability under cyclic loading conditions. Tests determine this property by repeatedly applying small loads to a material, which ultimately leads to fatigue and fracture. The measurement process typically involves bending or twisting a test sample and recording the number of stress cycles it can endure before failure occurs. We simulate real-world conditions by applying cyclical stress, which

subjects materials to repetitive loads over extended periods. Consequently, fatigue strength serves as an indicator of a material's likelihood of failure under long-term, repetitive stress. Understanding fatigue strength is particularly essential for predicting the lifespan and ensuring the reliability of composite resin materials used in dental restorations.<sup>46</sup>

## CONCLUSION

In dentistry, the large number of materials available complicates the selection process. Mechanical testing is important for evaluating material properties, although it may not fully replicate clinical conditions. Therefore, it is critical to use appropriate testing methods to select materials that will result in durable restorations that are functionally and esthetically compatible with oral tissues.

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