

Academy of Dental Materials guidance—Resin composites: Part I—Mechanical properties

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ABSTRACT

Objective. The objective of this project, which was initiated from the Academy of Dental Materials, was to review and critically appraise methods to determine fracture, deformation and wear resistance of dental resin composites, in an attempt to provide guidance for investigators endeavoring to study these properties for these materials.

Methods. Test methods have been ranked in the priority of the specific property being tested, as well as of the specific test methods for evaluating that property. Focus was placed on the tests that are considered to be of the highest priority in terms of being the most useful, applicable, supported by the literature, and which show a correlation with clinical findings. Others are mentioned briefly for the purpose of being inclusive. When a standard test method exists, including those used in other fields, these have been identified in the beginning of each section. Also, some examples from the resin composite literature are included for each test method.

Results. The properties for evaluating resin composites were ranked in the priority of measurement as following: (1) Strength, Elastic Modulus, Fracture toughness, Fatigue, Indentation Hardness, Wear—abrasion (third body) and Wear—attrition (contact/two body), (2) Toughness, Edge strength (chipping) and (3) Wear determined by toothbrush.

Significance. The following guidance is meant to aid the researcher in choosing the proper method to assess key properties of dental resin composites with regard to their fracture, deformation and wear resistance.

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1. Introduction

Any dental restorative or prosthetic material, as well as natural teeth, must have sufficient mechanical integrity to function in the oral cavity for an extended period of time, hopefully encompassing the lifetime of the patient. Thus, the study of the mechanical properties of these materials is highly clinically relevant. In support of this statement is the fact that one of the leading causes of failure of dental composite restoratives is premature failure due to fracture [1]. Though the occurrence of these failures is not rampant and its frequency may not even be greater than that for dental amalgam [2], enhancement of the fracture resistance properties of these materials is constantly being sought and if attained would likely enhance the longevity of dental composite restorations. While a standard methodology exists for testing of dental composite strength [3], the minimal strengths identified in the standard for various clinical uses does not represent a value determined from engineering design models or extensive clinical testing. Therefore, the mechanical properties of commercial dental composites vary widely, and the general consensus when formulating or developing a new product has basically been “higher is better.”

Regardless, the importance of understanding and fully characterizing the fracture and deformation resistance of dental composites cannot be overstated, and many methods exist for this purpose. These methods have been adopted from test methods developed for other materials, since composites are a relatively “new” dental material. Therefore, the test methods for dental composites have often been adapted to accommodate the unique specimen manipulation and formation needs for a material that is designed to be placed in situ in one state and then converted to its permanent, mechanically more stable state. A review and evaluation of many of these test methods follows in an attempt to provide guidance for investigators endeavoring to study the mechanical properties of dental composites. These test methods have been listed (Table 1) in a way that provides a ranking of the priority of the specific property being tested, as well as the ranking of the specific test methods for evaluating that property. To create this table, focus was placed on the tests that are considered to be of the highest priority in terms of being the most useful, applicable, and supported by the literature. Others are mentioned briefly for the purpose of being inclusive. In all cases, when a standard test method exists, including those used in other fields, these have been identified in the beginning of each section. Also, some examples from the composite literature are included for each test method.

It is also important to point out that because dental composite materials must be molded into the required test shape and polymerized, the quality of the specimen can influence the outcome of the test. It is probably best to ensure that the material is adequately and uniformly cured for the appropriate amount of time for self-cure materials and with sufficient light energy for photo-cured or dual-cured materials. While these conditions may not always be the most clinically relevant, i.e. they do not test for the effects of under-curing, they will provide the most valid test results and will characterize the optimum properties attainable for that material.

2. Guidelines/specific recommendations for measuring Fracture Resistance/Strength/Toughness

2.1. Strength

Strength is not an inherent property of a material. Therefore the recorded value is a function of the geometry and preparation of the specimen, as well as the testing method. Because force is applied in different ways to create internal stresses within a material, what is measured and recorded as strength, or resistance to catastrophic fracture, is dependent upon the conditions of the test. It may seem very logical to test the strength of a material under the typical loading conditions it will face in the oral cavity. To some extent this is useful, but it should be remembered that most external forces on a material will resolve themselves as stresses along various planes within the structure, often leading to tensile and shear stresses even when the specimen is placed under compression. This is especially true in the oral cavity where simple axial loading is almost never encountered due to the anatomy of teeth and the 3-dimensional nature of jaw mechanics. In addition, because the strength of the material is a function not only of its composition, but also of its quality of preparation, i.e. internal porosity, inclusions, surface flaws, etc., it is typically agreed that testing the material under its most challenging mechanical situation may be most instructive and lead to a reduced chance of accepting a material that ultimately fails prematurely due to inadequate strength under complex loading conditions. For this reason, testing of materials in tensile loading is typically considered most appropriate. Because tensile loading is also the most difficult to control experimentally, flexure is often the substituted mode, as it develops tensile, compressive and shear stresses during the test. In any case, one should keep this in mind when preparing specimens for testing to ensure that the material is sound and generally devoid of significant surface and internal flaws. The ISO standard 4049 for dental composites includes a flexure, or transverse, testing modality, and this will be described first. There is evidence from a systematic review on the correlation between flexural strength and clinical fractures of posterior resin composite restorations (31 different materials) that flexural strength correlates moderately with clinical wear but not with bulk fractures [4,5]. Artificial aging by storing the specimens in ethanol or some other solvent prior to breaking them may enhance the correlation with clinical results [5].

2.1.1. Flexure

2.1.1.1. *Transverse bending* (ISO 4049 [3]; ASTM D790-10 [6]; ISO 178-2010 [7]). This is a common test method for the strength of dental composites [8], and can be accomplished several ways based on the selection of loading supports and load applicators, and the geometry of the specimen, in addition to the method of preparing the specimen [9]. Typically, testing is accomplished in 3-point bending (Fig. 1), implying that the specimen is a beam of specific dimensions, supported on two rollers of a specified distance apart (span), and loading from a point source at the top-center of the beam. The test method for dental composites is specified in ISO 4049, and for

Table 1 – Summary of available methods to determine fracture, deformation and wear resistance of dental resin composites, ranked in the priority of the specific property being tested, as well as of the specific test methods for evaluating that property.

Clinical issue/ requirement	Properties	Property rank	Method	Test rank
Fracture and deformation resistance	Strength	1	3-Point bending (ISO 4049)	1
			4-Point bending	2
			Biaxial flexural (ASTM F394-78)	2
			Compression (ASTM D695)	3
			Tensile (ASTM D638)	1
			Diametral tensile (ANSI/ADA Spec 27)	4
			Impact (ISO 179/1961)	2
			Transverse impact (DIN 53 453)	2
			Shear	4
			Shear punch ASTM D732-46)	2
	Toughness	2	Calculated from strength test	1
			Impact (ISO 179/1961)	2
			Transverse impact (DIN 53 453)	2
	Fracture toughnessszr	1	Double torsion	2
			Indentation—Vickers	3
			Chevron notched specimens (ASTM E1304-97)	2
			Single-edge notched beam (ASTM E399-12)	1
			Compact tension (ASTM E399-12)	2
	Edge strength—chipping	2	CK10 instrument (with acoustic emission)	1
			Fatigue strength—Staircase	2
	Fatigue	1	Fatigue resistance—uniaxial loading (ASTM E606/E606M-12)	2
	Elastic Modulus	1	3-Point bending (ISO 4049)	2
			4-Point bending	1
			Biaxial flexural strength (ASTM F394-78)	3
			Compression (ASTM D695)	3
			Tensile	1
Diametral tensile (ANSI/ADA Spec 27)			4	
Indentation(ISO/FDIS 14577-1)			1	
Martens (universal) (E DIN 50359)			1	
Vickers (ISO 6507-1)			3	
Knoop (ISO 6507-1)			3	
Indentation Hardness	1	Rockwell (ISO 2039-2)	3	
		Brinell (ISO 6506-1:200)	3	
Wear resistance	Wear—abrasion/three body	1	OHSU abrasion (ISO TS No. 14569-2)	1
			Alabama generalized (ISO TS No. 14569-2)	2
			ACTA(ISO TS No. 14569-2)	3
	Wear—attrition/contact/two body	1	OHSU attrition(ISO TS No. 14569-2)	2
			Alabama localized (ISO TS No. 14569-2)	3
			Ivoclar-Willytec simulator	2
	Wear—toothbrush	3	Munich-Willytec simulator	2
			Toothbrush/toothpaste(ISO TS No. 14569-2)	3

other plastics in ASTM D790-10. Briefly, the beam should be produced with an appropriate size for the testing supports in order to satisfy the criteria for beam mechanics (i.e. approximately 10% additional material beyond the supports on each end), and should be tested on a rigid test frame of very low compliance (i.e. any deformation of the test frame/jigs during the test can be disregarded and all load is being transferred directly to the specimen). It is emphasized not to stray from these criteria, or the outcomes may not be valid. Testing is usually accomplished to failure, and it is expected that there will be minimal plastic deformation of the specimen. Significant bending of the beam may invalidate the test, and if this occurs, one option is to calculate a yield strength, basically the point at which the load-deflection curve deviates from linearity, rather than reporting a failure load. The ideal test method includes a strain gauge or extensometer to measure the true deformation of the beam, but this is rarely done out of convenience.

When the specimen is relatively rigid, and the test frame is very rigid, the true measurement of deformation during the test is less critical, and one can correlate cross-head motion to beam deflection within a reasonable error to measure the elastic modulus as well as the strength.

Flexure strength can also be tested by loading in four-point bending (Fig. 2), in which the load applicator is not a single point source, but consists of two points separated by a well prescribed distance. The benefit of the 4-point method is that it concentrates the stress over a wider area of the beam, and thus ensures that failure of the beam will occur within this region, a criterion for applying the beam equations accurately [10]. In the 3-point method, it is possible for the beam to fail from a position not directly under the applied load, which may violate the mechanics and lead to potentially erroneous results. In general, evidence suggests that strengths may be higher when tested in 3-point vs. 4-point bending [11]. Oth-

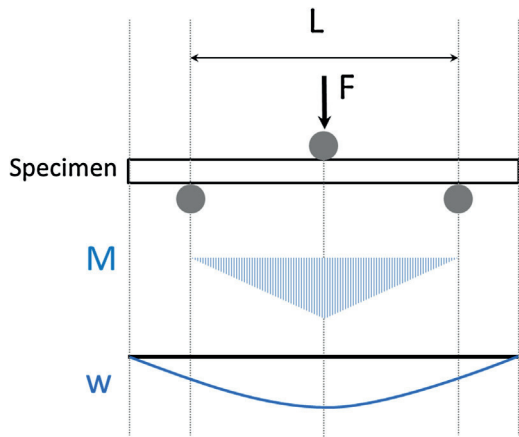


Fig 1 – Schematic of the three point bend test with graphs of bending moment M , and deflection w .

erwise, the two tests are conducted in the same manner and can be performed on specimens of the same dimensions.

2.1.1.2. *Biaxial flexure* (ASTM F394-78 [12]). This is a method where a disk-shaped specimen is placed on top of a support, either a three legged support or on a ring that supports the entire circumference of the specimen, which provides a uniform distribution of tensile stress within the specimen emanating from the bottom surface (Fig. 3). This method is typically used for brittle materials, and has only infrequently been used for dental composites. In part, this is because it is not difficult to conduct transverse flexure tests with composite. However, evidence suggests that the results from the biaxial test correlates with those from 3-point flexure tests and shows less variability in the data [13,14].

2.1.2. *Compression* (ASTM D695 [15]; ISO 604 [16])
 Compression testing is relatively simple in that an axial force is applied at a constant strain rate to a cylindrical specimen, setting up tensile and shear stresses within the material that cause failure (Fig. 4a). While it is logical that restorations should experience compressive forces, as stated previously,

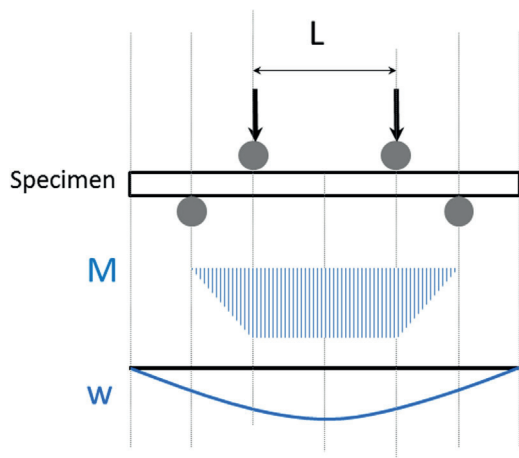


Fig. 2 – Schematic of the four point bend test with graphs of bending moment M , and deflection w .

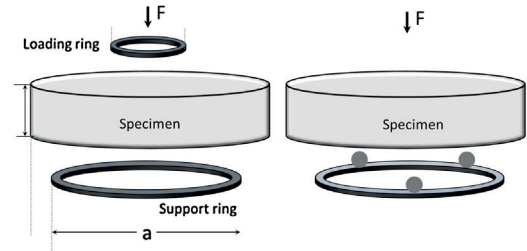


Fig. 3 – Schematic of the biaxial flexure test method, with loading and support ring (left) or with a three legged support (right).

mostly these resolve as more complex stresses within the body of the material and failure is not due to actual compression of the material. Many materials, especially brittle ones, will appear stronger in compression than in tension, because the compression test is generally less sensitive to internal flaws compared to tensile testing. One point to consider is that frictional forces at the surface of the platens where they meet the flat ends of the cylindrical specimen can create complex stresses that violate the principles of the testing method if the specimen itself is not perfectly parallel. To overcome this problem, it is common to place a piece of thin paper over the ends of the specimens to help distribute the forces more uniformly and avoid stress concentrations that cause failure by edge chipping, as opposed to internal stresses. Because of the lack of correlation between compression testing and clinical failure [4,5], and because many lower strength composites undergo significant plastic deformation during such tests, leading to inaccurately high strength values, this test is not particularly recommended for composites, though it is frequently reported [17].

2.1.3. *Tension* (ASTM D638-14 [18]; ISO 527-2 [19])

2.1.3.1. *Uniaxial tensile testing.* As stated previously, it is likely that most dental restoratives fail by tensile stresses set up within their structure due to complex loading of their complex geometries. This would suggest that tensile testing would be the most appropriate testing modality, and there are standards developed for other materials that can be followed. The typical shape of a specimen for tensile testing resembles a dumbbell, or dog-bone, which provides a center region (the gauge length)

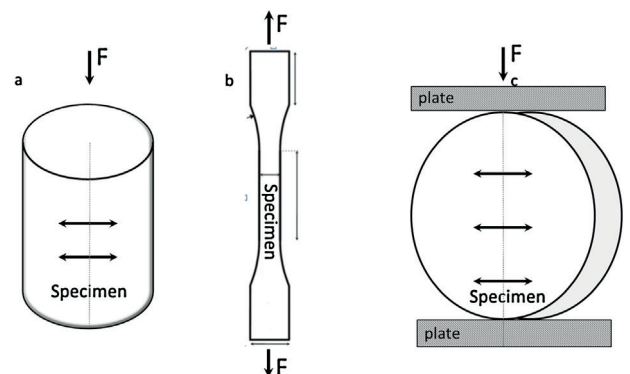


Fig. 4 – Schematic of the compression (a), tensile (b), and diametral tensile (c) method.

with a smaller diameter than the ends of the specimen, thus concentrating the stress in the middle and ensuring that failure will occur there (Fig. 4b). The difficulty with the test is that the specimen must be formed or shaped into the complex dumbbell, and this often leaves irregularities and stress concentrators from which failure occurs, thus nullifying the results of the test. The test therefore is fairly technique sensitive in that specimen alignment is also critical to ensure that loading is truly uniaxial. For these reasons, the tensile test is not very frequently used to measure the strength of dental composites, despite its applicability [20]. However, the need to be vigilant in terms of the testing sequence should not deter researchers from using this test method.

2.1.3.2. Diametral tensile testing (ANSI/ADA Specification #27 [21]). Owing to the difficulties in conducting high quality uniaxial tensile tests for brittle materials, the diametral tensile test (otherwise known as the Brazilian test) was developed and has been used frequently for dental composites [22]. This method is based on the breaking of a disk, or cylinder, resting on its side so that the upper and lower platens of the testing machine supply a force to a line axis across the entire length of the specimen (Fig. 4c). Again, specimen production is critical to ensure that the entire surface of the specimen is uniformly loaded, and for this reason, most studies with this method use a disk which has a shorter length dimension so less material must be in simultaneous contact with the loading and support platens. The diametral tensile test is only considered accurate when the specimen breaks uniformly down the middle, the principle being that as the disk is squeezed along its diameter, tensile stresses are established along its central diameter, pulling the material apart in a tensile manner. The failed specimen should be two halves of the disk or cylinder, and not a distribution of fractured pieces which would suggest a potentially invalid loading condition.

2.1.4. Impact (ASTM D256-10 [23]; ISO 179-1-2010 [24]) Impact testing is different than a test of static strength in that in the latter, the load is applied to a specimen from the point of initial contact and continuing through to failure at a constant loading rate. In impact testing, a specimen, often a beam or plate, is subjected to a sudden, single contact load from an applicator that is swung into or dropped onto the specimen from a specified height (potential energy). The specimen is therefore either broken at a very rapid rate of force delivery, or supports the weight of the load, and impact strength is calculated as the absorbed energy per specimen cross section upon fracture. It is actually a measure of the ability of the material to absorb shock, and in fact may better reflect its toughness, i.e. ability to absorb energy. A common method is the Izod test method, in which a beam is supported on only one side, like a cantilever. In another popular method, the Charpy method, the beam is supported at both ends. Both methods may be used with a specimen with a precise notch to ensure failure at a specified position, and also to determine the notch sensitivity, or brittleness of a material. The test is not commonly used for dental composites [25].

2.1.5. Shear (ASTM D732-10 [26])

The shear test is likely most suited to materials that are not brittle, but have ductile characteristics. Shear denotes a material's ability to resist sliding type stresses, where the material is sliding against itself in the direction of its length, rather than across itself. However, a punch test exists for plastics and composites in which a punch tool is driven through a plate or sheet of material and the force is divided by the cross-sectional area to determine a shear punch strength. This method has been used with dental composites [27,28]. Shear strength is not typically measured as a bulk property of a material, but rather is typically used to assess the adhesion between two different surfaces. A modification of a simple shear test has also been used to test the bond strength of one material to another by inserting or curing the second material within a hole in the first, and then "punching" out the circular specimen in the center by loading through a piston to determine the shear bond strength [29].

2.2. Fracture toughness

Fracture toughness differs from strength, in that it is an inherent property of a material, and therefore its value should be independent of testing modality or specimen geometry. Fracture toughness is a measure of a material's resistance to the propagation of a crack from a preexisting flaw of known size and infinite sharpness, i.e. a pre-crack. Because most materials contain flaws, load application typically causes failure from such a flaw, and strength properties are reduced as compared with those measured from perfect surfaces. As the flaw characteristics are difficult to determine a priori in strength tests, the fracture toughness test requires a specimen be produced with a specified flaw from which failure will be generated through crack propagation. For dental composites, this flaw can be produced by being molded into the specimen during curing, or can be cut into the specimen after it has been cured. The ideal and most correct method for fracture toughness testing is to load a slightly notched specimen in fatigue to create a true infinitely sharp "pre-crack". But this is often very difficult to do with brittle materials, especially those of the small size desired for testing dental composites, because the cracks are difficult to control once they are created and the specimen typically fractures during production. Therefore, alternative approaches for producing the crack initiating flaw, such as molding a very sharp notch with a razor blade or sharpening an existing notch with a sharp blade and an abrasive, have become common practice.

Because fracture is one of the primary failure modes for dental composites, this property is highly relevant for characterizing them. A systematic review has revealed a weak positive correlation (correlation coefficient $\rho = 0.34$) between fracture toughness and clinical fractures of posterior resin composite restorations (31 different materials) [5]. Others studies have shown correlations of fracture toughness and marginal breakdown and wear of composites [30,31]

There are many ways to test this property, as described below (Fig. 5). The choice is often made based on convenience. However, it is important to note that accurate and reliable testing is based on having proper equipment of very low compliance such that all deformation occurs in the spec-

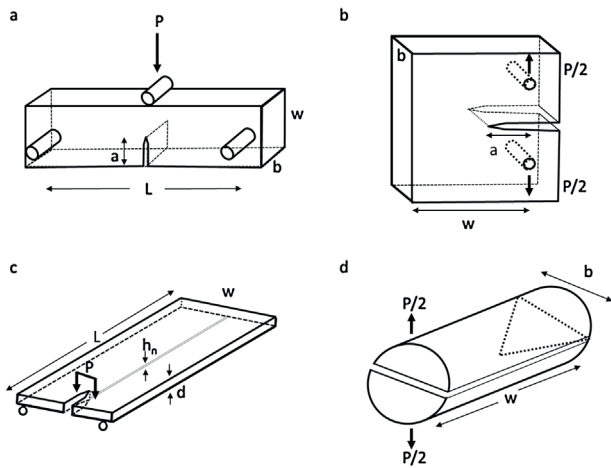


Fig. 5 – (a) Specimen geometry for the determination of fracture toughness by the single-edge notched method. (b) Specimen geometry for the determination of fracture toughness by the compact tension method. (c) Specimen geometry for the determination of fracture toughness by the double torsion method. (d) Specimen geometry for the determination of fracture toughness by the short rod method.

imen and is not lost to the testing jig or loading frame. This requirement is based on the fact that the equations for the calculation are based on the material being loaded in a state of linear elasticity. In fact, compliance is typically measured and then accounted for within the test to provide the most accurate result for fracture toughness, which is defined as the stress intensity factor, K . Because the test may be conducted in a number of ways, the stress intensity factor may denote a failure in plane strain (I), plane stress (II), or a combined mode. When the test is compliant with all of the requirements for minimal plastic deformation, i.e. brittle failure, the stress intensity is denoted as critical, designated by a “c”. Thus, the most common test for fracture toughness is completed by loading a specimen with a precisely measured pre-crack in tension and obtaining a load to failure to produce a value for K_{Ic} . Materials that have very high fracture toughness are typically not brittle, and rather fail in a ductile manner. The converse is true of brittle materials, and dental composites tend to fall into this latter category, typically having relatively low fracture toughness. However, composites do undergo some plastic deformation during testing, the extent being dependent upon the material’s characteristics, i.e. filler load predominantly, and this must be considered when testing in order to ensure that the reported value is truly accurate and valid. Sometimes a value for K will be reported, without the subscripts, because the validity of the test method cannot be confirmed. In such cases, it is possible to apply alternate analysis methods that account for significant amounts of plastic deformation, such as the J-integral method.

2.2.1. Single-edge notch—3-point bending (SENB) (ASTM D5045-14 [32]; ISO/NP 13586 [33])

A common method for testing fracture toughness of dental composites is to load a sharply notched beam in 3-point

bending, thus imposing a tensile force upon the notch (i.e. the pre-crack), causing fracture through the thickness of the specimen (Fig. 5a). The beam should have dimensions appropriate to the linear elastic fracture mechanics requirements, in which the length of the loading span is about $10\times$ greater than the thickness of the specimen. The critical assumption in this test is that the tip of the notch is infinitely sharp, as a true pre-crack would be. For many materials, such as metals, specimens are cycled in tension to produce a crack of measureable length from the tip of the notch, thus creating a pre-crack. As previously discussed, this is difficult to impossible in brittle materials. It is possible to do with composites, but the specimen needs to be very large, and this is typically cost prohibitive. Thus other attempts have been made to create true pre-cracks of infinite sharpness, such as sawing sharp notches, sharpening the tip of notches with a very sharp blade or abrasive paste, or tapping a blade into the notch to propagate the crack. The literature is full of studies using these varied techniques, and likely the differences in the extent to which the methods truly comply with the requirements of the test method account for the sometimes highly varied outcomes reported.

2.2.2. Compact tension (ASTM D504-14 [32]; ISO/NP 13586 [33])

The compact tension method (Fig. 5b) is similar to the 3-point bending method in that a tensile force is applied across a notch, but the specimen is typically in the form of a plate and the load is applied from holes in the top and bottom of the plate to produce tensile opening of the notch. This method typically requires the measurement of the opening of the crack using strain gauges or extensometers to accurately determine the distance the notch is opening for a given load. When precisely controlled, this test method can be used to provide more extensive data than the simpler beam bending method. It is possible to determine characteristics such as “R-curve behavior”, where the fracture toughness actually increases as the length of the crack increases. In other words, the material becomes tougher as the crack grows, and is more resistant to catastrophic fracture. This toughening occurs due to specific energy dissipating mechanisms that become active within the material during loading and cracking. The results from this method have been shown to correlate fairly well with those from the single-edge-notched beam [34].

2.2.3. Double torsion (ASTM C1421-10 for ceramics [35])

The double torsion method (Fig. 5c) is described by placing a plate specimen containing a groove on its bottom surface within which the crack will propagate during the test [36]. The plate is placed upon a set of parallel rods (rollers) running in its length direction, and load is applied near one end of the plate via two point surfaces placed on opposite sides of a precisely made notch to produce tension and crack propagation from the end of the notch. Once a critical load is reached, the crack will propagate down the groove and is visible and measureable on the top surface. The crack will stop when the critical stress is no longer present, thus allowing one to make several measurements on the same specimen by propagating the crack down the plate via multiple loadings and measurements of crack propagation vs. load. Another interesting aspect is that

even if the notch is not infinitely sharp, the first load condition produces a fresh pre-crack, and each crack then serves as the pre-crack for the next test. For dental composites, these values have been shown to correlate fairly well with those obtained from the single-edge-notched beam and the compact tension methods [34].

2.2.4. Chevron notch (ASTM E1304-97 [37])

The chevron notch method utilizes a cylindrical specimen that contains a chevron notch at one end, and the fracture is produced by loading the cylinder such that the notch is opened in tension (Fig. 5d). This method requires the notch to be either formed during curing or cut into the specimen after curing. The values from this method for dental composites have typically been shown to be higher than those obtained from the previously mentioned methods, though this may be due to the use of a testing setup of higher than acceptable compliance, allowing for extraneous deformation of the test system [34].

2.2.5. Indentation

A common, though controversial, method for measuring fracture toughness of ceramics is the indentation method, typically with a Vicker's pyramid indenter [38,39]. The object of the method is to create an indent on a highly polished surface, from which cracks will propagate from the tips of the indentation. This will only occur if the material displays brittle characteristics, as the cracks relieve the energy absorbed by the material from the indenter, the length being related to the toughness of the material, i.e. shorter lengths or less propagation for tougher materials. However, if the material deforms plastically to absorb the stress, crack propagation will not occur. Thus, this test method is typically not amenable to dental composites, which typically have sufficient plastic deformation and do not generate cracks during the indentation.

2.3. Edge strength—chipping

Dental materials, like resin composites, amalgam and porcelain, often suffer from chipping or fracture at the margins or at unsupported edges. This is a difficult characteristic to quantitate, but some have attempted to do this using an edge strength test where an indenter is applied near to the edge of a specimen, and the force required to cause a chip of material to be fractured off is quantitated [40–43]. There is no standard test method, but some have used a piece of equipment made specifically for this purpose to make indents at specific distances from the edge of a specimen and measured force to cause fracture. There is essentially a linear relationship between force and distance from the edge, so the edge strength is chosen as the maximum force to create a chip at a distance of 0.5 mm.

2.4. Fatigue

Fatigue may be the most important property for dental materials that are exposed to periods of cyclic loading while chewing food. It is likely that failures occur over time due to the accumulation of damage produced by cyclic forces that do not exceed the fracture strength. Fatigue is a complex phe-

nomenon, and many of the critical variables affecting fatigue of dental composites have recently been reviewed [44]. It is likely that cracks propagate from existing flaws, not catastrophically to failure due to a single loading event, though this is of course possible, but due to extension of the crack and the formation of other localized damage until the material can no longer support the loading conditions. Fatigue is more likely to be initiated from sites subject to high stress concentrations, such as sharp edges, grooves, surface and internal flaws, and other imperfections. Fatigue testing is generally fairly labor intensive and expensive in that it requires a lot of time and material to completely characterize it. For this reason, it is not as common as measuring strength or toughness of dental composites.

2.4.1. Fatigue resistance/limit (ASTM D7791-12 [45]; ASTM D7774-12 [46])

The most common method for measuring fatigue is to take a material, typically in a dumbbell shape in order to concentrate stress at the smaller diameter region in the center of the specimen away from the grips holding the specimen, and cyclically load it in tension at a specified frequency that represents its usage condition. The test can also be accomplished with beams in bending. The test is begun with a high stress (S), near the tensile stress of the material, and proceeds until the material fails by fracture, recording the number of cycles required. A new specimen is then tested at a slightly lower stress and again tested until failure. This is continued, recording the number of cycles to failure (N), and a curve is plotted of stress vs. log of cycles to failure (S - N curve). At some level of stress, the specimen can be cycled for an infinite period of time, perhaps 10 million cycles or more, without failure. This stress is denoted as the fatigue resistance, endurance limit, or the fatigue strength. One can then design a material to function at stress values that never rise above this value to ensure that it does not fail under fatigue conditions. For example, in the dental situation, chewing is typically performed at a frequency of 1–2 Hz (cycles per second), or 60–120 chews per minute. To determine the fatigue resistance, which may occur at more than one million cycles, would require cyclic loading of a single specimen for one to two weeks. In addition, many additional specimens would need to be loaded for various periods of time to produce the entire S - N curve, thus likely requiring one month to complete one material. Some have investigated the fatigue resistance of dental composites by testing to a maximum number of cycles, such as 100,000 [47]. While this may be the optimal way to describe fatigue behavior, the expense has caused the development of other methods that have been used for dental composites and other materials. The staircase method is an example.

2.4.2. Fatigue strength—staircase method

In the staircase method (Fig. 6), a specimen, typically a beam, is tested in 3-point or 4-point bending beginning at some stress level approaching one-half of the fracture strength of the material as determined from a typical static test as described above [48,49]. The specimen is cyclically loaded at an appropriate frequency, such as 1–2 Hz, for a pre-determined number of cycles, such as 5000–10,000. The choice of cycles is somewhat arbitrary, but should be enough to ensure that the specimen

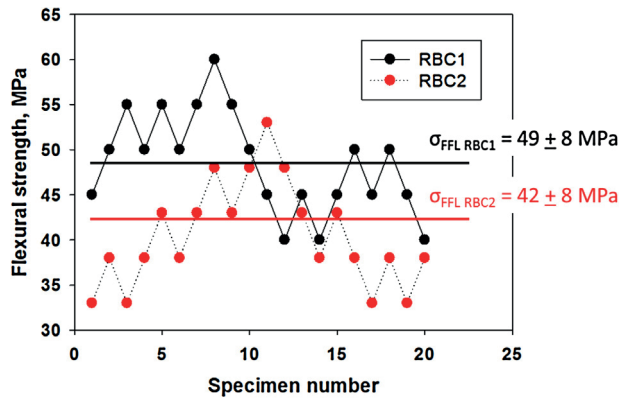


Fig. 6 – Example of the stair-step method for two resin-based composite (RBC) formulations (σ_{FFL} = flexural fatigue limit).

is subjected to enough cyclic stress to potentially generate internal damage. If the first specimen survives the number of applied cycles, a new second specimen is made and tested for the same number of cycles at a stress level that is raised by some amount, perhaps 5% higher. If the first specimen fails, the load level is reduced by the same amount for the second specimen. Testing proceeds in this way with about 20–30 specimens, raising and lowering the stress based on surviving or failing at the tested load in an effort to “focus in” on the fatigue strength of the material. This is calculated with an appropriate statistical method. Because loading is accomplished for a specific number of cycles, each specimen is tested for a maximum of an hour or so when 1–2 Hz is used. Thus, this test method is more economical than the fatigue resistance test, is easier to perform because a simple beam specimen can be used, and provides a value for fatigue strength, which for dental composites is typically about 55–65% of the static flexure strength [50].

3. Guidelines/specific recommendations for measuring the Elastic Modulus

An elastic modulus, or modulus of elasticity, is defined as an object or substance’s resistance to being deformed elastically (i.e., non-permanently) when a force is applied to it. The elastic modulus is calculated as the slope of the stress–strain curve in the elastic deformation region. Besides, an indentation modulus may be assessed from indentation-depth techniques, as the slope of the tangent line adapted to the beginning (at maximum force) of the non-linear indentation depth curve upon unloading [51]. For resin composites, a good correlation was identified between indentation and elastic modulus (three-point bending test) [52], both moduli correlating well with the total inorganic filler load of the material [53].

3.1. Elastic Modulus

A wide variety of mechanical tests involving a stress–strain curve recording are used to determine the elastic modulus of resin composites.

3.1.1. Tensile

Ideally, tensile tests should be used (Fig. 4), but no specific standards for dental materials are available, since standard dumbbell-shaped test specimens commonly used for plastics [19] are too large and expensive to machine for resin composites. Moreover, tensile properties are known to vary with specimen preparation and with speed and environment of testing. Consequently, precise comparative results are possible only if these factors are carefully controlled and indicated in standards. The fixture of specimens is also difficult and special clamping grips, adapted to a small specimen size, are needed. Bonding composite samples to jigs with cyanoacrylates or other adhesive is not recommended. For additional information about this test also consider Section 2.1.3.1. These experimental difficulties have motivated researchers to use alternative test methods.

3.1.2. Flexure (ISO 4049 [3]; NIST No. 4877 [54])

The most common and highly rated tests to evaluate the modulus of elasticity for resin composites involve three- and four-point loading (Figs. 1 and 2) of rectangular bar specimens ((2 × 2 × 25) mm, ISO 4049 [3] or (2 × 2 × 18) mm, NIST No. 4877 [54]). The latter is usually further specified by a description of the distance from the outer support points and the inner points, such as $\frac{1}{4}$ or $\frac{1}{3}$ for four-point loading. Direct observations of the deflection and the load are recorded, allowing one to determine the stress-strain relationship. The small size, low costs, and easy preparation of a flexural specimen account for its popularity, but there are distinct drawbacks as well. The fundamental differences between both testing procedures are the location of the maximum bending moment and maximum axial fiber stresses. The maximum stress occurs directly below the loading point in three-point loading and is spread out over the area between the loading points in the four-point system. The bending creates a stress gradient in the specimen and only a small volume is exposed to high tensile stress. The specimens are very sensitive to edge or surface defects. The test is deceptive in that it appears simple to set up and conduct, but misalignments and experimental errors can easily falsify final results.

When comparing a three- and a four-point loading test, the elastic modulus was proven to be less sensitive to the test procedure than the flexural strength [55]. While the strength measured in a three-point testing procedure is higher compared to a four-point testing procedure, no difference among 3 and 4-point bending were identified for elastic modulus (in denture-base polymers [55]), basically because its calculation is done by use of a tangent to the initial slope of the load-deflection curve. It must, however, be considered, that the elastic modulus of polymers increases with decreasing temperature and with increasing strain rate. Therefore, both conditions must be carefully controlled and standardized. It is recommended to carry out the test in a water bath maintained at 37 °C or at least at a constant and measured room temperature. As for the strain rate, there is a limit in most polymers above which they are brittle. This limit was estimated at around 10% per minute for polymethyl methacrylate (PMMA) at room temperature. A crosshead speed of 5 mm/min was determined to correspond to a strain rate of 3% per minute in denture-base polymers [55]. For resin materials, crosshead

speeds varying between 0.1 mm/min and 5 mm/min are often used. Besides, the span-to-depth ratio (refer to ISO 4049 [3] or NIST No. 4877 [54]), the light exposure (radiant exposure, exposure time, exposure procedure, curing unit characteristics, etc.) as well as the aging time and procedure must be clearly defined and indicated. A direct comparison of materials is only possible within identical measuring conditions as well as specimen sizes and preparation methods.

3.1.3. Other loading conditions

Beside the described methods, the elastic modulus may also be calculated from the elastic deformation region of stress-strain curves determined under other loading conditions (consider therefore Section 2.2), such as bi-axial flexural [56] (BFS-piston-on-three-ball; -ball-on-ring or -ring-on-ring, ASTM F394-78 [12]), compression [15] or diametral compression (Brazilian disk test) (Figs. 3 and 4) [21]. These types of tests are, however, less popular. In all tests, specimen alignment plays an important role in achieving even load distribution, which contributes to the consistency of the results. Test specimen preparation, conditioning, dimensions, and testing parameters must be clearly specified.

3.2. Indentation modulus (ISO/FDIS 14577-1 [57], ASTM E2546-07 [51], [58])

The indentation modulus determined by depth sensing methods is ranked in its importance as high as the elastic modulus determined in a bending test and might allow for a rapid and precise assessment of the material's resistance to deformation. While the needed equipment is expensive, the specimen preparation requires a surface of low roughness, ideally smooth polished and even (the maximum deviation in parallelism should not exceed 0.1 mm in 50 mm) [57]. A manual polishing is consequently insufficient and automatic polishing devices, allowing for good parallelism, are recommended. The specimen's surface must be completely free of any lubricants, polish pastes, waterproof pen marks or any visible surface flaws. The distance between the centers of two adjacent indentations shall be at least 20 times the indentation depth and the specimen thickness needs to be about 10 times the indentation depth. Test force must always be indicated and must be high enough to induce an indentation larger than the filler size, to avoid inhomogeneity of the results due to large filler particles, filler agglomerates or matrix rich areas. The indentation modulus can be determined either in a static or a dynamic test procedure.

3.2.1. Static

A static approach involves an automatic or semi-automatic universal-hardness device. The test procedure may be carried out under force- or indentation depth-control. The measuring principle involves forcing a diamond indenter into the surface of the specimen (usually a Vickers pyramid, or a Berkovich indenter, but also Knoop [59], spherical or cube corner indenters [60] are used). The test force and the indentation depth are measured during the test procedure at increasing as well as at decreasing force. The result of the test is the relation between test force and the relevant indentation depth. The load is increased and decreased at a constant speed (or force).

The indentation modulus is determined from the slope of the tangent line adapted to the beginning (at maximum force) of the non-linear indentation depth curve upon unloading [57,58]. A good correlation between indentation modulus and the elastic modulus determined in a three-point bending test was identified for resin composites [52].

3.2.2. Dynamic

A dynamic approach (dynamic mechanical analysis, DMA) measures the properties of materials as they are deformed under periodic stress. Specifically, in DMA a variable sinusoidal stress is applied, and the resultant sinusoidal strain is measured. Resin composites are viscoelastic materials. If the material being evaluated is purely elastic, the phase difference between the stress and strain sine waves is 0° (i.e., they are in phase). If the material is purely viscous, the phase difference is 90°. In viscoelastic resin composites, this phase difference, together with the amplitudes of the stress and strain waves, is used to determine a variety of fundamental material parameters, including storage modulus, loss modulus, complex modulus, and tan delta (the loss factor) [61].

4. Guidelines/specific recommendations for measuring hardness

The resistance of a solid to local deformation characterizes the general concept of hardness. Hardness is a result of a defined measurement procedure and is not an intrinsic material property. Basically, an indenter of a specified shape is pressed into the surface of the material to be tested under a specific load for a definite time interval, and a measurement is made of the size or depth of the indentation after the force has been removed.

Indentation tests or scratch tests have been used for more than one century to determine the hardness of materials [62]. The ease with which the hardness test can be carried out has made it one of the most common methods of characterization for resin composites. The traditional method to test hardness – Brinell [63], Knoop [64], Rockwell [65] or Vickers [66] – involves loading a hard indenter against the material (Fig. 7). With materials that behave in an elastic/plastic manner, the indenter leaves an indentation behind after it is withdrawn. Hardness is defined as a quantitative measure of the deformation resistance and is calculated as the maximum applied load divided by the projected area of contact.

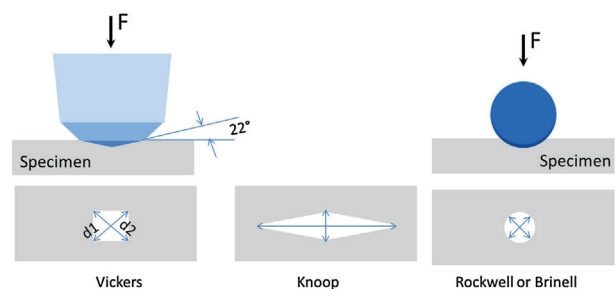


Fig. 7 – Indent shapes and their imprints in hardness measurements: Vickers, Knoop, Rockwell and Brinell Hardness.

Within these classical hardness measurements, only the plastic part of the indentation process is considered. The method was developed primarily for metals, but also indentation hardness measurements on relatively brittle materials are possible, considering that under localized indentation, the stress distribution around the indenter is equivalent to a hydrostatic pressure on which is superimposed a shear stress. In these circumstances brittle fracture is often prevented [62]. Since the deformation of resin composites is a mixture of plastic and elastic components, an extension to depth sensing hardness measurement approaches is recommended. Therefore, a dynamic measuring principle is applied with simultaneous recording of the load and the corresponding penetration depth of the indenter. The plastic as well as elastic part of the indentation can be separated from the analysis of the load-displacement data [60,67,68].

Hardness measurement can be defined as macro-, micro- or nano-scaled according to the forces applied and displacements obtained (ISO 14577-1:2015 [57]): macro range: $2\text{ N} \leq F \leq 30\text{ kN}$; micro range: $2\text{ N} > F$; $h > 0.2\text{ }\mu\text{m}$; and nano range: $h \leq 0.2\text{ }\mu\text{m}$ (where F =Force and h =indentation depth). Attention is drawn to the fact that the micro range has an upper limit given by the test force (2 N) and a lower limit given by the indentation depth of $0.2\text{ }\mu\text{m}$. For the nano range, the mechanical deformation strongly depends on the real shape of the indenter tip and the calculated material parameters are significantly influenced by the contact area function of the indenter used in the testing machine. Therefore, careful calibration of both instrument and indenter shape is required in order to achieve an acceptable reproducibility of the materials parameters determined with different machines. Besides, the specimen's surface must be smooth to permit a regular indentation shape and good visualization for measurement, and must be placed perfectly perpendicular to the indenter.

4.1. Indentation Hardness

The indentation hardness methods are described below in the order of their frequency of use for resin composites.

4.1.1. Vickers hardness ISO/CD6507-1 [66]

The Vickers hardness test method (Fig. 7) consists of indenting the test material with a diamond indenter, in the form of a pyramid with a square base and an angle of 136° between opposite faces subjected to a test force of between 1 gf and 100 kgf. The full load is normally applied for 10–15 s. The two diagonals of the indentation remaining in the surface of the material after removal of the load are measured using a microscope and their average calculated. The Vickers hardness is the quotient obtained by dividing the load by the square area of indentation. The Vickers hardness number should be reported together with the test force and the dwell time. Adequate sample preparation and indentation closeness must be considered, as presented above in paragraph 3.2. The indentations should be as large as possible to maximize the measurement resolution. The test procedure is thus subject to problems of operator influence on the optical reading of the diagonals. Modern devices allow for automatic measurement of indentations.

4.1.2. Knoop hardness (ISO 4545-1 [64])

In a Knoop hardness test a predetermined test force is applied with a pyramid-shaped diamond indenter (with angles of 172.5° and 130° between the opposite edges at the vertex) for a specified dwell time (Fig. 7). The time for the initial application of the force should not exceed 10 s, and the test force is maintained for 10 s–15 s. The Knoop hardness value is proportional to the test force divided by the projected area of the indentation. Compared to a Vickers indenter, the indenter used on a Knoop test is more elongated in shape. While in the Vickers hardness test the indentation length on the vertical and horizontal axes are measured and averaged, the Knoop method only uses the long axis. This measurement is then converted to a Knoop hardness number using a chart. The width of the Knoop indentation can provide more resolution for measurement and the indentation is also more superficial. The Knoop method is commonly used when indentations are closely spaced or very near the edge of the sample. Load and dwell time must be indicated. A strong correlation ($r=0.91$) was identified in resin composites between Knoop and Vickers hardness [69].

4.1.3. Rockwell hardness (ISO2039-2 [65])

The Rockwell method (Fig. 7) measures the permanent depth of indentation produced by a force/load on an indenter (either a 120° diamond cone with a 0.2 mm radius spherical tip or a ball indenter of a specified diameter). A two-step application of force is required: first, a preliminary test force (commonly referred to as preload or minor load) is applied to a sample, maintained for a duration that does not exceed 3 s, and an indenter depth reading is recorded. This load represents the zero or reference position that breaks through the surface to reduce the effects of surface finish. After the preload, an additional load (major load) is applied to reach the total required test load. This force is held for a predetermined amount of time (dwell time) to allow for elastic recovery. This major load is then released and the final position is measured against the position derived from the preload, i.e. the indentation depth variance between the preload value and major load value. This distance is converted to a hardness number.

4.1.4. Brinell hardness (ISO 6506-1:200 [63])

The Brinell method (Fig. 7) applies a predetermined test load to a carbide ball of fixed diameter which is held for a predetermined time period and then removed. The permanent width of indentation is then measured across at least two diameters—usually at right angles to each other and these results averaged. Test forces range from 500 to 3000 kgf. The Brinell hardness value is proportional to the test force divided by the surface area of the indentation. The greatest source of error in Brinell testing is, as for the other indentation methods, the measurement of the indentation; the method is thus somewhat subjective and operator dependent. Standardized, automatic optical Brinell scopes using computers and image analysis to read the indentations are available and might be considered. Load and dwell time must be indicated. The use of Brinell hardness tests in testing resin composites is less frequent.

4.2. Instrumented indentation (ISO/FDIS 14577-1 [57]; ASTM E2546-07 [51])

Compared to indentation hardness, instrumented indentation methods are depth sensing hardness measurements that provide the ability to measure the indenter penetration under the applied force throughout the testing cycle. They are therefore capable of measuring both the plastic and elastic deformation of the material under test. The instrumented indentation (ISO/FDIS 14577-1 [57], ASTM E2546-07 [51]) incorporates a method recording and evaluating the loading and unloading cycle while the test procedure may be carried out under force- or indentation depth-control. Therefore, a full set of data, including force, depth and time, is recorded with a qualified rate. The test force-indentation depth curve is used to determine the hardness under load, thus, including the elastic part of indentation work, and, further, it derives additional material characteristics. Load and penetration depth of the indenter can be continuously measured during the load-unload hysteresis. Instrumented indentation is therefore able to determine both the indentation hardness (H_{IT}) and the Martens hardness (HM) of a material.

4.2.1. Indentation hardness, H_{IT}

The indentation hardness (H_{IT}) is equivalent to the Meyer Hardness and similar to the Vickers Hardness when a Vickers indenter is used and hardness is calculated from the test force divided by the projected area of the indenter in contact with the test piece at maximum load. In many materials, the area of contact under moderate forces is a good approximation to that which remains when the indenter is fully unloaded and removed from the surface. In such cases, the indentation hardness is very similar to Vickers hardness. The difference is that the indentation area is calculated from measured displacement data instead of optical measurement and the Vickers scale assumes a perfect geometry, whereas instrumented indentation uses a measured shape of the indenter that makes allowance for tip rounding and other common deviations.

4.2.2. Martens Hardness, HM

The Martens Hardness (also known as Universal hardness, HU) is defined as the test force divided by the apparent area of indentation under the applied test force. Some software allow for a conversion of the Martens Hardness to the more familiar Vickers hardness.

5. Guidelines/specific recommendations for measuring wear resistance

Intraoral wear occurs by different mechanisms. When teeth come into contact without a food bolus or any other intermediary, this is called two-body or attrition wear [70]. When a person chews on food items or brushes their teeth with a toothbrush and toothpaste, three-body or abrasive wear results. Buccal and lingual tooth surfaces are mainly exposed to mechanical oral hygiene procedures causing abrasive wear, while the occlusal surfaces are subject to both attrition and abrasive wear, which occur almost simultaneously or in short

subsequent episodes. Another phenomenon is described as adhesion wear and occurs when two solid surfaces slide over one another under pressure. Surface projections or asperities are plastically deformed and eventually joined together by the high local pressure. In the process, material may be transferred from the artificial material on one tooth to the artificial material on the opposing tooth or to the tooth enamel. Likewise, a similar transfer of material may happen on the proximal surfaces of adjacent teeth. Fatigue wear occurs when large portions of dental hard tissues or restorative material chip off. If this occurs on the cervical part of the tooth, the term “abfraction” is used.

Resin composites show a particular wear pattern because many characteristics associated with their composition directly influence their wear resistance. Optimally, the loading force is completely transferred from the matrix to the filler particles. The size, shape and hardness of the fillers, the quality of the bonding between fillers and polymer matrix, and the polymerization dynamics of the polymer all have an effect on the wear characteristics of a dental composite [71]. The composition of the composite influences the physical and mechanical properties, such as flexural strength, fracture toughness, Vickers hardness, modulus of elasticity, curing depth, etc. [72], and these properties, in turn, may influence wear. When there is direct contact between composite and an antagonist, the wear pattern is mostly a combination of attrition/abrasive wear and microfatigue, and the friction coefficient and the surface roughness are determining factors for the wear rate. Thus, the size and volume of the fillers affect the wear rate. A low elastic modulus in the material leads to larger contact areas and consequently to lower pressures. Large filler particles, on the other hand, are associated with high friction coefficients and lead to high internal shear stress in the polymer matrix. The latter in particular was most evident in the early composites of the 1980's, which contained large fillers and showed excessive wear in the posterior region in a short period of time [74]. This was clinically visible as loss of contour.

To better study this phenomenon, researchers developed methods to predict wear through laboratory tests [73]. In 2001, the International Organization for Standardization (ISO) published a Technical Specification on “Guidance on testing of wear”, describing 8 different test methods of two- and/or three-body contact [74] (see Table 1). Another ISO Technical Specification covers the wear caused by tooth brushing [75]. However, contemporary restorative materials, including composites, have been shown to be very resistant to tooth brushing both in vitro [76,77] and in vivo [78].

The different two- and three-body test methods vary with regard to load, number of cycles and their frequency, abrasive medium, type of force actuator, sliding movement, etc. Many of these tests fail to define a qualification protocol for the test equipment or a validation procedure for the test method, which is run in conjunction with the equipment. Both qualification and validation, however, are indispensable prerequisites for a test to become a standard laboratory test [79]. The test equipment must operate within acceptable and reproducible limits and tolerances. Especially, the reproducibility of test results is a prerequisite to fulfil the criteria of a validated test method. Otherwise, it is always necessary to repeat wear tests with a reference or standard material, which is time-

consuming and reduces the significance and validity of the test method. Some of the methods listed in Table 1 do not allow for the generation of reproducible results. A systematic review showed that the wear rate results for the same composite subjected to the same wear method and using the same wear parameters varied between 30% and 70% [80]. This variability is probably attributable to the fact that employed devices were not appropriate for the intended use.

At present, only three chewing simulators which use two axes of movement (vertical and horizontal) fulfil the criteria of a qualified device: the Willytec chewing simulator [80,81], the MTS chewing simulator [82,83] and the Bose ElectroForce 3330 Dental Wear Simulator. For the latter, however, no studies have been published. Some institutes developed their own systems such as the OHSU machine [84], the Alabama machine [85], the Zurich machine [86], the Regensburg simulator [87], the ACTA machine [88] and the BIOMAT simulator [89]. More recently, a complex system with six actuators has been developed at the University of Bristol: the Dento-Munch Robo-Simulator [90]. This simulator tries to mimic the entire process of movements of the lower jaw by using a Stewart platform.

As all simulators and wear methods follow different approaches because they are based on different operational concepts, the results cannot be compared. This has been shown in a blind round robin test (the test centers did not know which material they were testing) on 9 materials (7 composites, 1 ceramic, 1 amalgam) which were assessed with 6 different methods (ACTA, Alabama, Ivoclar, Munich, OHSU, Zurich) [91].

A device that is used to test dental materials for wear should ideally have the following features:

- Force and force impulses should be reproducible and adjustable in the range of 20 N–150 N. Preferably, calibration should not be necessary before testing a material.
- A lateral movement of the stylus should be integrated in the system to be able to test the material for microfatigue.
- Constant water exchange should also be integrated to remove abraded particles from the interface between stylus and material.
- All movements should be computer-controlled and adjustable.

One of the best compromises in terms of cost and efficiency is the commercially available two-axis chewing simulator, Willytec (SD Mechatronik, Germany). Systematic tests have shown reproducibility of results, easy modification of test parameters, costs and maintenance are all in an acceptable range. This chewing simulator operates with dead weights that are put on vertical bars which are descended with a servo engine. Additionally, a lateral movement can be integrated into the wear method. Both the vertical and horizontal axes are computer-controlled. Speed and length of all movements can be varied and the simulator can also be used for fatigue testing. The chewing simulator comes with 2/4/8 chambers so that 2/4/8 specimens can be tested at the same time. The simulator can optionally be combined with simultaneous flooding and evacuation of each chamber with water of different temperatures (i.e. thermocycling).

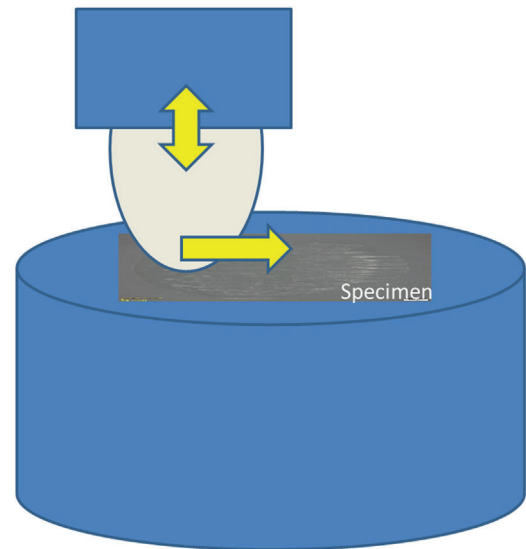


Fig. 8 – A conical shaped ceramic stylus with enamel-like properties moves on a flat composite specimen with a lifting movement and standardized force (eg. 50 N) and speed (eg. 20 mm/s) creating a wear facet.

The following wear influencing factors should be taken into consideration:

Surface roughness of specimen: The surface roughness of the specimens prior to carrying out the wear should be standardized, although the influence appears to be small [92].

Number of specimens: The scattering of the results expressed by the coefficient of variation (mean divided by standard deviation) determines the number of specimens required to statistically differentiate between materials. The variability of the test results mainly reflects the quality of the wear testing device but also the quality of the specimens can contribute to the variability. The more robust a device is constructed and the more reproducible test parameters such as force, speed of stylus, etc. can be maintained, the lower is the variability [80].

Loading force: Higher forces produce more wear. However, the relationship does not seem to be linear. There might even be a certain threshold at which an increase in the loading force no longer results in an increase in wear [80,93].

Type of stylus material: Enamel should be the material of choice due to its relevance. But it is not possible to standardize the composition of a biological substrate, and extracted teeth are often in short supply, which makes it necessary to employ alternative materials. For instance, the pressable leucite-reinforced ceramic IPS Empress is a suitable material for this purpose and generates a similar wear rate as an enamel stylus of the same shape (Fig. 8) [94].

Size and shape of stylus: A pointed stylus produces more wear than a ball-shaped stylus as a ball-shaped stylus has a larger contact area between stylus and material than a sharp one, thus producing less fatigue stress on the material [81,95,96].

Sliding of stylus: Sliding is an essential component of a wear testing method in order to subject the material to microfatigue [97].

Descent/lifting speed of stylus: The speed with which the stylus contacts the surface of the specimen creates a force

impulse, which is different with varying speeds. If weights are used to exert a force, then the force that is generated on the material is the product of the weight and the descent speed ($F = m \times a$, N). Another variable is the time during which the force is exerted, i.e. the force impulse is the product of the force and the time the force is applied ($F = F \times t$, Ns).

Lubricant: Lubricants, such as artificial saliva, reduce the wear as they lower the friction coefficient. A constant change of water removes the worn particles from the interaction zone between stylus and material, thus reducing the effect of the worn material, which, otherwise, may act as an abrasive medium.

Number of cycles: The wear increases with increasing number of cycles. Most in vitro wear test methods demonstrate a “running-in” phase with a steep increase in wear in the initial phase and a flattening of the wear curve thereafter.

Abrasive medium: An abrasive medium may reduce wear compared to water [98]. Furthermore, the composition as well as the type of the abrasive medium affects the wear rate [99,100].

The wear simulation should follow physical principles: The wear simulation should imitate tribological phenomena that occur in the mouth in a standardized way [101].

A wear method should not only be internally valid, which means that the results for the same material tested at two different points in time are similar, but the wear method should also be externally valid, which means that the results correlate with in vivo findings. The raw data on clinical wear of restorative materials was used to correlate the clinical wear results with those of the most frequently cited wear methods [102]. A moderate correlation was found for OHSU (abrasion). Almost no correlation was found for the ACTA method and a weak correlation for the Alabama, Ivoclar, Munich and Zurich methods (see Table 1). The combination of different methods did not improve the correlation.

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REFERENCES

- [1] Beck F, Lettner S, Graf A, Bitriol B, Dumitrescu N, Bauer P, et al. Survival of direct resin restorations in posterior teeth within a 19-year period (1996–2015): a meta-analysis of prospective studies. *Dent Mater* 2015;31:958–85.
- [2] Alcaraz MGR, Veitz-Keenan A, Sahrman P, Schmidlin PR, Davis D, Iheozor-Ejiofor Z. Direct composite resin fillings versus amalgam fillings for permanent or adult posterior teeth. *Cochrane Database Syst Rev* 2014.
- [3] ISO 4049. Dentistry—polymer-based restorative materials; 2009.
- [4] Ferracane JL. Resin-based composite performance: are there some things we can't predict? *Dent Mater* 2013;29:51–8.
- [5] Heintze SD, Ilie N, Hickel R, Reis A, Loguercio A, Rousson V. Laboratory mechanical parameters of composite resins and their relation to fractures and wear in clinical trials—a systematic review. *Dent Mater* 2017;33:e101–14.
- [6] ASTM D790-10. Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials; 2010.
- [7] ISO 178:2010. Plastics—determination of flexural properties; 2010.
- [8] Ilie N, Bauer H, Draenert M, Hickel R. Resin-based composite light-cured properties assessed by laboratory standards and simulated clinical conditions. *Oper Dent* 2013;38:159–67.
- [9] Kumar N. Inconsistency in the strength testing of dental resin-based composites among researchers. *Pak J Med Sci* 2013;29:205–10.
- [10] Hammant B. The use of 4-point loading tests to determine mechanical properties. *Composites* 1971;2:246–9.
- [11] Chitchumnong P, Brooks SC, Stafford GD. Comparison of 3-point and 4-point flexural strength testing of denture-base polymers. *Dent Mater* 1989;5:2–5.
- [12] ASTM F394-78. Test method for biaxial flexure strength (modulus of rupture) of ceramic substrates; 1996.
- [13] Palin WM, Fleming GJP, Burke FJT, Marquis PM, Randall RC. The reliability in flexural strength testing of a novel dental composite. *J Dent* 2003;31:549–57.
- [14] Palin WM, Fleming GJP, Marquis PM. The reliability of standardized flexure strength testing procedures for a light-activated resin-based composite. *Dent Mater* 2005;21:911–9.
- [15] ASTM D695. Standard test method for compressive properties of rigid plastics; 2010.
- [16] ISO 604:2003-12. Compressive properties of plastics; 2012.
- [17] Park JK, Lee GH, Kim JH, Park MG, Ko CC, Kim HI, et al. Polymerization shrinkage, flexural and compression properties of low-shrinkage dental resin composites. *Dent Mater J* 2014;33:104–10.
- [18] ASTM D638-14. Standard test method for tensile properties of plastics; 2014.
- [19] ISO 527-2-2. Plastics—determination of tensile properties—part 2: test conditions for moulding and extrusion plastics; 2012.
- [20] Hotta M, Kawano S, Sekine I. Direct tensile strength of visible light-cured restorative materials containing fluoride. *J Oral Rehabil* 2001;28:854–9.
- [21] ADA specification no. 27—resin-based filling materials; 1993.
- [22] Penn RW, Craig RG, Tesk JA. Diametral tensile-strength and dental composites. *Dent Mater* 1987;3:46–8.
- [23] ASTM D256-10. Standard test methods for determining the Izod pendulum impact resistance of plastics; 2010.
- [24] ISO 179-1. Plastics—determination of Charpy impact properties—part 1: non-instrumented impact test; 2010.
- [25] Takahashi Y, Chai J, Tan SC. Effect of water storage on the impact strength of three glass fiber-reinforced composites. *Dent Mater* 2006;22:291–7.
- [26] ASTM D732-10. Standard test method for shear strength of plastics by punch tool; 2010.
- [27] Nomoto R, Carrick TE, McCabe JF. Suitability of a shear punch test for dental restorative materials. *Dent Mater* 2001;17:415–21.
- [28] Ikejima I, Nomoto R, McCabe JF. Shear punch strength and flexural strength of model composites with varying filler volume fraction, particle size and silanation. *Dent Mater* 2003;19:206–11.
- [29] Cunha LG, Alonso RCB, Pfeifer CSC, De Goes MF, Ferracane JL, Sinhorette MAC. Effect of irradiance and light source on contraction stress, degree of conversion and push-out bond strength of composite restoratives. *Am J Dent* 2009;22:165–70.

- [30] Ferracane JL, Condon JR. In vitro evaluation of the marginal degradation of dental composites under simulated occlusal loading. *Dent Mater* 1999;15:262–7.
- [31] Xu HHK, Quinn JB, Giuseppetti AA. Wear and mechanical properties of nano-silica-fused whisker composites. *J Dent Res* 2004;83:930–5.
- [32] ASTM D5045-14. Standard test methods for plane-strain fracture toughness and strain energy release rate of plastic materials; 2014.
- [33] ISO 13586:2000. Plastics—determination of fracture toughness (GIC and KIC)—linear elastic fracture mechanics (LEFM) approach; 2000.
- [34] Fujishima A, Ferracane JL. Comparison of four modes of fracture toughness testing for dental composites. *Dent Mater* 1996;12:38–43.
- [35] ASTM C1421-10. Standard test methods for determination of fracture toughness of advanced ceramics at ambient temperature; 2010.
- [36] Shyam A, Lara-Curzio E. The double-torsion testing technique for determination of fracture toughness and slow crack growth behavior of materials: a review. *J Mater Sci* 2006;41:4093–104.
- [37] ASTM E1304-9. Standard test method for plane-strain (Chevron-notch) fracture toughness of metallic materials; 2014.
- [38] Quinn GD, Bradt RC. On the Vickers indentation fracture toughness test. *J Am Ceram Soc* 2007;90:673–80.
- [39] Kruzic JJ, Ritchie RO. Determining the toughness of ceramics from Vickers indentations using the crack-opening displacements: an experimental study. *J Am Ceram Soc* 2003;86:1433–6.
- [40] Watts DC, Issa M, Ibrahim A, Wakiaga J, Al-Samadani K, Al-Azraqi M, et al. Edge strength of resin-composite margins. *Dent Mater* 2008;24:129–33.
- [41] Baroudi K, Silikas N, Watts DC. Edge-strength of flowable resin-composites. *J Dent* 2008;36:63–8.
- [42] Quinn JB, Quinn GD. Material properties and fractography of an indirect dental resin composite. *Dent Mater* 2010;26:589–99.
- [43] Quinn GD. On edge chipping testing and some personal perspectives on the state of the art of mechanical testing. *Dent Mater* 2015;31:26–36.
- [44] Lohbauer U, Belli R, Ferracane JL. Factors involved in mechanical fatigue degradation of dental resin composites. *J Dent Res* 2013;92:584–91.
- [45] ASTM D7791-12. Standard test method for uniaxial fatigue properties of plastics; 2012.
- [46] ASTM D7774-12. Standard test method for flexural fatigue properties of plastics; 2012.
- [47] Lin LH, Drummond JL. Cyclic loading of notched dental composite specimens. *Dent Mater* 2010;26:207–14.
- [48] Garcia-Godoy F, Frankenberger R, Lohbauer U, Feilzer AJ, Kramer N. Fatigue behavior of dental resin composites: flexural fatigue in vitro versus 6 years in vivo. *J Biomed Mater Res Part B Appl Biomater* 2012;100B:903–10.
- [49] Draughn RA. Compressive fatigue limits of composite restorative materials. *J Dent Res* 1979;58:1093–6.
- [50] Turssi CP, Ferracane JL, Ferracane LL. Wear and fatigue behavior of nano-structured dental resin composites. *J Biomed Mater Res Part B Appl Biomater* 2006;78B:196–203.
- [51] ASTM E2546-07. Standard practice for instrumented indentation testing; 2007.
- [52] Ilie N, Rencz A, Hickel R. Investigations towards nano-hybrid resin-based composites. *Clin Oral Investig* 2013;17:185–93.
- [53] Ilie N, Hickel R. Investigations on mechanical behaviour of dental composites. *Clin Oral Investig* 2009;13:427–38.
- [54] Quinn GD. Room-temperature flexure fixture for advanced ceramics. NISTIR 4877. Gaithersburg, MD: National Institute of Standards and Technology; 1992.
- [55] Chitchumnong P, Brooks SC, Stafford GD. Comparison of three- and four-point flexural strength testing of denture-base polymers. *Dent Mater* 1989;5:2–5.
- [56] Wang Y, Darvell BW. Effect of elastic modulus mismatch on failure behaviour of glass ionomer cement under Hertzian indentation. *Dent Mater* 2012;28:279–86.
- [57] ISO14577-1:2015. Metallic materials—instrumented indentation test for hardness and materials parameters—part 1: test method; 2015.
- [58] Oliver WC, Pharr GM. An improved technique for determining hardness and elastic-modulus using load and displacement sensing indentation experiments. *J Mater Res* 1992;7:1564–83.
- [59] Rieister L, Bell TJ, Fischer-Cripps AC. Analysis of depth-sensing indentation tests with a Knoop indenter. *J Mater Res* 2001;16:1660–7.
- [60] Fischer-Cripps AC. Nanoindentation. third edition; 2011. p. 235–76.
- [61] Asif SAS, Wahl KJ, Colton RJ, Warren OL. Quantitative imaging of nanoscale mechanical properties using hybrid nanoindentation and force modulation. *J Appl Phys* 2001;90:1192–7.
- [62] Tabor D. The hardness of solids. *Rev Phys Technol* 1970;1:145–79.
- [63] ISO6506-1. Metallic materials—Brinell hardness test—part 1: test method; 2014.
- [64] ISO4545-1. Metallic materials—Knoop hardness test—part 1: test method; 2005.
- [65] ISO2039-1. Plastics—determination of hardness—part 2: Rockwell hardness; 2010.
- [66] ISO/CD6507-1. Metallic materials—Vickers hardness test—part 1: test method; 2006.
- [67] ISO14577-1:2015. Metallic materials—instrumented indentation test for hardness and materials parameters—part 1: test method; 2015.
- [68] Cramer NB, Stansbury JW, Bowman CN. Recent advances and developments in composite dental restorative materials. *J Dent Res* 2011;90:402–16.
- [69] Poskus LT, Placido E, Cardoso PEC. Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations. *Dent Mater* 2004;20:726–32.
- [70] Mair LH, Stolarski TA, Vowles RW, Lloyd CH. Wear: mechanisms, manifestations and measurement. Report of a workshop. *J Dent* 1996;24:141–8.
- [71] Roulet J-F. Degradation of dental polymers. Basel: Karger; 1987.
- [72] Van Noort R. Introduction to dental materials. Edinburgh: Mosby Elsevier; 2007.
- [73] Lambrechts P, Debels E, Van Landuyt K, Peumans M, Van Meerbeek B. How to simulate wear? Overview of existing methods. *Dent Mater* 2006;22:693–701.
- [74] ISO. Dental materials—guidance on testing of wear. Part 2: wear by two-and/or three body contact. Technical specification; 2001. p. 14569–72.
- [75] ISO. Dental materials—guidance on testing of wear resistance—Part 1: wear by toothbrushing. Technical Report; 2007:14569–71.
- [76] Teixeira EC, Thompson JL, Piascik JR, Thompson JY. In vitro toothbrush-dentifrice abrasion of two restorative composites. *J Esthet Restor Dent* 2005;17:172–80.
- [77] Wang L, Garcia FC, Amarante de Araujo P, Franco EB, Mondelli RF. Wear resistance of packable resin composites after simulated toothbrushing test. *J Esthet Restor Dent* 2004;16:303–14.

- [78] Folwaczny M, Mehl A, Kunzelmann KH, Hickel R. Determination of changes on tooth-colored cervical restorations in vivo using a three-dimensional laser scanning device. *Eur J Oral Sci* 2000;108:233–8.
- [79] European Directorate for the Quality of Medicines and HealthCare (EDQM), Qualification of equipment—core document (PA/PH/OMCL (08) 73 2R). Quality Management Guidelines, 2011.
- [80] Heintze SD. How to qualify and validate wear simulation devices and methods. *Dent Mater* 2006;22:712–34.
- [81] Kunzelmann K-H. Verschleissanalyse und –quantifizierung von Füllungsmaterialien in vivo und in vitro. Aachen: Shaker Verlag; 1998.
- [82] DeLong R, Douglas WH. Development of an artificial oral environment for the testing of dental restoratives: bi-axial force and movement control. *J Dent Res* 1983;62:32–6.
- [83] DeLong R, Douglas WH. An artificial oral environment for testing dental materials. *IEEE Trans Biomed Eng* 1991;38:339–45.
- [84] Condon JR, Ferracane JL. Evaluation of composite wear with a new multi-mode oral wear simulator. *Dent Mater* 1996;12:218–26.
- [85] Leinfelder KF, Suzuki S. In vitro wear device for determining posterior composite wear. *J Am Dent Assoc* 1999;130:1347–53.
- [86] Krejci I, Reich T, Lutz F, Albertoni M. In-Vitro-Testverfahren zur Evaluation Dentaler Restaurationssysteme 1: Computergesteuerter Kausimulator. *Schweiz Monatsschr Zahnmed* 1990;100:953–60.
- [87] Rosentritt M, Leibrock A, Lang R, Behr M, Handel G. Gerät zur Simulation des Kauorgans (Regensburger Kausimulator). *Materialprüfung* 1997;39:77–80.
- [88] de Gee AJ, Pallav P. Occlusal wear simulation with the ACTA wear machine. *J Dent* 1994;22(Suppl. 1):S21–7.
- [89] Yap AU, Ong LF, Teoh SH, Hastings GW. Comparative wear ranking of dental restoratives with the BIOMAT wear simulator. *J Oral Rehabil* 1999;26:228–35.
- [90] Alemzadeh K, Raabe D. Prototyping artificial jaws for the Bristol Dento-Munch Robo-Simulator: 'A parallel robot to test dental components and materials'. *Conf. Proc. IEEE Eng. Med. Biol. Soc.* 2007 2007:1453–6.
- [91] Heintze SD, Barkmeier WW, Latta MA, Rousson V. Round robin test: wear of nine dental restorative materials in six different wear simulators—supplement to the round robin test of 2005. *Dent Mater* 2011;27:e1–9.
- [92] Turssi CP, Ferracane JL, Serra MC. Abrasive wear of resin composites as related to finishing and polishing procedures. *Dent Mater* 2005;21:641–8.
- [93] Ferracane JL, Musanje L. Effects of load and antagonist shape on wear of composite. *J Dent Res* 2003;82. Abstract 825.
- [94] Heintze SD, Cavalleri A, Zellweger G, Ferracane JL. Influence of the antagonist material on the wear of different composites in two different wear simulators. *Dent Mater* 2006;22:166–75.
- [95] Krejci I, Lutz F, Zedler C. Effect of contact area size on enamel and composite wear. *J Dent Res* 1992;71:1413–6.
- [96] Condon JR, Ferracane JL. Effect of antagonist diameter on in vitro wear of dental composite. *J Dent Res* 2003;82. Abstract 954.
- [97] McKinney JE, Wu W. Relationship between subsurface damage and wear of dental restorative composites. *J Dent Res* 1982;61:1083–8.
- [98] Condon JR, Ferracane JL. Factors effecting dental composite wear in vitro. *J Biomed Mater Res* 1997;38:303–13.
- [99] Schnabel C, Kunzelmann K-H, Hickel R. The influence of different abrasion media on three-body-wear of composites. *J Dent Res* 1995;74:90. Abstract No 625.
- [100] Rosentritt M, Behr M, Hofmann N, Handel G. In vitro wear of composite veneering materials. *J Mater Sci* 2002;37:425–9.
- [101] Heintze SD, Zellweger G, Zappini G. The relationship between physical parameters and wear of dental composites. *Wear* 2007;263:1138–46.
- [102] Heintze SD, Faouzi M, Rousson V, Özcan M. Correlation of wear in vivo and six laboratory wear methods. *Dent Mater* 2012;28:961–73.