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## Tribology in the oral cavity: a review

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**Abstract:** There are various applications of tribology in industry, biomedical field as well as dentistry. wear of screws and plates in bone. Dental applications of tribology involve mechanisms such as tooth brushing and mastication. This work offers a review of tribological aspects of human teeth, including; hardness, friction and wear.

**Keywords:** Tribology, Hardness, Friction, and Tooth Wear.

### Introduction

**Tribology** is a Greek word driven from “Tribos” which means rubbing. It is a branch of science and engineering that deals with the interacting surface in related motion and related subjects and practice. It involves friction, lubrication, and wear.<sup>(1)</sup>

There are various applications of tribology in industry, biomedical field as well as dentistry. Industrial examples include gear and brake, biomedical application is related to joints' design, and the wear of screws and plates in bone. While dental applications of tribology involve mechanisms such as tooth brushing and mastication. <sup>(2)</sup>

Friction is the rubbing action of an object on the surface against each other, while wear is material's surface loss when exposed to another surface or an active chemical substance. <sup>(2)</sup>

Generally, the oral biomechanical function can result in some tribological motions of teeth, restorations, and implants. The process of chewing food involves contacting of teeth or restorations and their movement; therefore, friction and wear will occur with the lubrication action of saliva. <sup>(3)</sup>

Wear represents a clinical problem that affects most patients, but it shows an increasing rate among elderly people. It is important to understand dental friction, hardness property, and wear behavior of both teeth and dental materials to minimize the causal factors. <sup>(2)</sup>

### I- Hardness

Hardness is defined as the ability of a material or structure to resist permanent indentation or penetration. It is the ability to resist plastic deformation caused by an indentation force. <sup>(4)(5)</sup>

Hardness has a dental significance that influences the ease of cutting, finishing, and polishing, which has aesthetic consideration. Also, the scratches can affect fatigue strength and lead to premature failure. <sup>(5)</sup>

### **Method of Testing Hardness**

Testing the hardness of tooth structure or dental material depends on applying an indenter with a specific weight (force) on the surface to produce an indentation. The hardness value is obtained by measuring the depth, width, or area of the formed indentation. Generally, the hardness value is inversely proportional to the dimension of indentation, i.e., hard material shows small indentation while softer material has a greater indentation. Hardness is measured as force per unit area. <sup>(5)</sup>

The selection of the hardness test depends on the material used, the hardness range, and the desired localization. There are macro-hardness and micro-hardness tests. The most common methods can be summarized as the Brinell hardness test, Rockwell hardness test, Knoop hardness, Vickers's hardness test, Barcol hardness test, and shore A hardness test. <sup>(5)</sup>

**Brinell hardness test (HB)** is one of the oldest and simplest methods to test the hardness of metallic materials and alloys used in dentistry. Brinell hardness is related to other mechanical properties such as proportional limit, and the ultimate tensile strength of the dental gold alloy. The test is performed by pressing a ball indenter made of steel or tungsten carbide under a specific load to the polished surface of the material. Both Brinell and Rockwell are macro-hardness tests with wide use, but they are not suitable to test brittle materials. <sup>(5)</sup>

**Rockwell hardness test (HR)** has an indenter shape of either a steel ball or diamond cone and is used to measure the depth of indentation by a dial gauge in the instrument. The indenter has several diameters, as well as different load applications (60 to 150 kgf [588 to 1470 N]), with each combination described as a special Rockwell scale, Rockwell A to G, denoted RA, RB, and so on. It has several uses in dentistry especially suitable with viscoelastic material. <sup>(5)</sup>

With this system, a hardness number is determined by the difference in depth of penetration resulting from the application of an initial minor load followed by a larger major load; utilization of a minor load enhances test accuracy. On the basis of the magnitude of both major and minor loads, there are two types of tests: Rockwell and superficial Rockwell. For Rockwell, the minor load is 10 kg, whereas major loads are 60, 100, and 150 kg. For superficial tests, 3 kg is the minor load; 15, 30, and 45 kg are the possible major load values. (Fig.2) <sup>(5)</sup>

**The superficial Rockwell** method has been used to test plastics used in dentistry. This method uses a relatively light (30 kgf [294 N]) load and a large diameter (12.7 mm) ball in comparison with the standard Rockwell methods. The test is made by first applying a preload (minor load) of 3 kgf (29.4 N). A major load of 30 kgf (294 N) then is applied to the specimen for 10 minutes before a reading is taken. Because dental plastics are viscoelastic, recovery of the indentation occurs once the major load has been removed. The percent recovery can be determined on the same specimen by the following equation:

$$\text{Percent recovery} = [(A - B) / A] \times 100\%$$

**Where:**

**A** is the depth of the indentation caused by the application of the major load for 10 minutes.

**B** is the depth of the indentation after the major load has been removed for 10 minutes.

The advantages of the Rockwell hardness test are that hardness is read directly and it is good for testing viscoelastic materials. The disadvantages are that a preload is needed, greater time is required, and the indentation may disappear immediately on removal of the load. <sup>(5)</sup>

**Vickers's hardness test (HV)** has a diamond pyramid indenter to form a square shape indentation. (Fig.3.a) It is suitable to test tooth structure, brittle dental material as well as dental casting alloys. Yet, it is unsuitable for viscoelastic materials. <sup>(4)(5)</sup>

While the **Knoop hardness test (HK)** has a diamond pyramid indenter that produces rhomboidal outline indentation with a specific geometrical configuration of a ratio of 7:1 length to width. (Fig.3.b) It has a wide load range from 0.1kgf to more than 1 kg. Therefore, hard and soft materials can be measured and could be considered a universal test. Knoop and Vickers are classified as micro-hardness tests as they employ load less than 9.8 N and small indentation produced with limited depth less than 19 $\mu$ m. <sup>(4)(5)</sup>

Shore A and Barcol tests are used for measuring rubber and plastic-type dental materials respectively. **Shore A durometer** is used in the rubber industry to determine the relative hardness of elastomers, where hardness is measured in terms of material elasticity. It consists of a blunt-pointed indenter 0.8 mm in diameter. The indenter is attached by a lever to a scale that is graduated from 0 to 100 units. If the indenter completely penetrates the specimen, a reading of 0 is obtained, and if no penetration occurs, a reading of 100 units results. <sup>(5)</sup>

Because elastomers are viscoelastic, an accurate reading is difficult to obtain because the indenter continues to penetrate the elastomer as a function of time. The usual method is to press down firmly and quickly on the indenter and record the maximum reading as the Shore A hardness. The test has been used to evaluate soft denture liners, mouth protectors, and maxillofacial elastomers. <sup>(5)</sup>

**The Barcol hardness** test has a spring- needle with a diameter of 1 mm that is pressed against the surface to be tested, and connected to the gauge to read the hardness value directly. It is used to study the depth of cure of resin composites. <sup>(5)</sup>

The depth of cure of a resin composite is tested by preparing specimens varying in thickness from 0.5 to 6.0 mm or more in increments of 0.5 mm. Then the top surface of a specimen is activated by a light-curing unit. The Barcol hardness of the top surface is compared with that of the bottom surface. The depth of cure is defined as the maximum thickness at which the Barcol

reading of the bottom surface does not change by more than 10% of the reading of the top surface. Research has shown that a 10% decrease in Barcol hardness of a resin composite results in a 20% decrease in flexural strength. <sup>(5)</sup>

**Nano-indentation test** is the latest technology of using an indentation with a nanoscale size to eliminate the limitation as many materials have microstructural constituents as in the case of micro and nano-filled composites, and filler phases substantially smaller than the dimensions of the traditional indenter. To accurately measure the properties of these microphases, it is necessary to be able to create indentations of a smaller size scale and to spatially control the location of the indentations. <sup>(5)</sup>

Nano-indenter can apply load in the range of 0.1 to 5000 mgf, the resulting indentation of size  $1\mu m$ . In addition, indentation depth is continuously monitored, eliminating the need to image the indentation to compute mechanical properties. This technique is not only suitable to test the hardness but also can measure other mechanical properties like elastic modulus, also for brittle material fracture toughness, and yield strength may be determined. <sup>(5)</sup>

## II- Friction

Friction is the force that prevents one solid item from slipping or rolling over another. Frictional forces are advantageous, but they also provide a lot of resistance to motion. <sup>(6)</sup>

The forces of attraction, known as adhesion, between the contact zones of the surfaces (microscopically rough), appear to be the primary cause of friction between metals. Friction is caused by the roughness of the tougher surface plowing through the softer surface. <sup>(6)</sup>

The apparent area of contact (**A**) is defined as the overall exterior dimensions of the contact surface, while the real area of contact (**A<sub>r</sub>**) has a considerably more fundamental effect on frictional processes and is defined as the sum of the areas in contact at the asperity tips. <sup>(7)</sup>

The asperity tips of the two surfaces make contact initially when they approach each other. The asperity tips' shapes, dimensions, locations, and contacts are completely random; thus, the interactions are random. The asperities flatten as the force increases, and the real area of contact grows until it approaches the value of the apparent area. It should be noted that the load or weight that presses the surfaces together determines friction. <sup>(6,7)</sup>

As a result, the friction to load ratio remains constant. The coefficient of friction is the name given to this constant ratio. <sup>(6,8,9)</sup>

$$\mu = F_f / N$$

**Where:**  $\mu$  is the static ( $\mu_s$ ) or kinetic ( $\mu_k$ ) coefficient of friction

**F<sub>f</sub>** is the frictional force

$N$  is the normal force between the 2 surfaces

The coefficient of friction is dimensionless since both  $F$  and  $L$  are measured in force units (newtons or pounds). Each of the static and dynamic friction has a specific value of coefficient of friction. <sup>(6,8,9)</sup>

When an object moves, it creates kinetic (sliding) friction, whereas when an object makes an effort to move, it creates static friction. <sup>(9)</sup>

E.g.: The coefficient of kinetic friction for an object sliding on a clean surface is around 0.5, which means that a force equal to half the weight of the object is necessary only to overcome friction and keep it moving at a constant pace, and the coefficient of static friction is about 0.6. The frictional force is directed in the opposite direction as the object moves. <sup>(8)</sup>

### III- Wear

Wear, as defined previously, represents a critical clinical problem due to the loss of teeth surfaces as well as various restorative materials. Wear can be formed by direct contact between teeth and abrasive particles or devices during mastication, bruxism, tooth brushing, and other function. <sup>(2)(5)</sup>

Teeth are constantly subjected to mechanical stress and chemical changes, so wear is unavoidable. Dental wear is a natural part of the teeth's life cycle. It can be excessive when the occlusal surface is lost, such as in the case of overbite, or bruxism. <sup>(10)</sup>

Excessive wear might result in:

- Lack of optimal contact between opposing teeth.
- Disruption in the masticatory system's function.
- Elimination of the masticatory surfaces.

The oral cavity is frequently compared to a tribological system in order to correctly assess the many types of wear between teeth and restorative materials.

The system is often made up of four elements:

- The first is a solid body (the tooth).
- The second is the counter-body, which is usually a solid (e.g. an object or an opposing tooth).
- The third is the interfacial element, which is usually a solid (food bolus particles, etc.), a lubricant liquid (saliva), less frequently a gas, or a combination of these elements.
- The fourth is the air, which is the environment where the wear occurs. <sup>(10)</sup>

Several factors make the wear of biomaterials unique. Most important, wear can produce particles that can elicit an inflammatory response. The wear process can also produce shape changes in the object that can affect function. For example, the wear of teeth and restorative materials is characterized by the loss of the original anatomical form of the material. <sup>(5)</sup>

Wear can be categorized as physiological wear, pathological wear, and prophylactic wear. Physiological wear that occurs during normal mastication and results in attrition and loss of surface convexity of tooth cusp or incisal edge forming a flat surface. There are some pathological factors that can cause wear to teeth and restorations as reported clinically that wear is highly significant due to erosion, bruxism, xerostomia, and others. Pathological wear can be caused by bad oral habits such as biting on hard objects like pens or chewing tobacco or it may be related to some occupations. Prophylactic wear can occur during preventive condition as tooth brushing and improper use of dental floss or toothpicks. <sup>(2)</sup>

**Abrasion** is the form of wear in which friction between a tooth or restoration and an exogenous agent (such as food, toothpaste, toothpick, and dental floss), **Erosion** in dental literature is the surface loss of either teeth or restorations caused by chemical or electrochemical action. **Abfraction** (Fig.8) is used to term stress-induced dental hard tissue loss, which occurs most commonly in the cervical region of teeth. <sup>(2)</sup>

### Types of wear

Wear can be classified into:

- ❖ **Abrasive wear** involves a harder material cutting or plowing into a softer material. It has two types two-body and three-body abrasive wear.
  - Two-body abrasion occurs when abrasive particles are firmly bonded to the surface of the abrasive instrument with no other abrasive particles. E.g., diamond bur abrading tooth during tooth preparation.
  - Three-body abrasions can occur when there are free abrasive particles between two surfaces. E.g., dental prophylaxis paste. <sup>(4)</sup>
- ❖ **Adhesive wear** occurs by the distraction of the micro junction of material, pulling an object from the microregion and transfer to another.
- ❖ **Surface Fatigue wear** occurs when certain small free particles or asperities contribute to high localized stresses and produce surface or subsurface cracks. Particles break off under cyclic loading and sliding.
- ❖ **Corrosive wear** which occurs by the loss of the protective layer in the presence of chemicals and sliding action (friction) between surfaces can increase the rate of corrosion. <sup>(2)(5)</sup>

Generally, it was found that metal may show adhesive, corrosive, and three-body wear. While polymers are susceptible to abrasive and fatigue wear. (5)

### **Methods of measuring wear**

There are several methods for measuring the wear of teeth and restoration which can be classified into *in-vitro* laboratory simulation testing as well as *in-vivo* observation and measurement. (2)

*In-vitro* laboratory simulation is usually used by tribology researchers to explore the wear mechanisms of natural teeth and artificial materials. However more recently, the *in-vivo* method has been introduced to investigate dental tribology. *In-vivo* observation and measurement are generally used by clinicians to observe and evaluate clinical manifestations of the wear of teeth and restorations intra-orally, during the oral examination. This can be done by using a study cast or macro-photography and a 3D intraoral scanner. (2)(11)

### **In-vitro Testing:**

In-vitro wear test, which allows exact control of target parameters, were designed and developed in response to the drawbacks of in-vivo measurements.

Based on the wear mechanism to be simulated (e.g., abrasion, attrition, etc.), associated parameters such as load condition, number of cycles, the intensity of testing, and particular lubricating medium allow accurate selection and adjustment. (2)

#### **Advantages:**

1. Accurate parameter control.
2. Wear susceptibility indication.
3. High testing efficiency.

#### **Disadvantages:**

Difficulties in duplicating the exact complicated oral environment. (2)

### ***In-vitro tests include:***

#### **A. Conventional tribometers:**

The method is based on the use of two-component wear. A body in the form of a pin or a non-rotating ball is applied to the disc-shaped sample surface. A specific force is applied to the pin or ball at a predetermined distance from the sample center. The

disc begins to rotate at the chosen speed and completes the specified number of cycles. As a result, the pin or ball body creates a track (path) on the sample surface that is examined. <sup>(10,11)</sup>

**Advantages:**

1. This test is straightforward, standardized, and low-cost.
2. Abrasive food particles or artificial saliva are added to produce more realistic test conditions for chewing examination of the third component of wear. <sup>(11)</sup>

**Disadvantages:**

1. The circumstances in the mouth cavity vary greatly in comparison to the test instrument.
2. This rotational action is considerably different from the mastication movement. <sup>(11)</sup>

**B. Reciprocating Tribometer:**

As in tribometer, a pin or a ball is in touch with a flat specimen under a vertical force, but it moves along a linear stroke alternately back and forth with a known frequency. <sup>(10)</sup>

**Advantages:**

1. Simulating tooth movement in a more realistic manner.
2. Slurries of different kinds can be used to imitate abrasive particles and various lubricants. <sup>(2)</sup>

**Disadvantages:**

The environment in the mouth cavity varies greatly in comparison to the test instrument. <sup>(2)</sup>

**C. Toothbrush simulator:**

Hard dental tissues and dental materials undergo wear during tooth brushing. This test employs a toothbrush that glides on the surface of the tested material in a cyclic and linear way, with a pressure force that can be adjusted. It is similar to the pin-on disc method, however instead of a disc, a toothbrush is used. Regarding the device and the toothbrush chosen, standardization of this test is critical. <sup>(11)</sup>

**Advantages:**

1. It is possible to include a third component of wear, such as toothpaste, for a more accurate simulation.
2. It is of low-cost and easy. <sup>(11)</sup>

**Disadvantages:**



1. The abrasive granules come in a variety of sizes.
2. Toothpaste alters the pH of the oral cavity, which is not applicable in the test. <sup>(3)</sup>

**D. Jaws (chewing) simulator:**

The jaws simulator was made composed of a model of upper and lower teeth (or just one tooth with a filling) that slide against each other owing to two or more servo-hydraulic motors. The first motor ensures vertical movement, while the second ensures horizontal movement. The computer controls both engines to ensure the most precise recreation of physiological movements during chewing. <sup>(11)</sup>

**Advantages:**

1. Individual adjustments of biting strength and cycle rate are feasible.
2. The device can include an artificial oral environment, allowing for temperature control similar to the human body, the addition of artificial saliva, and food samples for a more realistic simulation. <sup>(11)</sup>

**Disadvantages:**

There were significant variations between the results of tests in vivo and in vitro using this simulation, as such standard conditions in a patient's mouth do not exist. <sup>(1)</sup>

***In-vivo* Wear Testing**

❖ **Tooth Wear Indices**

According to Smith and Knight, the degree of hard dental tissue wear could be determined and scored as reported in Table 1. The incisal edge, occlusal surface as well as the cervical area of the teeth are the most used sites-for measuring the wear process according to the amount of enamel and dentin lost. <sup>(2)(11)</sup>

**Table 1 Tooth Wear Index (TWI) according to Smith and Knight (1)**

Score	Tooth surface	Criteria
0	Labial/oral/occlusal/incisal Cervical	Without signs of enamel loss Without signs of crown contours loss
1	Labial/oral/occlusal/incisal Cervical	Loss of enamel surface relief Minimal loss of crown contours
2	Labial/oral/occlusal Incisal Cervical	Loss of enamel; dentin baring less than 1/3 of surface Loss of enamel with minimal baring of dentin The depth of defect is less than 1 mm
3	Labial/oral/occlusal Incisal Cervical	Loss of enamel; dentin baring more than 1/3 of surface Significant loss of enamel and dentin The depth of defect is 1–2 mm
4	Labial/oral/occlusal/incisal Cervical	Extensive loss of enamel and dentin with baring of dental pulp The depth of defect is more than 2 mm; baring of dental pulp

### ❖ Basic Erosive Wear Examination system (BEWE)

For the diagnosis of erosion according to this system, the following scoring is

used <sup>(2)(11)</sup>:

0: intact surface.

1: initial loss of surface enamel layer

(Moderate erosion)

2: obvious defect of hard dental tissue affecting up to 50% of tooth surfaces (Advanced erosion)

3: extensive defect of hard dental tissue affecting more than 50% of tooth surfaces (Serious erosion)

### Disadvantages of *in-vivo* testing:

In the tribological analysis of dental materials, examining the tooth in its natural surroundings can be challenging:

1. Wear processes cannot be accelerated *in-vivo*, so tests are often carried out over a long period of time, making them time-consuming and costly.
2. Measurement and assessment rely heavily on volunteer collaboration.
3. As the complex oral environment and mechanisms are involved, studying the influencing parameters separately and controlling them is impossible. <sup>(2)</sup>

### Dental Applications of Tribology

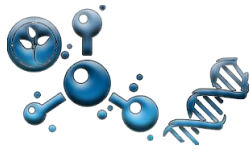
- One of the important considerations for restorative dental materials is their high wear resistance. Dental resin composite may show inadequate wear resistance, although several enhancements can be done recently to improve its wear resistance. <sup>(3)(4)</sup>
- Although wear has an undesirable effect it has the advantage of easily finishing and polishing the dental material. <sup>(4)</sup>
- Wear and topographic modification can occur during implant insertion and that can have clinical implications. Accordingly, the analysis and prediction of the wear behavior of dental implant surfaces are mandatory. <sup>(2)</sup>
- Tribological factors affecting tooth enamel include the choice of toothpaste and toothbrush. <sup>(2)</sup>
- The surface finishing and contouring of ceramics have a significant effect on the rate and extent of wear. <sup>(4)</sup>
- Ceramics are aesthetically favorable and very wear-resistant, yet care should be taken to avoid any destructive potential to the opposing dentition due to high hardness of ceramics. <sup>(4)</sup>

Patient habits, such as bruxism, increase the rate of teeth wear. This eventually leads to dentin exposure and sensitivity and might end with pulpal exposure in severe cases. For such patients, an appliance such as a night guard should be used. <sup>(4)</sup>

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Review article

## Fatigue in dentistry: a review

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**Abstract:** Dental materials are subjected to complex stresses in the oral cavity over a long period during function. Therefore, it's important to understand how dental materials function and the types of failure inside the patient mouth. Fatigue may result in microcrack formation and eventually failure.

**Keywords:** *Fatigue, Fatigue in dentistry, Fatigue mechanism*

### Introduction:

Fatigue is a form of failure that occurs within structures subjected to dynamic and fluctuating stresses. It is possible to occur at a stress level below the yield strength. This type of failure occurs after a period of repeated stress or strain cycle. Fatigue is catastrophic and occurring suddenly.<sup>1</sup>

Tensile, compressive, and shear stresses can all lead to fatigue failure. Fatigue failure is like a brittle failure in nature that there is little plastic deformation with the failure. Failure occurs by crack initiation, and propagation.<sup>2</sup>

### Different modes of stress cycles

Three different stress-time modes are possible.

#### *1) Reversed stress cycle*

It is regular, sinusoidal time dependence, where the maximum and the minimum stresses are equal. The material is loaded in tension (positive stress), and compression (Negative stress) within a cycle.<sup>3</sup>The amplitude is symmetrical around zero stress level, alternating with a maximum tensile and minimum tensile stress of equal magnitude.<sup>1</sup> (Figure 1)

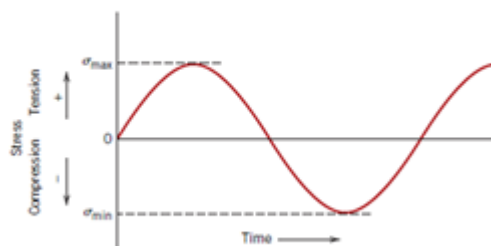


Figure 1: Reversed stress cycle

**2) Repeated stress cycle**

It is the stress cycle where the maximum and the minimum stresses are asymmetrical around zero stress level.<sup>1</sup> (Figure 2)

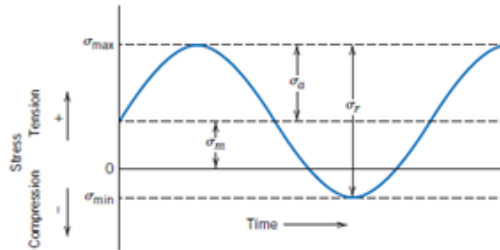


Figure 2: Repeated stress cycle

**3) Random stress cycle**

The stress level is subjected to random loads in amplitude or frequency. (Figure 3)

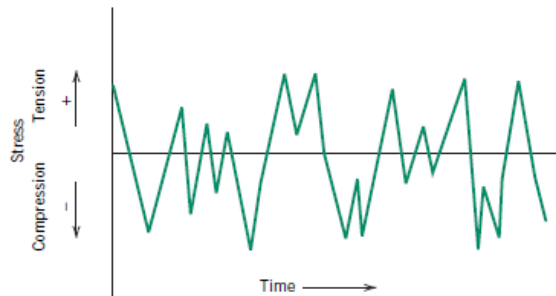


Figure 3: Random stress cycle

Most common type occurring in the oral cavity is the reversed cycle<sup>3</sup>

- ❖ Tensile stresses are positive, and compressive stresses are negative.
- ❖ There are several parameters used to describe the fluctuating cycle:

Mean stress  $\sigma_m$ : is the average of the maximum and minimum stresses in the cycle

$$\sigma_m = \frac{\sigma_{max} + \sigma_{min}}{2}$$

Range of stress  $\sigma_r$ : is the difference between  $\sigma_{max}$  and  $\sigma_{min}$

$$\sigma_r = \sigma_{max} - \sigma_{min}$$

Stress amplitude  $\sigma_a$ : is half the range of stress

$$\sigma_a = \frac{\sigma_{max} - \sigma_{min}}{2} = \frac{\sigma_r}{2}$$

- Stress ratio(R): is the ratio between the minimum and the maximum stress<sup>1</sup>

$$R = \frac{\sigma_{min}}{\sigma_{max}}$$

**Mechanism of failure**

Generally, the process of fatigue failure includes:

- **Crack initiation**: where small crack forms at some point of high concentration.
- **Crack propagation**: where crack advances with each stress cycle.

- **Final failure:** occurs rapidly once the crack reached a critical size.

During fatigue failure, cracks are initiated on the surface at the point of stress concentration. Crack sites are surface scratches, keyways, threads, and dents. Cyclic loading can produce microscopic surface discontinuities from dislocation slips that may act as stress raisers and crack initiation sites.<sup>1</sup>

### Mechanism of failure among the different materials

#### **1) Polymer:**

Fatigue is a common reason for mechanical failure of polymers. Crack propagates in the material when cyclic load on the material reaches a critical value leading to fatigue failure.<sup>4</sup> Polymer fatigue is sensitive to environment (temperature), molecular weight, molecular density and aging. In polymer matrix, fatigue resistance increases as crystallinity increase. During fatigue, there is change in van der Waal force that results in the movement of the polymer chains and creating unfolding of the polymer chains.<sup>5</sup>

The contact between the polymer and the sliding counter-face results in increasing the mobility of the macromolecule chains, and this increases the irreversible shift of the macromolecule chains due to the contact stresses. As the number of the intermolecular bond breakage increases, microdefects appear in polymers. During friction microdefects accumulate resulting in the appearance of microcracks.<sup>6,7</sup>

#### **2) Ceramics:**

Cyclic fatigue in ceramics refers to fatigue crack propagation. It is a rate dependent crack growth in the presence of moisture. As water enters the fissures and break down the cohesive bonds within the material forming what is called Subcritical or slow crack growth which progress over time, accelerating at higher stress levels and leading to failure. The good fabrication procedures and avoiding of the surface damage is critical in the prosthetic dentistry to avoid the failure process.<sup>8</sup> Water can enter the crack tip and accelerate the chemical corrosion attack in ceramics especially silicate base, by breaking the primary chemical bonds of ceramics at the crack tip causing subcritical propagation until the crack reached critical crack size for failure, and hydrolyzing the siloxane bonds even at moisture level as low as 0.02%.

#### **3) Metals:**

For metals, fatigue is expressed in the form of irreversible motion of dislocation and their interactions. The primary mechanism of fatigue in metals is the localization of strain as first step in crack initiation. Strain accumulates through dislocation resulting in slip. Slip moves until it is irreversible. In many metals, the strain is localized in the form of slip bands. The accumulation of slips during cyclic loadings, leads to plastic deformation and the nucleation of micro-cracks. The lifetime of metal depends on the amount of cycles needed to initiate a crack and the amount of cycles required to propagate the crack to the failure point.<sup>9,10</sup>

#### **4) Composite**

In resin composite, many mechanisms participate in fatigue damage of composite, including matrix degradation, void formation, filler debonding and filler failure. Fatigue fracture occurs from internal or external micro-flow, from which crack propagates and leads to fracture.<sup>6</sup> Crack growth in composite is related to the composition, microstructure and susceptibility to degradation. Water exposure will lead to weakening of resin composite, so this accelerates the crack growth. As the microstructure affect the fatigue resistance; high filler loaded materials and small filler particles are more fatigue resistant.<sup>11</sup>

### **Microscopic and Macroscopic Features of fatigue**

The region of the crack formed during crack propagation is characterized by one of two types of markings: **beach-marks and striation**.

Concentric ridges expand from the crack initiation sites in a circular or semicircular pattern. Beach-marks are a macroscopic feature and are also called clamshell marks. They are formed when the material experiences interruption during the crack propagation stage. Beach-marks indicate a change in growth rates when fatigue is applied in a consistent number of cycles. Each beach-mark represents a period of time where crack growth occurred. Each package produces a total crack extension than can be seen without enlargement. Packages of load repetition may be shorter or longer than periods of rest. Each beach-mark is a single or more packages of cycles, not a single cycle and it is a progressive stage of crack propagation.<sup>1</sup> (Figure 4)

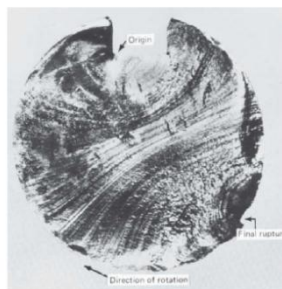


Figure 4: Detected beachmark in fatigued material

Striation is microscopic in size and can be observed by using scanning electron microscope or transmission electron microscope. Each striation represents a single cycle. It is the advanced distance of a crack during a single load cycle. Striation depth increases with increasing the stress range. During propagation of fatigue cracks and on a microscopic level, there is localized plastic deformation at crack tips even though the maximum stress to which the object is exposed is below the yield strength of the metal. The applied stress is amplified at the crack tip so that the local stress exceeds the yield strength of the metal.<sup>1</sup> (Figure 5)

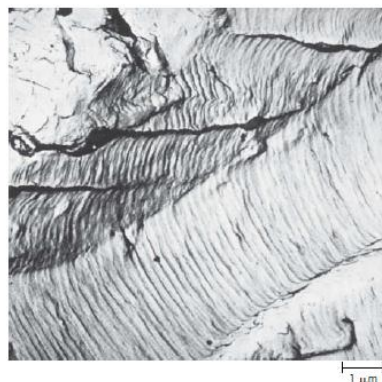


Figure 5: Striations observed in fatigued material

Although beach-marks and striations are fatigue surface features having a similar appearance, they come from different origins and sizes. There may be thousands of striations in a single beach-marks.

The presence of beach-marks and striations confirms that the cause of failure was fatigue, but the absence of either or both doesn't exclude fatigue. The appearance of striations may depend on the stress state. Detection of striation decreases with time because of the formation of surface oxide films or corrosion products. Beach-marks and striations don't appear in the region of rapid failure. Rapid failure may be ductile or brittle.<sup>1</sup> (Figure 6)

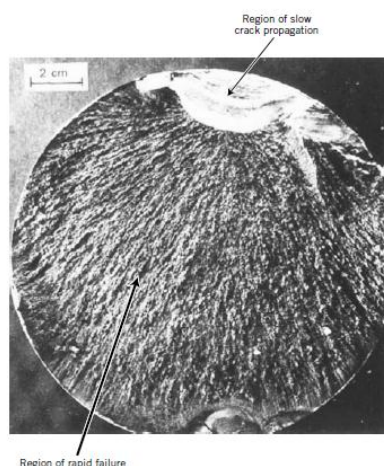


Figure 6: Crack on the top is propagated slowly, while dull and fibrous area is fast

**Fatigue testing method**

The most common method to test fatigue is the *rotating-bending beam*.

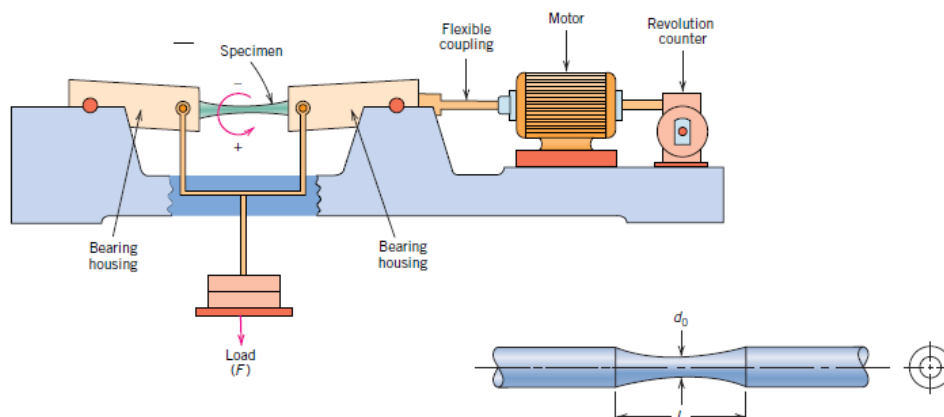


Figure 7: Rotating bending beam

It is alternating tension and compression of equal magnitude on the specimen as it is bent or rotated. In this case, the stress cycle is reversed, so that the stress ratio is -1. The lower surface of the specimen is subjected to tension (+ positive) while the upper part of the specimen is subjected to compression (- negative). (Figure 7)

Series of tests are started by subjecting the specimen to stress cycling at large maximum stress, usually on the sequence of two-thirds of the tensile stress, and then the number of cycles to failure is counted and recorded. The procedure is repeated with decreasing stress levels. Then the data is described on a graph where S is the stress and N is the number of cycles to failure for each specimen (S-N Curve). In the S-N curve, the higher the magnitude, the smaller the number of cycles the material can withstand before failure.<sup>1</sup>

Failure could be tested simulating the actual clinical condition for the various dental restorations.

**Types of S-N Curves:**

Two types of S-N curves are observed:



**1) S-N curve with Fatigue limit:**

There is a stress limit level called Fatigue limit or endurance limit. (Figure 8)

**Fatigue limit or the endurance limit** is the largest stress the material can withstand an infinite number of cycles without failure. Below such value fatigue failure doesn't occur. For many steels, this limit is 35-60% of the tensile strength. E.g. Fatigue for ferrous (Iron-base) and titanium alloys.<sup>1</sup>

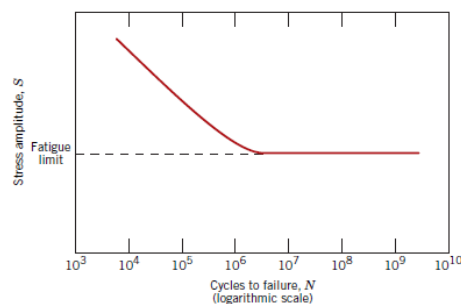


Figure 8: Fatigue limit

**2) S-N curve without fatigue limit:**

The S-N curve continues downward at a greater number of cycles. Therefore, fatigue occurs regardless of the number of cycles. Fatigue in those alloys is defined as the fatigue strength.

**Fatigue strength** is the stress level at which failure will occur for a specific number of cycles ( $10^7$ ).

**Fatigue life** is the number of cycles that cause failure at a specific stress level. <sup>1</sup>(Figure 9) E.g. fatigue for non-ferrous alloys

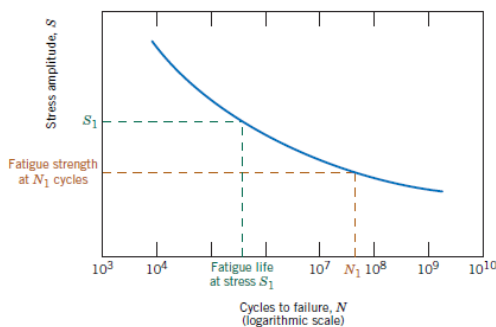


Figure 9: Fatigue life

From the graph below (Figure 10) Cast iron, Mg alloy, and steel have a fatigue limit, while Aluminum alloy, titanium alloy, and brass don't show a fatigue limit.<sup>1</sup>

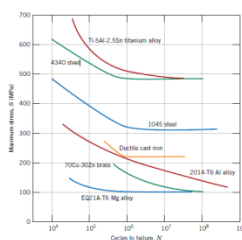


Figure 10: Materials with fatigue limit

Fatigue from the previous S-N curves shows the best-fit curves, while fatigue fails at a stress level 25% below the curve.

### Factors affecting fatigue life:

Fatigue is affected by many variables including mean stress level, geometric design, surface effect, metallurgical variables, and environment.

#### 1) Loading condition

##### a) Mean stress:

Fatigue life depends on stress presented on the S-N. Those data are taken for a constant mean for the reversed cycle, the mean stress is  $\sigma$ . Mean stress also affects the fatigue life. Increasing the mean stress level leads to decreasing the fatigue life.<sup>1</sup> (Figure 12)

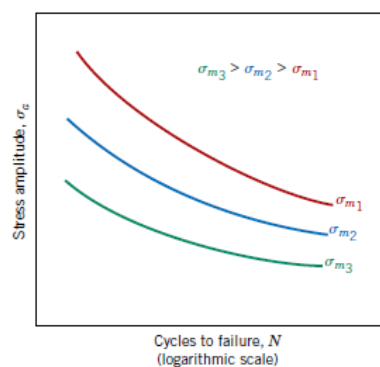


Figure 12: mean stress

##### b) Number and load of cycles

###### 1) Low cycle fatigue:

If the number of cycles is small, then this is called low cycle fatigue. This is associated with high loads that produce not only elastic strain but also some plastic strain.<sup>1</sup>

###### 2) High cycle fatigue:

If the number of cycles is large, then this is called high cycle fatigue. This is associated with a low stress levels where the deformation is elastic.<sup>1</sup>

#### 1) Specimen condition

##### a) Surface effect

The maximum stress within a structure occurs at its surface. Most cracks occur at the surface causing stress amplification leading to fatigue. Therefore, fatigue life is sensitive to the surface of the component. Design criteria and surface treatments affect the fatigue resistance, which improves the fatigue life.<sup>1</sup>

##### b) Surface treatment

During operation, small scratches are introduced to the structure. Those scratches decrease fatigue life. Polishing and improving the surface finish enhances the fatigue life. One of the effective methods to increase the fatigue performance is to introduce residual compressive stresses in the thin outer surface layer. So, the tensile stress of external origin is nullified and reduced by the compressive stress, therefore the net effect is decreasing the crack formation and the fatigue failure.<sup>1</sup>

Residual compressive stresses are induced in ductile metals by localized plastic deformation within the outer surface. This is accomplished by a process called shot peening. Shot peening is a process where small, hard particles having 0.1 to 1.0 mm diameters are projected with high velocities to the surface to be treated. The deformation induces compressive stresses to a depth of one quarter and one half of the shot diameter.<sup>1</sup> (Figure 13)

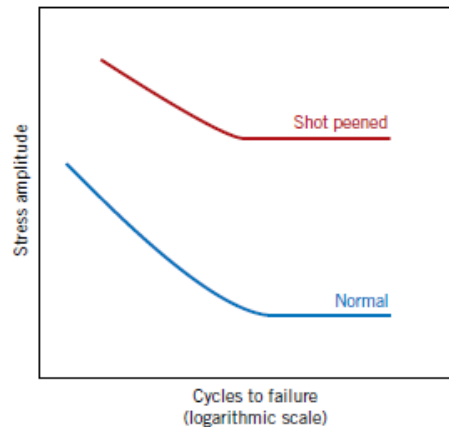
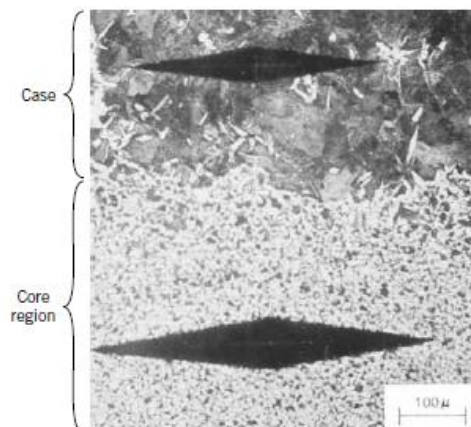


Figure 13: Shot peening effect

Fatigue life for steel is improved by **Case hardening**. This is accomplished by carburizing and nitriding process where exposure to carbon or nitrogen atmosphere at elevated temperature. Therefore, carbon or nitrogen-rich outer layer is introduced by atomic diffusion from the gaseous phase. This layer is 1mm deep and is harder than the core material. This will lead to the improvement of fatigue and increasing the hardness as well as introducing desired residual compressive stresses. As shown in the following figure, the hardness in the outer case is greater than the core <sup>1</sup>(Figure 14)

Figure 14: Hardness in the outer case is greater than the core



### 3) Material

#### a) Type of material

Fatigue failure differs according to the material from metals, ceramics, composite and polymer.

#### b) Design factor

The design of the structure influences the fatigue characteristics. Any geometrical discontinuity can act as a stress raiser and crack initiation site. Geometrical discontinuity includes grooves, holes and threads; the more severe the discontinuity

the more the stress concentration. Fatigue failure can be reduced by avoiding those geometrical discontinuities, or by making design modifications.<sup>1</sup>

### 3) Environmental effects:

Environmental factors may affect fatigue behavior; those factors include thermal fatigue and corrosion fatigue.

#### a) Thermal fatigue:

Thermal fatigue is induced at high temperatures by fluctuating thermal stresses when the mechanical stresses are absent. Thermal stresses are induced due to the restraint to the dimensional expansion and/or contraction that would occur with changing the temperature. The magnitude of thermal stresses developed by temperature change ( $\Delta T$ ) depends on the coefficient of thermal expansion ( $\alpha$ ) and the modulus of elasticity ( $E$ ).<sup>1</sup>

$$\sigma = \alpha_1 E \Delta T$$

Thermal stresses do not rise if the mechanical restraint is absent. So, to reduce this thermal fatigue, this is performed by eliminating the restraint source, so this will allow dimensional changes with temperature changes or choosing a material with appropriate physical properties.<sup>1</sup>

#### b) Corrosion fatigue:

Corrosion fatigue occurs as a result of simultaneous cyclic stresses and chemical attacks. Corrosive environments produce shorter fatigue lives. Small pits may be formed as a result of a chemical reaction between the environment and the material which may act as a stress concentration point and crack nucleation site.<sup>1</sup>

Corrosive fatigue is caused by the rupture of the material protective passive film. The material become weakened due to cyclic loading, the oxide layer become damaged due to alternating tensions and compression. The loss of the oxide layer invites more corrosion, so lowering the strength of the material and shortening the lifespan.<sup>12</sup>

Lowering the load application leads to exposing the opened crack to the environment for long time, reducing the fatigue life.

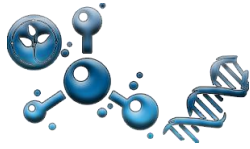
## CONCLUSION

Dental materials are subjected to fatigue in the oral cavity. Protection of the dental prosthesis is one of the methods to decrease the fatigue from decreasing the normal fatigue failure reasons to selecting corrosion resistant materials and reducing the corrosiveness for the environment.

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Type of the Paper (Review Article)

## Composites structure in dentistry and evolution of filler systems and their effects on properties of composites structure

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**Abstract:** The properties of composite material are dependent on the properties of the constituent phases forming the composite, their relative amounts, and the geometry of the dispersed phase .The concept of multiphase composites provided opportunities for designing a large variety of materials with different combinations of properties that cannot be achieved by using a single metal, alloy, ceramic, or polymeric material.

**Keywords:** Tribology, Hardness, Friction, and Tooth Wear.

### Introduction

Composite material is a solid formed from two or more distinct phases (e.g., filler particles dispersed in a polymer matrix) that have been combined to produce properties superior to or intermediate to those of the individual constituents.<sup>(1)</sup>

The properties of composite material are dependent on the properties of the constituent phases forming the composite, their relative amounts, and the geometry of the dispersed phase (shape, size, distribution, and orientation) .

The concept of multiphase composites provided opportunities for designing a large variety of materials with different combinations of properties that cannot be achieved by using a single metal, alloy, ceramic, or polymeric material.

Composites structures occur in nature. As in bone which is a composite of soft collagen proteins and hard brittle mineral apatite. And wood consists of strong and flexible cellulose fibers surrounded and held together by a stiffer material called lignin.

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Most composites have been created to improve the properties whether mechanical, thermal, electrical, magnetic or optical properties of different materials to suit the required application. <sup>(2, 3)</sup>

## **I. Classification of composite structures:**

- a. According to the matrix phase.
- b. According to the dispersed phase.
- c. According to the matrix filler interface.

### **II.a. Classification according to the matrix phase:**

The matrix phase may be metal, polymer, or ceramic. In general, metals and polymers are used as matrix materials because some ductility is desirable. For ceramic-matrix composites the reinforcing component is added mainly to improve fracture toughness.

#### **Functions of the matrix phase:**

1. It binds the filler phase together and acts as the medium by which an externally applied force is transmitted and distributed to the fillers, only a very small proportion of the applied load is sustained by the matrix phase.
2. It protects the individual fillers to an extent from surface damage because of mechanical abrasion or chemical reactions with the environment. Such interactions may introduce surface flaws capable of forming cracks, which may lead to failure at low tensile stress levels.
3. It separates the fillers and prevents the propagation of brittle cracks from filler to filler which could result in catastrophic failure (the matrix phase serves as a barrier to crack propagation).
4. It maintains the orientation of the reinforcing phase for the high strength development.

#### **• Polymeric matrix composites:**

Polymeric matrix composites consist of a polymer resin as the matrix. These materials are used with great diversity in different applications.

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## Applications in dentistry

### METHACRYLATE MONOMERS

The majority of monomers used for the matrix phase of resin composite are dimethacrylate compounds. Two monomers that have been commonly used are (Bis-GMA) and urethane dimethacrylate (UDMA). Both contain reactive carbon double bonds at each end that can undergo addition polymerization initiated by free-radical initiators.<sup>(4)</sup>

- Advantage: Bis-GMA has a higher molecular weight than methyl methacrylate (MMA), which helps to reduce the polymerization shrinkage. The polymerization shrinkage value for MMA resins is 22 vol. %, whereas for a Bis-GMA resin it is 7.5 vol%.<sup>(5)</sup>
- Disadvantage: The viscosity of the monomers, especially Bis-GMA, is rather high and diluents must be added, so a clinical consistency can be reached when the resin mixture is compounded with the filler.

Low- molecular-weight compounds with difunctional carbon double bonds, for example, triethylene glycol dimethacrylate (TEGDMA), or Bis-EMA are added by the manufacturer to reduce and control the viscosity of the compounded composite. These compounds showed high polymerization shrinkage so they can't be used with high concentrations.

### LOW-SHRINK METHACRYLATE MONOMERS

A variety of other methacrylate monomers have been introduced since 2008 for controlling the volumetric shrinkage and polymerization stress of composites. The general approach relies on increasing the distance between the methacrylate groups resulting in lower cross-link density. This could be done using dimer acids, incorporation of cycloaliphatic units, and photocleavable units to relieve stress after polymerization.<sup>(4)</sup>

### LOW-SHRINK SILORANE MONOMER

Silorane has been developed to reduce shrinkage and internal stress build-up resulting from polymerization. The name silorane was coined from its chemical building blocks siloxane and oxirane. The siloxane group provides hydrophobicity to the composite. The oxirane groups undergo ring-opening cross-linking via cationic polymerization.<sup>(4)</sup>



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## Bio HPP (High Performance Polymer)

- BioHPP (High Performance Polymer) is based on polyether-ether-ketone (PEEK) polymer.
- Microstructure: Thermoplastic polymer network based on PEEK containing ceramic fillers of 0.3-0.5 microns and occupy 20% of the total volume of BioHPP.
- Their strength is due to the special ceramic filler (with the grain size of 0.3 to 0.5  $\mu\text{m}$ ).
- Modulus of elasticity of around 4 GPa, BioHPP is close to modulus of elasticity of bone, which helps in reducing the stress shielding thereby makes it a very interesting material for the restoration of dental implants. <sup>(6)</sup>

- **Metallic Matrix Composites**

In metallic matrix composites the matrix is a ductile metal, and the reinforcement may improve stiffness, strength, abrasion resistance, creep resistance, thermal conductivity, and dimensional stability. The reinforcement may be in the form of particulates, fibers, and whiskers. Concentrations normally range between 10 and 60 vol%.<sup>(2)</sup>

### Advantages over polymer matrix composites:

1. Metallic matrices have high toughness, stiffness, strength, and modulus.
2. Metals have high melting points. It can withstand high temperatures and sustain their shape at high temperatures.
3. Lower coefficients of thermal expansion and contractions.
4. Higher electric and thermal conductivities
5. Better wear & fatigue resistances.

### Disadvantages of metallic matrix composites:

- It has higher densities
- Need very high processing temperatures. <sup>(4)</sup>

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## • Ceramic Matrix Composites

It is composed of a ceramic matrix and embedded fillers of other material (dispersed phase; metal, ceramic, or polymer). Particulates, fibers, or whiskers have been embedded into a ceramic matrix. Different mechanical and optical properties have been improved significantly by this combinations. <sup>(2)</sup>

### Advantages:

- Enhancement of the toughness while retaining the high temperature properties.
- High melting points and superior corrosion resistance.

### Disadvantages:

- low tensile strength, impact resistance.

### Applications in dentistry:

Zirconia-reinforced Lithium Silicate (ZLS)  
(Vita Suprenity & Celtra Duo)

- The microstructure: The zirconia-reinforced lithium silicate (ZLS) is based on a lithium-metasilicate ( $\text{Li}_2\text{SiO}_3$ ) glass ceramic, reinforced with 10% of zirconium dioxide ( $\text{ZrO}_2$ )
- The final crystallization process, leads to the formation of fine-grained microstructure ( $\text{Li}_2\text{O-ZrO}_2\text{-SiO}_2$ ).
- ZLS combines the positive mechanical characteristics of the zirconia with the glass-ceramic aesthetic appearance. <sup>(7)</sup>

## II.b. Classification according to the shape of the dispersed phase

### Role of the dispersed phase in composite structures:

- 1) Enhance the mechanical properties of the final composite structure.
- 2) Reduce the amount of matrix material. Thus, decreasing the polymerization shrinkage in case of polymeric matrix and decreasing the cost.
- 3) Adjusting the rheological properties according to the application. Thus, improving its workability.
- 4) The use of heavy metals, such as barium and strontium provide radiopacity.

- 5) The fillers provide the ideal means of controlling various aesthetic features such as color, translucency, and fluorescence.

### 1. Particle reinforced composites:<sup>(2)</sup>

The dispersed phase for particle-reinforced composites is *equiaxed* (i.e., particle dimensions are approximately the same in all directions). Particle reinforced composites could be subdivided into **large-particle** and **dispersion-strengthened composites** the difference between both is the reinforcement or strengthening mechanism.

#### Large-particle composites

The term large is used to indicate that particle–matrix interactions cannot be treated on the atomic or molecular level.

The particulate phase is harder and stiffer than the matrix.

These reinforcing particles tend to restrain movement of the matrix phase in the vicinity of each particle.

The matrix transfers some of the applied stress to the particles, which bear a fraction of the load.

The degree of reinforcement depends on mechanical properties of the particle & strong bonding at the matrix–particle interface.

#### Nano-particle composite (Dispersion-strengthened composites)

Particles are normally much smaller (10 and 100 nm).

Particle–matrix interactions that lead to strengthening occur on the atomic or molecular level.

The mechanism of strengthening is similar to that for precipitation hardening. The small, dispersed particles hinder the plastic deformation.

Thus, plastic deformation is restricted such that yield strength, tensile strength, and hardness are improved.

### Factors influencing the effectiveness of reinforcement:

- 1) The particles should be small and evenly distributed throughout the matrix.
- 2) The volume fraction of the two phases influences the behavior; mechanical properties are enhanced by increasing particle content.

The **rule-of-mixtures** equations predict that the elastic modulus should fall between an upper limit repre-

$$E_c(u) = E_m V_m + E_p V_p$$

sented by

And a lower limit represented by

$$E_c(l) = \frac{E_m E_p}{V_m E_p + V_p E_m}$$

Where,  $E$  and  $V$  denote the elastic modulus and volume fraction, respectively, and the subscripts  $c$ ,  $m$ , and  $p$  represent composite, matrix, and particulate phases, respectively.

## Applications in dentistry:

### a) Fillers in dental adhesives:

Lohbauer et al. incorporated Zirconia nanoparticles into the adhesive of a commercial adhesive system with different concentrations (5, 10, 15 and 20 wt.%). It was found that Zirconia nanoparticles incorporation **increased bond strength to dentin** by reinforcing the interface adhesive layer. Moreover, nanofiller incorporation into the adhesive showed a tendency to increase bond strength with increasing concentration. At high concentrations (20 wt.%) nano-filler incorporation was more efficient in increasing bond strength.<sup>(8)</sup>

### b) Fillers in denture base:

Alhareb et al. mixed the powder particles of the PMMA with **nitrile butadiene rubber** (NBR) as impact modifier particles and **ceramic fillers** as reinforcement particles fixed at 5 wt% ( $\text{Al}_2\text{O}_3$  and YSZ).

It was found that the **impact and fracture resistance** of heat polymerized PMMA denture resin was enhanced after reinforcement with NBR particles and treated ceramic fillers. These properties can be improved by using silane coupling agent which offers the potential to further improved properties. This fillers combination offered resin with high impact strength and fracture toughness.

Thus, PMMA denture base reinforced by NBR & ceramic fillers is a good choice in removable prosthodontics.<sup>(9)</sup>

### c) Fillers in glass ionomer

When zirconia and alumina fillers were added to glass ionomer cement.

- Alumina fillers increased the compressive strength of the glass ionomer cement.
- Zirconia particles (agglomerates) above 50  $\mu\text{m}$  in the microstructure of the glass ionomer cement had a negative impact on compressive strength of the glass ionomer cement. As it acts as stress concentration areas.
- Alumina and zirconia containing GICs are promising restorative materials only if the appropriate particle size distribution is used.<sup>(10)</sup>

## **2. Fiber reinforced composites:**<sup>(11)</sup>

Fiber shaped fillers are axial particulates embedded in fitting matrices. The primary objective of these fillers is to obtain materials with high strength in conjunction with higher elastic modulus. However, the strength elevation is affected with the applied load transmitted from matrix to fibers, interfacial bonding between fiber-matrix, their relative alignment and nature of fiber scheming the overall material behaviors.

The alignment of fibers may however be continuous or random depending on the end applications. The choice of the fiber reinforcement and its fitting matrix also depends on application requirements. In recent years, the advent of composite technology has led to the development of different fiber reinforced composite systems via varying manufacturing methodologies to obtain advanced material behaviors.

## **3. Whiskers reinforced composites:**<sup>(2)</sup>

Whiskers have the following properties:

1. It has very thin single crystals
2. It has extremely large length / diameter ratios
3. It has high degree of crystalline perfection. Thus, nearly flawless.
4. It is exceptionally strong

Whiskers are not used extensively as a reinforcement medium:

- 1) They are very costly
- 2) The process of incorporating them in matrix phase is tedious

## Applications

### Whisker reinforced dental resin composite (DRC)

- Xu et al. studied the influence of the amount of silicon nitride ( $\beta$ -Si<sub>3</sub>N<sub>4</sub>) whiskers on the mechanical properties of DRCs:

The flexural strength and vickers hardness of the DRCs increased monotonically with the amount of added  $\beta$ -Si<sub>3</sub>N<sub>4</sub> whiskers.

The flexural strength of the whisker-filled DRCs were more than twice those of conventional SiO<sub>2</sub>-filled DRCs<sup>(12)</sup>

- According to the type of the whiskers:

**Silicon nitride** whiskers were helpful to improve the strength and toughness of the material

**Silicon carbide** whiskers improved the modulus and hardness of the material<sup>(13)</sup>

### 4. Urchin reinforced composites:

This morphology was designed to address the problem of **aggregation of whiskers**. Urchin-like fillers were developed which combine features of a whisker and a globule. These fillers were easier to disperse than whiskers. The fillers could be inserted into the resin matrix tightly through their spikes and improve the interfacial interaction between the resin matrix and the fillers.<sup>(14)</sup>

### 5. Nanotubes reinforced composite:

Nanotubes are hollow fibrous fillers with a high aspect ratio. They have the same strengthening and toughening mechanisms as fibrous fillers. Nanotubes vary in chemical composition, with the most widely studied being carbon nanotubes.

#### Disadvantages:

⇒ Severe CNT agglomeration

⇒ Poor interfacial interaction with the matrix. (non-reactive surface of the nanotubes)

- **Single-walled carbon nanotube (SWCNT)**

It has excellent mechanical properties. To improve interfacial compatibility with the resin matrix,

Zhang et al. firstly coated SWCNT with a layer of nano-SiO<sub>2</sub>, followed by modification using allyltriethoxysilane. The addition of modified SWCNT improved the mechanical properties of DRC, but it also had an adverse influence on the aesthetics of the material due to its dark color. <sup>(14, 15)</sup>

## 6. Halloysite nanotube (HNT)

- ⇒ It is a nanomaterial with characteristics of hollow tubular structure with high aspect ratio. The elastic modulus of HNT ranges between 230 ~ 340 Gpa. <sup>(16)</sup>
- ⇒ Chen et al. added silanized HNT to DRCs and found that small amounts (1 or 2.5% mass fraction) of HNT could be dispersed evenly in the matrix and improved the mechanical properties, while excessive HNT tended to form aggregates.
- ⇒ The impregnation of HNTs into dental resins/composites could result in **two opposite effects**: the reinforcing effect due to the highly separated and uniformly distributed HNTs, and the weakening effect due to the formation of HNT agglomerates/particles. <sup>(17)</sup>
- ⇒ Moreover, Utilizing the hollow structure of HNT, Liu et al. **loaded silver nanoparticles** into HNT and obtained DRC with excellent antibacterial activity and mechanical properties. The flexural strength of the DRC was increased by 25% when 3 wt% Ag-HNT was added. <sup>(16)</sup>

## 7. Porous fillers

- Porous fillers were introduced to solve the problem of **silane coupling agent degradation** by time which weakens the interfacial bonding between the fillers and the matrix. The porous fillers can form a stable physical interlocking with resin matrix through the pore structure. <sup>(18)</sup>
- The filler loading of DRC only reached 40 ~ 50 wt%, due to the high specific area of the porous fillers. This was increased by combining nonporous SiO<sub>2</sub> and mesoporous SiO<sub>2</sub>. <sup>(18)</sup>
- Wang et al. synthesized wrinkled mesoporous (WMS) SiO<sub>2</sub> nanoparticles with a size of ~ 500 nm (controlled filler pore size and uniformity). Compared with non-porous SiO<sub>2</sub> nanoparticles similar in size, the wrinkled mesoporous SiO<sub>2</sub> nanoparticles were more conducive to the improvement of flexural

strength of DRC. WMS are beneficial for creating **strong micromechanical interlocking** between the filler and the resin matrix during the photo polymerization process and enhance the **interfacial retention**. Consequently, the stress may be more efficiently transferred between the resin matrix and WMS, increasing the fracture resistance and other mechanical properties <sup>(19)</sup>

## 8. Glass flakes

Glass flakes are generally modified c glass fillers with high aspect ratio. The geometrical structure and the inherent properties of the glass flakes stands for:

- Improvement in the overall mechanical properties of the composite structure as compressive strength and hardness.
- Improvement in the aesthetic properties (better translucency when compared with other irregularly shaped particle reinforced composite) attributed to:
  - Flat surface of the glass flake which suppress light scattering.<sup>(18, 19)</sup>

## Applications in dentistry

Mohseni et al. studied that addition of silanized glassy flakes (GF) in spherical nanosilica particle containing dental composites.

The evenly distributed flakes among the spherical filler particles of the dental composites significantly improved the fracture toughness of the composite as it caused deviation of crack direction, which increased the energy required for fracture of the composites. The highest mechanical properties were observed for a dental composite sample with 15 vol % of GF. Further increase in the GF content resulted in a drop in the mechanical properties of the dental composite due to the stacking of the flakes on each other. <sup>(20)</sup>

## 9. Microcapsules

A microcapsule is a spherical particle with a polymer or glass shell that contains a healing liquid. Microcapsules are embedded into the matrix, upon exposure to forces induced by the propagating crack, it ruptures and releases the healing liquid that flows into the crack and meets the catalysts incorporated within the matrix leading to its polymerization, thus healing the crack.



### (C) SEM showing typical microcapsules

Huyang et al. developed a novel self-healing dental composite using silica microcapsules containing aqueous solutions of polyacrylic acids (silanated to the matrix) and a healing powder (strontiumfluoroaluminosilicate). Huyang et al. reported that the average healing efficiency reached up to 25% when 10 wt% microcapsules were added without affecting the mechanical properties.<sup>(21)</sup>

## 10. Laminar composites:<sup>(2)</sup>

- A **laminar composite** is composed of two-dimensional sheets or panels bonded to one another. Each sheet has a preferred high-strength direction.
- A multi-layered structure such as this is termed a laminate.
- Laminate properties depend on how the high-strength direction varies from layer to layer.
- There are four classes of laminar composites: unidirectional, cross-ply, angle-ply and multidirectional.

modulus of elasticity and strength of unidirectional laminates are highly anisotropic.

While, multidirectional can be fabricated to be most isotropic; degree of isotropy decreases with angle- and cross-ply materials.

Laminar composites

- (a) Unidirectional laminates
- (b) Cross-ply laminates
- (c) Angle-ply laminates
- (d) Multidirectional laminates

## 11. Sandwich Composites<sup>(2)</sup>

- It is constructed by sandwiching foam core between two skins of high strength laminates.
- Face sheets carry most of the loading and stresses. Core may be a honeycomb structure which has low density than the face sheets and resists perpendicular stresses and provides shear rigidity.
- A well-designed sandwich panel should be able to withstand both compressive, tensile and shear stresses without failure.

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## **II. c. Classification according to the interface** <sup>(23)</sup>

The interface is defined as the region where the filler and matrix are either chemically or physically attached to each other. Stronger interfacial interaction between the composite constituents can lead to a better isotropic stress distribution.

### **Types of interfacial interactions:**

The properties of the composite materials are dependent on the interfacial characteristics of the fillers and matrices. The interactions between the filler and matrix are categorized as **covalent** and **non-covalent** interactions.

### **Non-covalent interaction:**

(Van der Waals , electrostatic, and hydrogen bonding)

The noncovalent interaction between the matrix and fillers can be enhanced by employing:

- **Bridging:**It happens when a polymer chain interacts with two or more reinforcing fillers simultaneously. By increasing the filler content and using higher molecular weight polymer, the chance of bridging phenomenon will be higher.
- **Wrapping fillers by polymer chains:** It is useful for better dispersion of the fillers in the matrix
- **Crystallization of the semicrystalline polymer:** It improves the interfacial interactions. In this process, the fillers will act as a nuclei and the semi-crystalline host will crystallize at the interface.

### **Covalent interactions**

Covalent interaction happens when polymer chains are chemically connected to the reinforcing fillers. For that purpose, proper chemical treatments are required to attach:

- ⇒ Functional group to filler surface to react with the matrix.
- ⇒ Polymer chains to the filler
- ⇒ Different functional groups to the same filler using plasma treatment

### **Role of functionalized fillers:**

- 
- ⇒ Enhance the interaction between fillers and matrix
  - ⇒ Improve their dispersion
  - ⇒ Improve the final properties of the composites compared to conventional fillers. This could be achieved by in situ polymerization, where the monomers react with each other and the fillers simultaneously.

### **Influence of interface on mechanical properties**

The interface is important for the evaluation of the mechanical load transfer from polymer matrix to fillers. For instance, strength and toughness of the composites strongly depend on the interfacial adhesion.

#### **Strength**

The tensile strength of the composite depends on the efficiency of stress transfer between the constituents of the composite.

- By increasing the strength of interfacial bonding between the composite constituents. The applied load will be transferred to the fillers and the strength will be improved.
- The smaller particles have larger interface area at a constant volume fractions of fillers, leading to a large portion of stress transfer regions. Thus, increasing the final strength.

On the contrary, strength will be decreased in composites with poorly bonded fillers. This is due to the presence of discontinuity or de-bonding at the interface, which prevents the filler from carrying the applied load efficiently. This could be avoided by the introduction of chemical bonding to filler-matrix interfaces to effectively enhance the strength of composites.

#### **Toughness**

Toughness can be significantly enhanced by the enhancement of the interfacial adhesion between the thermoplastic matrix and filler.

- If the interaction between the matrix and fillers was weak. The crack will propagate through easily.
- If the interaction was strong. The crack pattern will be changed by crack deflection & debonding.

Liu et al.. studied the effect of silanization and BisGMA grafting on mechanical properties and polymerization shrinkage. The HA whiskers were firstly silanized, followed by the graft of poly(BisGMA) onto their surface. Compared with the HA whiskers without any treatment and with only silane treatment, the poly(BisGMA)-grafted HA whiskers showed better mechanical properties and lower polymerization shrinkage<sup>(24)</sup>

## **Evolution of fillers system in dental composite<sup>(22)</sup>**

In an attempt to overcome the drawbacks of the silicate and acrylic resin materials, composite restorative materials came out in the early 1960s. Over the years. Fillers have been modified in three main trends: size of fillers, type of fillers and fabrication technique.

In the early 1960s, **macro-filled resin composite** was introduced using irregular quartz particles with size ranging between (5-25 $\mu$ ). Quartz fillers were hard that cannot be cut in smaller size. Filler content was approximately 75 to 80 wt%. It showed physical & mechanical properties better than unfilled. But, poor surface finish, low wear resistance & more prone to staining and wear resistance. Later, fine filled resin composite was introduced with quartz filler particles (0.5-3 $\mu$ ). It showed good mechanical properties with superior finish and polish than macro-filled.

In late 1970s, **Micro-filled resin composite** was introduced. With filler particle size ranging between (0.04-0.2 $\mu$ ) formed by hydrolysis and precipitation of fine silica fillers allowing better polishability. Filler content was approximately 35-50wt%. microfiller particles exerted extremely high surface area limiting the filler loading . Lower filler load resulted in inferior mechanical properties as low modulus of elasticity, low tensile strength, high water sorption, high coefficient of thermal expansion & contraction and high polymerization shrinkage.

One way to increase the filler content and mechanical properties, was the addition of microfiller-based complexes or prepolymerized resin fillers into the resin matrix of microfilled composites. These organic-inorganic fillers were produced by milling the prepolymerized microfilled composite into particles (1–200  $\mu$ m) or by incorporating silica microfillers into prepolymerized spherical particles (20–30  $\mu$ m). Prepolymers were able to increase the filler content in microfilled composites up to 60 wt%. However, prepolymers could

not be covalently bonded to the resin matrix due to the lack of available C=C bonds at their surface resulting in low wear resistance in load-bearing areas.

Shortly after, in early 1980s, **hybrid composites** were introduced as a true combination of macrofilled and microfilled composites. They contained macrofiller quartz, glass, or (Ba/ Sr/Al/Zr)-silicate particles (1–50  $\mu\text{m}$ ) with amorphous silica microfiller particles (0.04  $\mu\text{m}$ ). Hybrid composites were considered an optimal combination for favorable mechanical and optical properties and improved wear resistance.

Quartz was being replaced with other types of fillers due to its high abrasiveness toward enamel and lack of radiopacity. Ba-glass and ytterbium/yttrium trifluoride fillers were added for radiopacity and potential fluoride release. However, it was shown that trifluoride-containing composite released significantly lower amounts of fluoride than a glass-ionomer cement excluding anticariogenic potential of such composites.

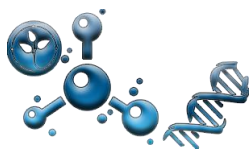
Around the year 2000, nanotechnology allowed further improvement of dental composites and the launch of nanofilled and nanohybrid composites. Nanofilled composites contain silica and/or zirconia particles (5–20 nm) in the form of non-agglomerated particle and agglomerated nanoclusters (avg. size = 0.6– 10  $\mu\text{m}$ ). Nanohybrid composites contain silica/ zirconia nanoparticles and larger 0.6–1  $\mu\text{m}$  glass/ zirconia/silica particles. Nanohybrid composites also contain prepolymerized resin fillers and nanoclusters.

The new sol-gel technology used to create nano-sized filler particles allowed greater nanofiller content (more than 80 wt%) compared to the traditional microfilled composites. This led to excellent optical properties, high polishability, and gloss. While their mechanical properties, hardness, strength, and wear resistance did not exceed microhybrid composites.

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Type of the Paper (Mini-Review Article)

## Dental Implant–Tissue Interface

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**Abstract:** The biomechanical interface between implant and tooth surfaces are critical for both the soft and tissue tissue integration. Here, the current review discuss the clinical importance of these dental implant–tissue Interface.

**Keywords:** Interface; surface; dental implant.

### Introduction

Main factors affecting materials –tissue interface (General Guidelines). Factors greatly affect the materials–tissue interface include material surface properties and processes at such interface.

#### a) Material Surface Properties

The surface of a material is a termination of an extended; three-dimensional structure. Thus, generally represents an increase in energy due to unsaturated bonds (the surface energy). If there is a reactive environment at the surface, the bonds react to form new bonds and compounds, to *lower its surface energy*.<sup>(1)</sup>

If the reacted surface is placed in another new environment, it is likely to react again to lower further the energy of the system. There is thus a *built-in thermodynamic driving force* for reactions.

An *example* here is titanium, which is used for dental implants and in orthopedic devices. When fresh titanium is exposed to air it reacts rapidly with atmospheric oxygen to form a surface oxide which is typically a few nanometers thick. The oxide stoichiometry is approximately TiO<sub>2</sub>. The surface is never perfectly clean TiO<sub>2</sub>, for the TiO<sub>2</sub> terminated surface tends to bind molecules or atoms from the surroundings as a mono-molecular layer. Even *inert surfaces* such as gold or diamond tend to lower their surface energy by suitable terminations on the *atomic scale*. For example, diamond may terminate by C-H bonds at the surface.<sup>(1)</sup>

At the tissue-material interface, surfaces properties such as *chemical composition*, *wettability*, and *topography* play a determinant role in the biological interactions, particularly the *microstructure* and *surface roughness*.

#### b) Processes at the Material-Tissue Interface

The molecular events at the material-tissue interface involve *small molecules*, like water. The molecular events also involve *larger molecules* like proteins. Eventually the larger structures like *cells*, which have lower mobility,

will reach the surface with its organic over-layer. Depending on the nature of the material surface and its organic coating, cells react differently to different biomaterials.

### Dental Implant–Tissue Interface

One of the challenges in implantology is to achieve and maintain the *osseointegration* as well as the *epithelial junction* of the gingiva with implants. An intimate junction of the gingival tissue with the neck of dental implants may prevent bacteria colorizations which cause peri-implantitis while direct bone bonding may ensure a biomechanical anchoring of the artificial dental root.<sup>(1-5)</sup>

The first step of the osseointegration of implants is called *primary stability* and is related to the mechanical anchorage, design of implants, and bone structure. This primary interlock decreases with time at the benefit of the *secondary anchorage*, which is characterized by a biological bonding at the interface between bone tissues and implant surface. *Between* the primary mechanical and secondary biological anchorage, a decrease of implant stability could be observed.<sup>(6)</sup>

Many studies have attempted to enhance the osseointegration of implants by *various surface modifications*. The aim is to provide implants with surface biological properties for the adsorption of proteins, the adhesion and differentiation of cells, and tissue integration. However, the control of these surface properties at the protein and cell levels, thus in the nanometer range, remains a challenge for researchers and dental implants manufacturers.

*Nanotechnologies* may produce surfaces with controlled topography and chemistry that would help understanding biological interactions and developing novel implant surfaces with predictable tissue-integrative properties<sup>(6,7)</sup>. Various surface treatments such as lithography, ionic implantation, anodization, and plasma treatments may be applied to the surfaces of dental implants to produce controlled features at the nanometer scale. These surfaces may then be screened by using high throughput biological assays *in vitro*. For instance, specific protein adsorption, cell adhesion, and differentiation of stem cells should be studied in relation to the surface properties. This approach may define the ideal surface for a specific biological response. Following *in vitro* screening, nanostructured surfaces may then be tested in animal models<sup>(4)</sup>.

#### **a) Interactions of Surface Dental Implants with Blood**

During surgery, blood vessels are injured and, thus, dental implant surfaces interact with blood components. Various plasma proteins get adsorbed on the material surface within a minute, depending on the surface properties of the material<sup>(8)</sup>. A hydrophilic surface is better for blood coagulation than a hydrophobic surface. Consequently, dental implants manufacturers have developed high hydrophilic and rough implant surfaces for better osseointegration<sup>(9)</sup>. Adsorption of proteins such as fibronectin, vitronectin on surface of dental implants could promote cell adhesion by *cell-binding RGD domain* (arg-gly-asp). This RGD sequence interacts with integrin present on the cell membrane<sup>(10)</sup>. Interactions between cell membrane integrins and proteins coated onto



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implant surface play a key role in adhesion of many cells types. After proteins absorption, the osseointegration is characterized by platelets adhesion and fibrin clots formation at the injured blood vessels site.<sup>(4)</sup>

There is a controversy about the role of platelet-rich plasma on the osseointegration. It has been shown that implants in contact with platelet-rich plasma (PRP) with a platelet concentration of approximately 1,000,000 platelets/ $\mu\text{L}$  have a positive effect on osteointegration. At lower concentrations of PRP, the effect was not optimal, while higher concentrations resulted in a paradoxically inhibitory effect of bone regeneration. Other studies were not in agreement with this PRP beneficial effect on the osseointegration of dental implants.<sup>(11)</sup>

The assessment of bioactivity of surface-treated dental implants should be tested *in vitro* using biological fluids containing blood components<sup>(12)</sup>.

## **b) Interactions between Surfaces and Mesenchymal Stem Cells**

Following blood clotting around dental implants, several cells interact with surfaces for tissue healing. Mesenchymal stem cells (MSCs) attracted to the injured site by chemotactic factors have a determinant role in peri implant tissue healing.

### **b-i) Origin of Mesenchymal Stem Cells**

Mesenchymal stem cells (MSCs) are stem cells derived from somatic tissue which can be differentiated into mesenchymal lineages such as bone, cartilage, fat, and skin. In addition, MSCs are present in blood at low concentrations serving as a sort of internal repair system.

Mesenchymal stem cells are distinguished from other cell types by two important characteristics. *First*, they are unspecialized cells able to renew themselves through cell division, sometimes after long periods of inactivity. *Second*, under certain physiologic or experimental conditions, they can be induced to become tissue- or organ-specific cells with special functions. Thus, MSCs have high proliferative and multipotent capacity leading to differentiated cells.<sup>(4)</sup>

### **b-ii) Migration, Adhesion, and Proliferation**

Cell migration, adhesion, and proliferation on implant surfaces are a prerequisite to initiate the tissue regeneration. Authors have shown that some factors present in tissues and secreted during the inflammatory phase are

able to attract MSCs to the injured site<sup>(13, 14)</sup>. MSCs migration and proliferation were stimulated by many growth factors<sup>(15-19)</sup>. These factors are certainly released in the injured sites by cells involved in tissue healing. Furthermore, plasma clot serves as storage to fibrin molecules and releases system for a variety of bioactive factors including growth factors that attract and differentiate MSCs into specific lineages<sup>(20-22)</sup>.

After MSC recruitment in the injured site, cells adhere on the local extracellular matrix as well as on the implant surface beginning an extensive proliferation in order to build up new tissue. Again, surface modifications of implants in the nanometer range condition the biological responses.<sup>(4)</sup>

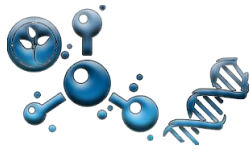
### **b-iii) Differentiation**

MSCs are stimulated by *some specific factors* to differentiate into the adequate cell line. Under the influence of these factors, MSCs switch to osteoblastic cells in contact to bone tissue while they differentiate into fibroblastic lineage in the gingival tissue region. These two differentiation pathways are in concurrence around dental implants. In some cases, implants are encapsulated by fibrous tissue due to the proliferation and differentiation of MSCs into fibroblastic cells causing implant failure<sup>(23)</sup>. However, fibroblasts adhesion and proliferation have been shown to be lower on nanoscale implant surface<sup>(24-26)</sup>.

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## Dental Composite/ Adhesives –Tooth Interface

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**Abstract:** The fast progress in dental adhesive technology has extensively influenced modern restorative dentistry. Although decayed and fractured teeth can be reconstructed minimal invasively and nearly invisibly using adhesive technology, the clinical longevity of composite restorations is today still too short. Notwithstanding the enormous advances made in adhesive technology during the last 50 years, the bonded interface remains the main challenge of an adhesive filling.

**Keywords:** Dental; composites; adhesives; Interface; surface.

### Introduction

The fast progress in dental adhesive technology has extensively influenced modern restorative dentistry. Although decayed/fractured teeth can be reconstructed minimal invasively and nearly invisibly using adhesive technology, the clinical longevity of composite restorations is today still too short <sup>(1,2)</sup>. Despite the enormous advances made in adhesive technology during the last 50 years, the bonded interface remains the main challenge of an adhesive filling <sup>(3,4)</sup>. Modern adhesive approaches include (1) etch & rinse, (2) self-etch.

#### a- Etch & rinse

The multi-step etch&rins approach involves a phosphoric acid-etch step that at enamel produces deep etch-pits in the hydroxyapatite (HAP)-rich substrate, and at dentin demineralizes up to a depth of a few micrometers to expose a HAP-deprived collagen mesh. The next step involves either the application/curing of a combined primer/adhesive resin, or a separate primer and adhesive resin step. The final objective is to micro-mechanically interlock upon diffusion and *in situ* polymerization of monomers into the enamel etch-pits, the opened dentin tubules and the exposed collagen network, the latter forming the well-documented hybrid layer.

Without doubt, the micro-mechanical interlocking of tiny resin tags within the acid-etched *enamel* surface is still today the best achievable bond to enamel <sup>(5)</sup>. It not only effectively seals the restoration margins on the long term, but also protects the more vulnerable bond to dentin against degradation <sup>(6)</sup>.

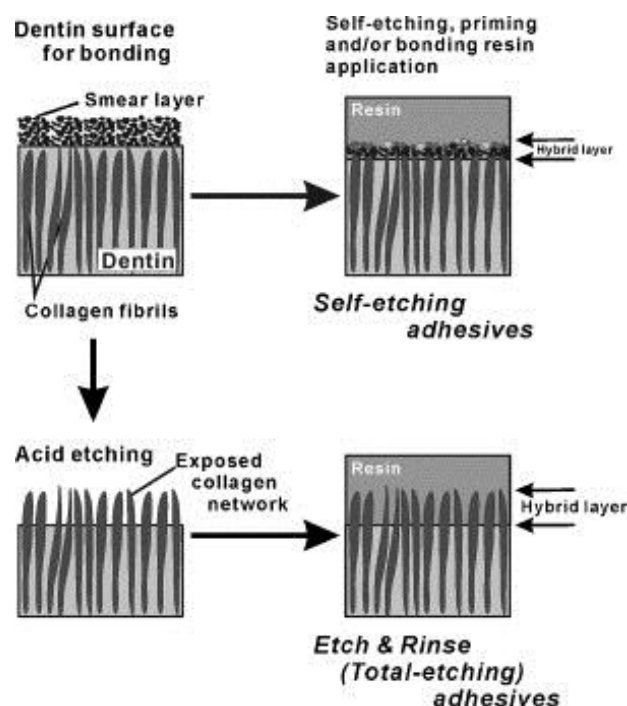
On the contrary, etching *dentin* is a rather aggressive procedure as it dissolves and removes (through rinsing) the natural protection of collagen, thereby producing a resin–collagen complex that is vulnerable to degradation upon water sorption, possibly enhanced by the documented enzymatic degradation process <sup>(7-9)</sup>. As the most intimate and stable intermolecular interaction possible, primary chemical interaction between resin and the mainly organic substance remaining at acid-etched dentin would definitely contribute to the bond durability but

is however lacking<sup>(10,11)</sup>. This deficient chemical interaction should most likely be regarded as the major short-coming of today's etch & rinse approach. Nevertheless, traditional 3-step etch & rinse adhesives are still today regarded as 'gold-standard'<sup>(12)</sup>.

### *b- Self-etch*

The self-etch approach can be further subdivided into a 'strong' (pH < 1), an 'intermediately strong' (pH≈1.5), a 'mild' (pH≈2), and an 'ultra-mild' (pH≥2.5) self-etch approach depending on the self-etching or demineralization intensity<sup>(13)</sup>.

Self-etching only dissolves the smear layer, but does not remove the dissolved calcium phosphates, as there is no rinse phase. In particular '**mild**' (pH≈2) self-etch adhesives appear to deal reasonably well with bur-smear, producing a submicron hybrid layer with substantial HAP-crystals still protecting the collagen fibrils.



**Schematic illustration of bond structure of a self-etching system and etch-and-rinse system (total-etching system).**

Figure Reference: [Hashimoto M et al: A review: Biodegradation of resin–dentin bonds. Japanese Dental Science Review Volume 47, Issue 1, February 2011, Pages 5-12](#)

**Important:**

Functional monomers, in particular like 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), have been proven to interact with this residual HAp through primary ionic binding<sup>(14, 15)</sup>. The resultant two-fold micro-mechanical and chemical bonding mechanism closely resembles that of glass-ionomers<sup>(16-20)</sup>. However, chemical bonding potential on its own is insufficient; the formed ionic bonds should also be stable in an aqueous environment. Chemical bonding promoted by 10-MDP appeared not only more effective, but also more stable in water than that provided by other functional monomers like 4-MET (4-methacryloyloxyethyl trimellitic acid) and phenyl-P (2-methacryloyloxyethyl phenyl phosphoric acid), in this order<sup>(15)</sup>.

### *Examination of Restorative Material-Tooth Interface*

As the longevity of an adhesive composite restoration is mainly affected by leakage of oral fluids along the interface between restorative material and tooth substrate<sup>(21, 22)</sup>. Examination of this interface included micro-leakage, marginal adaptation/gap formation, bacterial leakage permeability, nano-leakage and 3D-leakage. Acoustic emission and Micro-CT have been used for non-destructive examination of interfacial debonding<sup>(23)</sup>.

Bond durability can be measured using bond strength test (macro or micro test, basically depending upon the size of the bond area), measured in 'shear', 'tensile', or using a 'push-out' protocol. Despite the importance of laboratory studies attempting to predict clinical performance of biomaterials, clinical trials remain the ultimate way to collect scientific evidence on the clinical effectiveness of a restorative treatment<sup>(24, 25)</sup>. Clinical effectiveness of adhesives should best be determined using Class-V clinical trials.

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