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Type of the Paper (Editorial)

Utilizing Nanostructures to Provide the Best Dental Care

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Abstract: Nature is the riddle and pursuing and unfurling that secret is human instinct. In biomimicry, we take a gander at nature as model, measure, and tutor. Biomimicry should be possible by utilizing either natural substitutes or synthetic substitutes. Dental restorative materials developed, in the past have been created on the micro molecular level of matter; however, the interests of researchers have shifted towards similarity to nature by construction of matter at the nano-size hence the field nanotechnology.

Keywords: Nanostructure; dental care; nanotechnology.

Human instinct drives us to seek out and unravel nature's mystery. In biomimicry, we use nature as a guide, benchmark, and teacher. Using either artificial or natural alternatives should make biomimicry practicable. In the past, dental restorative materials were generated at the micromolecular level of matter, but now researchers are more interested in creating materials that are more like nature by building them at the nanoscale, giving rise to the discipline of nanotechnology.

The most fundamental components of life systems are what biomimetic nanotechnology identifies with, as is the transfer of these components' qualities to useful human uses. The majority of natural materials, structures, and processes are thought to have nanoscale functions. From their first degree of association, the most fundamental traits and abilities of every single natural framework are characterised at the nanoscale.

The main idea of nanotechnology is to use low-energy bonding to hierarchically arrange molecules into objects. Materials and techniques for creation and nanoscale analysis are provided by nanotechnology.

In my opinion, all modern dental materials, especially their active ingredient, should be made at the nanoscale for the best possible replication of natural dental tissues. Nanoscale will guarantee that mechanical qualities are best suited to their intended use, with the best aesthetics, repairability, and energy efficiency, making materials intelligent in their response to their surroundings.

All branches of science have quickly embraced nanotechnology, which presents important alternatives for addressing issues in science and medicine. With some notable success, nanotechnology has been used in dentistry to create restorative materials. This paper examines nanointerfaces that may jeopardise the durability of dental restorations and how nanotechnology has been used to change them to provide long-lasting, effective restorations.

Additionally, it focuses on several difficult dental problems, such as oral biofilm and malignancies, and how nanotechnology solves these problems. Recent developments in

nanodentistry and novel diagnostic, preventive, and therapeutic approaches to oral health, which are necessary to achieve and maintain optimal oral health, have been discussed. The most recent developments in nanotechnology show promise for bringing about a paradigm shift in the dental industry. The cost of synthesis and implementation must be carefully considered when using any of the various complicated medicines being developed to treat a wide range of ailments.

Clinical dental practise will change as a result of nanotechnology. Dental restoration materials will soon be extremely accurate, intelligent, and similar to natural ones. Consequently, every effort should be made to produce dental restorative materials, such as dental medication, resin composite, cements, sealers, ceramics, impression materials, remineralizing agent, dentures, bone replacement agent, root fillings, and dental implant materials, in the nanoscale.

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Type of the Paper (Review Article)

Factors affecting the durability of adhesive junction :a review

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Abstract:. Recently, the durability and serviceability of dental restoration is a concern not only to the dentist but also to the patient. Although there are huge improvements and modifications in the adhesive system, several factors still control the success and stability of the adhesive junction**.**

Keywords: *adhesive junction, durability, degrdation and aging, enhancement.*

Introduction

The conservative approach of restoring tooth structure in both function and esthetic is the prime objective in the dental field. This approach mainly depends on using adhesive to bond tooth-colored restoration to the tooth structure either directly or indirectly. Although there are huge improvements and modifications in the adhesive system, several factors still control the success and stability of the adhesive junction. (1)

Clinical longevity is a critical aspect because the adhesive interface deteriorates over time. Marginal leakage is the primary factor in filling failure. it may cause marginal discoloration, secondary caries, and a loss of retention. The majority of the existing dental adhesives show excellent short- and immediate-term bonding effectiveness, but the longevity and stability of resin-bonded surfaces are still questionable. (2)

The fundamental factors affecting the durability of the adhesive junction are the tooth substrate, the dentin hydrophilicity, the adhesive material characteristic, and their physicochemical properties. Some factors related to the clinical procedure include tooth preparation, handling and application of restoration, and curing mode. While patient-related factors such as preventive and oral hygiene measurements after

treatment. (3,4)

The basic technique for bonding to enamel and dentine is mainly based on an exchange process. In which minerals taken out of dental hard tissues are substituted by resin monomers. This resin monomer with polymerization will

interlock the produced pores micromechanically. The adhesive interface is liable to degradation and is considered an area of weak bonding that affects the adhesive junction's durability and restoration. [3]

Factors affecting the durability of adhesive junction

1. **Bonding tooth substrate (Enamel and Dentin)**

The tooth consists of two main structures. Enamel is responsible for stiffness, hardness, and brittleness characters. While dentin forms the main bulk of the tooth and protects the pulp. Also, it makes the tooth flexible and softer and can withstand stresses from chewing without fracture. During tooth structure replacement, it is difficult to achieve stable bonding due to the difference between enamel and

dentin. [3]

Physicochemical properties of enamel and the acid etching effect:

Enamel mainly consists of a high mineral content of 90 vol% mineral, 4 % protein, and 6 % water. Insufficient roughness to the enamel surface may result in poor adhesion to enamel. That is why acid etching is a critical step that depends on using a suitable etchant (phosphoric acid). With several modifications, nowadays the etchant concentration ranges from 32 to 37 %, and the time of application range from 15-30 second. [3]

Acid etching is used to remove organic pellicles in enamel. Also, it removes the smear layer which is composed of debris and bacteria from cutting the enamel surface during caries removal. The etch will remove enamel rods leaving micropores, resin infiltrates to form micromechanical interlocking by forming a resin tag which will provide a stable resin enamel bonding interface. [3]

Physicochemical Properties of dentin:

Adhesion to the dentin surface is challenging and more complex because of the dentin composition. Dentin is composed of 50% vol mineral and 30% organic matrix and 20% water. The organic part is divided into 90% collagen type I and 10% non-collagenous protein. Dentin consists of dentinal tubules that are not regularly distributed. The dentinal tubules are responsible for the wet characteristic of dentin. The fluids increase by cutting tooth structure. [5]

Additionally, by applying an acid etch the minerals in dentin will solubilize and interchange with water. This led to raising water content to 70%. Therefore, displacing water is mandatory to obtain the adhesion of the resin to dentin this may be done by using the liquid mixture of solvents and adhesive monomers. [6]

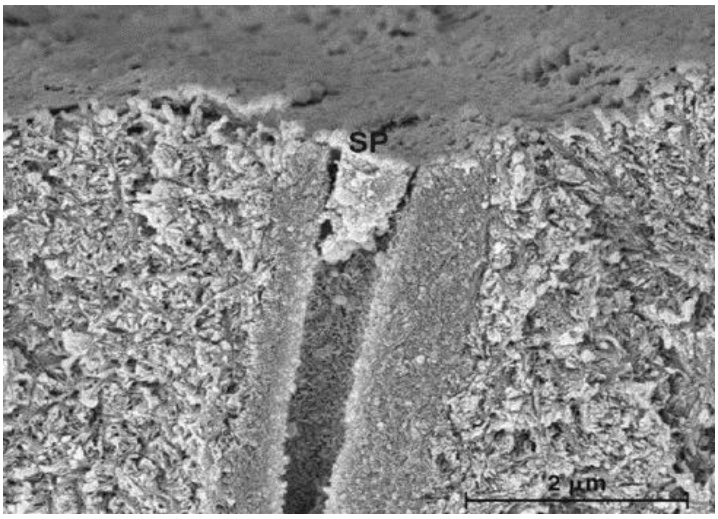
Dentin demineralized and give collagen fibrils that engage with the adhesive resin by using a light cure to polymerize it to form a hybrid layer. Although dentin shows high bond strength at the beginning, it is reduced with

time. this is because of increasing water sorption due to the nature of dentin, the amount of remaining water, and the hydrophilicity of the adhesive. [7]

The dentin smear layer:

The smear layer is formed during cavity preparation and carious removal in the dentin part plugged into the tooth structure. It is difficult to be removed by washing or blowing it with air. There is a point of argument on whether to remove the smear layer or not. As some studies declare that some bonding agents show the ability to be used over it as it contains calcium. While others claimed that the bond strength improved when the smear layer is removed. It can be removed by using suitable acid etching. Also, it can be modified by using adhesive resin with mild acidity to penetrate it and bond to intact dentin. The smear layer may interfere with good bonding. (**Figure.1**)[1,3]

Figure.1 Scanning electron micrograph of a smear plug blocking the entrance of a dentinal tubule.



2. Adhesion mechanism technique

The main concept of the adhesion technique to tooth substrate depends on exchanging the inorganic content with synthetic resin in two phases. The first phase is based on creating micropores in the enamel and dentine surface. This can be done by removing the calcium phosphate by using suitable acid. While the second phase is based on the infiltration of the resin to the created pores to form hybridization. [8]

There are several clinical strategies for adhesion as etch and rinse or self-etch approach. Although the etch-and-rinse is considered the gold standard approach, the self-etch eliminates the technique sensitivity of the other approach. For instance, over-etching and drying. Etch-rinse can be classified according to the technique steps into three steps:

separate etching and rinsing, primer, and adhesive application. While the two steps approach is simpler it involves etching and rinsing as a step and the primer is added to the adhesive and considered as one step. [6,9]

The self-etch approach is accomplished by using a system of acidic monomers (such as phosphoric acid or carboxylic acid esters) that act on tooth structure without rinsing. It can be classified into two steps: the etch and primer in one step and the adhesive resin in the second step. While one step is known as all-in-one as it relays on using a single compound of etch, primer, and adhesive resin as one step. [3,8,9]

Although self-etch provides a user-friendly adhesive system by reducing the number of steps, the quality of mechanical interlocking is lower than the etch and rinse technique. Also, some of the adhesive monomers may combine with the remaining hydroxyapatites. This will result in a weak, non-stable adhesive junction. [3]

3. Hydrolytic degradation of the adhesive

Vital dentin is inherently wet, and complete drying of dentin is difficult to be achieved clinically. Water has been considered an obstacle to attaining an effective adhesion of resins to dentin. Therefore, certain modification of bonding involves a wet bonding technique to inhabit collagen matrix collapse. Many adhesives combine hydrophilic and hydrophobic monomers in the same bottle, dissolved in an organic solvent such as ethanol or acetone. [9]

The hydrophilic monomer such as hydroxy ethyl methacrylate (HEMA). Its hydrophilicity promotes adhesion and enhances bond strength. On the other hand, it may absorb water leading to dilution of the monomer before curing. Also, it is liable to hydrolysis because of the presence of an ester group.[3,8,9]

In addition to hydrophilicity, water, as well as organic solvents (ethanol/acetone) trapped inside the hybrid layer, can degrade their integrity. The remaining solvent and water are difficult to be removed and compromise the bond strength as well as the hybrid layer. The absence of a hydrophobic resin seal and the hydrophilicity of water may trap the adhesive layer forming a “water tree” pattern that is more prominent by silver nitrate tracer and considered as nano-leakage. This problem is evident in the self-etching “all-in-one” approach. (**Figure**.**2**) This will decrease bond durability.[1,3,4,6]

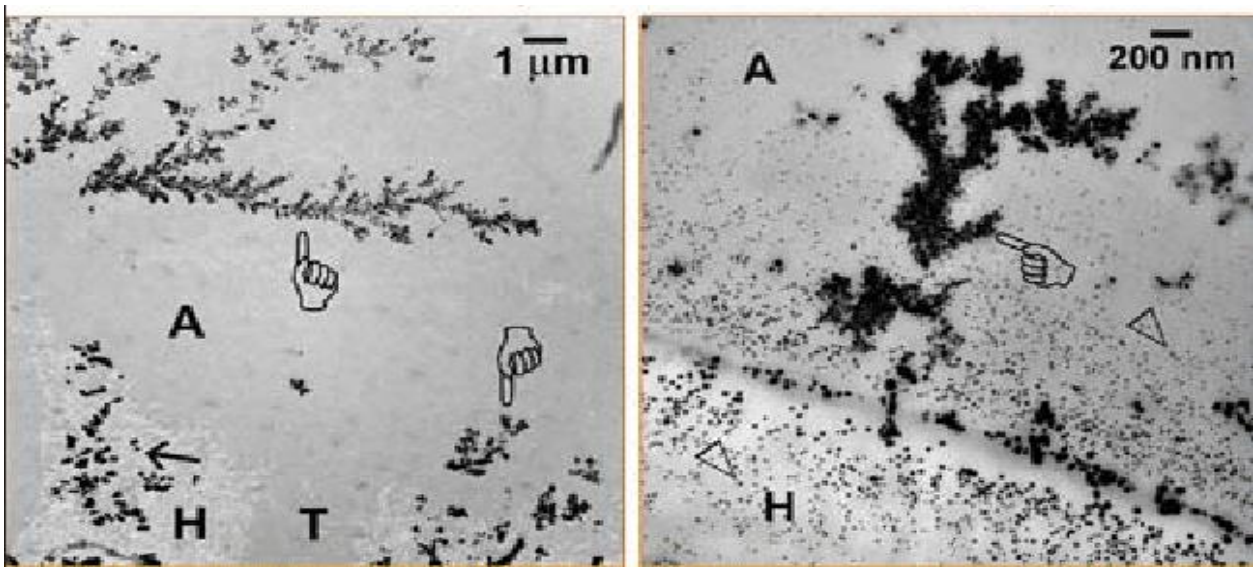


Figure.2 Transmission electron micrograph showing "water tree" pattern detected by using silver nitrate tracer.

4. Degradation of exposed collagen fibrils

The degradation of resin and collagen could be due to increasing the moisture content of the bond interface, which reduces the longevity of the bond.

Water is said to be one of the main causes of the breakdown of collagen. The hybrid layer degradation patterns can be observed as resin loss and disassembly of interfibrillar spaces and collagen fibril. (**Figure.3**) The hydrolysis will cause the deterioration of resin and/or collagen. Hence, reducing the physical properties of the resin-dentin bond.[2,3,9]

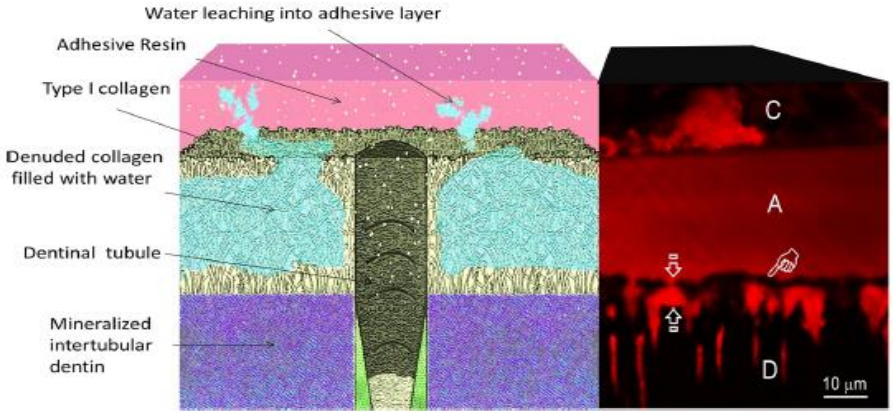


Figure.3 (a) 3D schematic showing the denuded collagen filled with water. (b) confocal laser scanning showing resin-dentin (black region) while water (red region) distributed in the hybrid layer C: composite resin A: adhesive D: dentin. [3]

5. Collagenolytic Enzymes in Dental Tissues

Matrix metalloproteinase (MMP)is an enzyme that plays a role in hybrid layer degradation. MMPs are a family of endopeptidases, a class of zinc and calcium that can degrade the extracellular matrix components. MMP has several species found normally in human dentin such as MMP-2, MMP-8, MMP-9, and MMP-20. Collagen is mainly attacked by MMP-8, while gelatin is attacked by MMP-2 and MMP-9.[2,3,5]

The host-derived proteinases are present in dental caries, erosion, and periodontal pathogen. They show a potent decrease in dentin bonding. Some studies declared that proteolytic enzymes in absence of bacteria can slowly degrade the dentin matrices. MMPs contain a catalytic domain composed of cysteine which is responsible for the activity of proteolytic enzymes. It was found that the activity of both MMPs and cysteine cathepsin is higher and more prominent in acidic environments and carious dentin than intact dentin.[2,3,5,8]

6. Polymerization shrinkage

Polymerization shrinkage is one of the huge problems facing resin restoration. Because of the contraction stresses created by curing the restoration. These stresses may result in microleakage and failure of the adhesive junction. It is more prominent at the interface if the contraction stresses are higher than dentin bond strength. [4]

7. Aging and quality of the hybrid layer

The quality of the hybrid layer is one of the critical factors affecting bond durability. Certain nano spaces may be created and lead to nano-leakage which will reduce the bond strength. Also, this may lead to the degradation of the adhesive junction. [2,4]

The aging of the hybrid zone relies on certain physical and chemical factors. Physical factors related to the force of occlusion with the chewing process. Besides, continuous shrinkage and expansion from temperature fluctuation within the oral environment will interfere with the adhesive interface's durability. Acidic chemical factors that come from food, beverages, and bacterial product in saliva may interfere with the durability of the tooth/ restoration interface. This may give rise to certain deterioration of collagen fiber and resin content. [4]

Clinical factors affecting the durability of the adhesive junction

Some clinical factors may control the longevity and stability of adhesive junction and restoration. This is related to cavity preparation, the quality of the remaining tooth structure, and the complete removal of the carious lesion. It was found that bond strength to intact dentin is higher than to carious affected dentin. Caries propagation may lead to mineral contents reduction and distribution of collagen and non-collagenous protein structures. This will result in a decrease in mechanical properties and adhesive junction durability. [4]

Proper cleaning of tooth structure and removal of plaque and calculus is critical to improving adhesive durability. Also, tooth dehydration from over-drying may affect adhesive durability. As it may result in a collapse of dentinal collagen and prevent the hybrid layer formation. [4]

Intra-oral contamination and improper isolation before the adhesive application can cause a decrease in adhesive junction durability and failure of the restoration. Therefore, before adhesive application, it is critical to avoid salivary or blood contamination. [4,5]

It was found that salivary contamination may decrease bond stability. Although it is a point of debate as some studies claimed that saliva is not affecting the adhesion. But more analyses are needed to declare the effect of salivary contamination on long-term bond strength. Sometimes contamination may be also from oil leakage of the handpiece affecting the adhesive junction's durability. [4]

Some studies claimed that the degree of surface roughness may affect bonding. The irregularities created depend on the type of bur used in cavity preparation as well as the size of the abrasive particles. It will increase mechanical retention and restoration durability. [4]

Methods to enhance the durability of the adhesive junction:

1) **MMPs inactivation and inhibition**

There are several strategies to counteract the effect of MMPs either by inactivation or inhibition. Some studies claimed that cross-linking agents are considered as a method for MMPs inactivation either by using glutaraldehyde, carbodiimide HCL, or riboflavin. The use of glutaraldehyde is limited because of its toxic effect. [5,10]

MMPs inhibitor may be used as chlorohexidine gluconate 2% which is an antiseptic and antimicrobial agent used widely in dentistry. It is recommended to be applied after the acid etching step and before the primer and adhesive application. Other MMP inhibitors may be used such as benzalkonium chloride (BAC) used as a cavity desensitizer. [5,10]

Furthermore, tetracycline is used as an antimicrobial agent. This may improve the durability of dentin bonding. MDPB is 12-Methacryloyloxydodecylpyridinium bromide may be added to inhibit MMPs. It preserves the hybrid layer and prevents the loss of bond strength to improve bonding durability. [5]

2) Removal of carious affected dentin

It is important to remove all the carious lesions on the dentin. To provide a proper marginal seal to the intact tooth structure. This will result in preventing caries progression and the formation of secondary caries. [4,5,10]

3) Chemical bonding

There are some approaches to adding functional monomer to the adhesive resin such as 10-methacryloyloxidecyldihydrogen phosphate (10-MDP). This functional monomer plays a role in increasing the chemical bonding to hydroxyapatite present in tooth structure. Some functional monomers may be added to mild self-etch adhesive systems such as N, N- Di ethanol p-toluidine (phenyl-P). This tends to enhance adhesion durability. [5,10]

4) Biomimetic remineralization

One of the latest approaches is to mimic hard tissue mineralization. This can take place by exchanging water matrix with appetite crystals in the hybrid layer. It is a process to increase mechanical properties by replacing water with minerals. Some polyanions such as polyacrylic and poly aspartic acid are used to merge with collagen and regulate physiological mineralization. This gives calcium a chance to bind and promote appetite nucleation. [5]

Some in vitro studies declared that the biomimetic remineralization strategy shows a great effect on enhancing bond strength and durability with time. Although several investigations, research, and developments for clinical application are needed. To prevent the loss of the hybrid layer integrity. [5]

5) Strategies to decrease the hydrolytic degradation of the adhesive

It is advised to use ethanol to remove the residual water, because of its high vapor pressure. Ethanol shows an effect in increasing the resin infiltration and prevention of monomer phase separation. Although it shows enhancement, especially in wet bonding, it is a sensitive technique. It requires complete evaporation as it may affect the polymerization process. [3]

Long-term adhesion to the tooth structure

It is advisable to use the selective etching technique by applying the etch on the enamel. Also, a certain conditioner is used on the smear layer and intact dentin to form exposed collagen. Hence allow space for resin infiltration. It is preferred to use MMP inhibitor or inactivator to extend the durability of the restoration and prevent hydrolytic degradation. Crosslinking agents such as DMSO may be used to make the exposed collagen matrix stiffer, and prevent its degradation. [3,5]

Conclusion

There are several factors controlling the durability of the adhesive restoration. Tooth substrate is one of the important factors as bonding to dentin require certain modification in the clinical procedure. Research and improvement in resin monomer infiltration are required. Regardless of the huge enhancement in the resin adhesive material, it is still essential to understand the process of bonding properly. [3,5]

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Ductility, brittleness, and fracture toughness

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Abstract: There are many important mechanical properties related to dental materials like ductility, brittleness, and fracture toughness. Those properties describe the resistance of materials to plastic deformation, crack propagation, and fracture under the applied force. It is important to predict how dental materials react to forces (masticatory and fabrication) to prevent their early fracture and prolong their proper clinical performance.

Keywords: *ductility, brittleness, fracture toughness, failure.*

Introduction:

There are many important mechanical properties related to dental materials like ductility, brittleness, and fracture toughness. Those properties describe the resistance of materials to plastic deformation, crack propagation, and fracture under the applied force. It is important to predict how dental materials react to forces (masticatory and fabrication) to prevent their early fracture and prolong their proper clinical performance.

Failure of prosthetic and restorative dental materials depends on both their mechanical properties and microstructure; the mechanical properties are defined by the law of mechanics as the physical science that deals with the energy and forces and their effects on the bodies.

According to the magnitude of the applied force, the amount of the induced stresses, and the nature of the materials (type and nature of the interatomic bonding), the material could deform elastically (temporarily) or plastically (permanently). [1, 2] Generally, most metals behave in a ductile manner while glass and ceramics materials tend to behave in a brittle manner. [1]

According to the type of bonding. In metals, their metallic bonds allow the atoms to slide past each other easily. The sliding of rows of atoms results in slip, which allows the metal to deform plastically instead of being fractured.

On the other hand, the presence of ionic bonds in ceramics results in such sliding motion resistance. Since in ionic bonding, every other atom is of opposite charge, when a row of atoms attempts to slide past another row, positive atoms

encounter positive atoms and negative atoms encounter negative atoms. This results in a huge electrodynamic repulsion which inhibits rows of ceramic atoms from sliding past other rows Accordingly, ceramics cannot plastically deform. Instead, they fracture in a brittle manner.

Ductility

Ductility is defined as the relative ability of metals or alloys to withstand vast permanent deformation by a tensile load to the fracture point, the metal can be drawn into thin wires. While **Malleability** is a Latin word that comes from malleus or harmer, which means that the metal can be hammered into thin sheets without fracture. In other words, the material can withstand permanent deformation under compressive load without fracture. [(1)(2)]

Ductility indicates the workability of the material (the burnishability of casted metal margin). It is related to the force needed to make permanent deformation during burnishing which is called the burnishing index. [(2)] The burnishing index determines the simplicity of burnishing the alloy and is the division of the %elongation to the yield strength. The most malleable and ductile pure metal used in dentistry is gold and silver, while the most ductile alloy is stainless steel. [(1) (2)]

How to measure the ductility:

There are three approaches used for measuring ductility which are: (1) the percent elongation after fracture, (2) the reduction in the cross-sectional area after the tensile test, and (3) the maximum number of bends (a cold bend test). [(2)(3)]

1. **The percent of elongation (% EL)** is the simplest quantitative method used to measure the ductility of the material. It represents the maximum amount of permanent deformation. It is calculated by measuring the change in length of a wire or rod after fracturing under tension to its original length before fracture, which can be calculated as

Percent Elongation (% EL) = (change in length / original length) X100

$$\%EL = (L_F - L_O)/ L_O X100$$

Where:

L_F is the final length, while L_O is the original length.

This is done by placing two marks on the wire or the rod and is related to the original gauge length, the common gauge length for dental materials equal to 51 mm, by pulling the wire or the rod by tensile load, the change in gauge length is remeasured as final length. [(2)(3)]

A material with % EL of 20% at the time of fracture that has increased in length by one-fifth of its original length is considered a material with a high value for plastic or permanent elongation. E.g., most dental gold alloys. On the other hand, a material with only 1% elongation has a limited amount of permanent elongation and is considered brittle like nickel-chromium alloys.

2. **The Percent reduction (%RA)** is calculated by measuring the change in a cross-sectional area divided by the original area. The (reduction) change in cross-sectional area is known by necking or cone-shaped constriction that occurs at the fractured end of a ductile metal wire that ruptures under a tensile load to its original area. It is calculated by the following equation:

$$\%RA = (A_F - A_O)/A_O X100$$

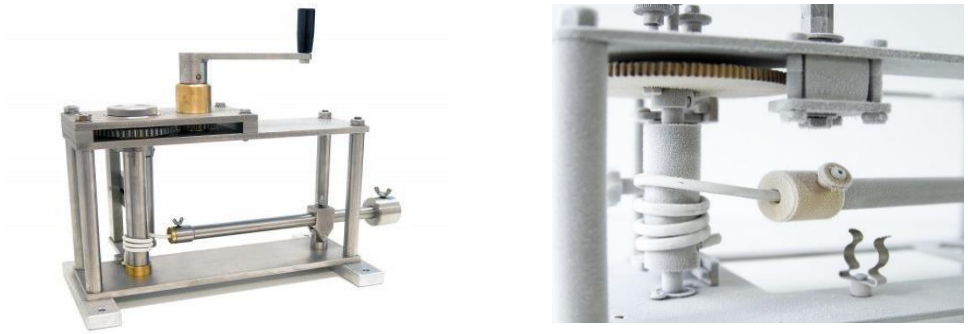
Where:

A_F is the final area, while A_O is the original area. [3]

3. **The cold bend test** is calculated by counting the number of bends of material by tightening the material in a vise and bending around a mandrel with a predetermined radius. The number of bends to fracture is recorded. The more the bends numbers, the larger the ductility. The first bend is formed at 90 degrees from vertical to horizontal, but all succeeding bends are formed at 180 degrees. [3] **(Fig. 1)**

Fig. 1: Cold Bend Machine

The cold bend test is considered the simplest qualitative test not only for the ductility but also used to measure the



soundness of material, "used as quality control test" because the outside of the bend is extensively plastically deformed so that any defects in, or embrittlement of the material will be revealed by the premature failure.

Importance of ductility and malleability in dentistry:

- Fabrication of endodontic files and reamers.
- Adjustment of partial denture clasps.
- Adaptation of orthodontic wires.
- burnishing of crowns and inlays

Brittleness

Brittleness can be defined as a relative inability of a material to deform plastically before its fracture. Brittle material fractured at or near its proportional limit.

Examples of brittle dental materials: amalgam, resin composite, ceramics, cement, and some base metal alloys.

Brittle materials can withstand compressive strength more than tensile strength because of their inability to reduce tensile stress at flow tips. This fact should be taken into consideration during cavity preparation to reduce the tensile stresses subjected to brittle restorative material. (1)(2)

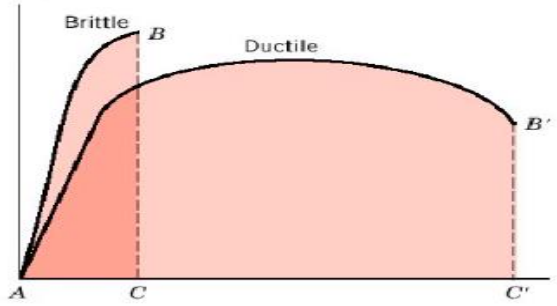
The temperature may affect the brittleness of dental material inside the oral cavity as amalgam, resin composite, and ceramics are brittle in temperatures ranging from 5 to 55 ℃. (2)

If the material is brittle this does not mean that it is weak, some examples show brittleness with high strength and low percent elongation, for example, cobalt-chromium partial denture alloy is a brittle material with 1.5% elongation and high ultimate strength of nearly 870 MPa. Additionally, glass infiltrated alumina core ceramic has 0% elongation and high strength equal to 450 MPa. (2)

Fig. 2: showing the difference between ductile and brittle materials on the stress-stain curve.

The ultimate strength of brittle material

The ultimate tensile strength of brittle material can be measured indirectly by the Diametral compression test or the Brazilian method or indirect tensile. This test depends on applying two compressive forces along the long axis of a cylindrical-shaped specimen. Tensile stresses are developed on the plane perpendicular to the applied compressive



forces that are directly proportional to them. [1] Accordingly, the tensile stresses are calculated from the following equation:

$$\text{Tensile stress} = {}^{2P}/_{\pi DT}$$

Where,

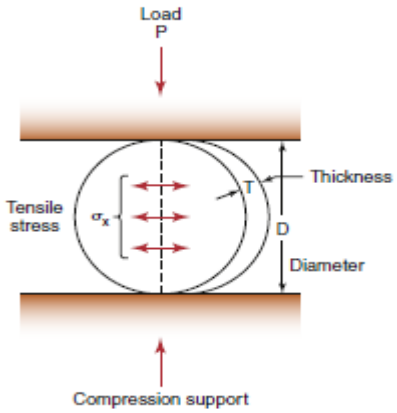
P: The compressive load. **D:** Diameter of the specimen.

π **:** Constant value. **T:** Thickness of specimen.

Fig .3: Diametral compression test for measuring the ultimate tensile strength of brittle materials.

Fundamental of fracture

Fracture is dividing or separating the body into two or more fragments after subjecting it to stress. Fracture is



divided into two main categories which are brittle and ductile fractures or maybe a mix of both. This depends on several factors such as the type of materials, the nature of applied force, temperature, and strain rates. Some ductile metals may possess brittle fractures. [3][4]

The main difference is that ductile metals exhibit a substantial amount of plastic deformation with high energy absorption before fracture while the brittle material has limited or no plastic deformation with low energy absorption before fracture.

Brittle fracture is a serious complication that can occur suddenly without warning and should be avoided while ductile fracture occurs with warning as more strain energy is needed to be induced. It is called a **shear fracture**. [3]

Brittle material fractures by cracks and crack propagation while **Ductile fracture** results in decreasing in the area at the site of fracture known by necking.

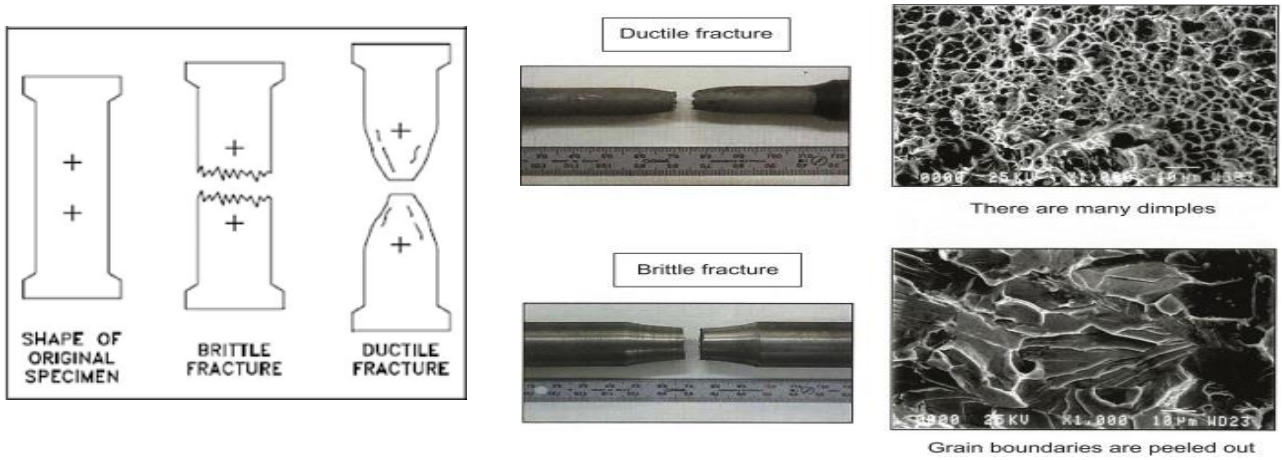


Fig. 4: Difference between brittle and ductile fracture

Ductile fracture

It is characterized by a certain feature on both microscopic and macroscopic levels, fracture takes place in several steps starting with necking when the induced stress is equal to the (ultimate) tensile strength. The ductile fracture appears fibrous and dull. [3]

Complex stress arises in the neck region resulting in the highest stress values at the center of the specimen. Cavities nucleate forming microvoids at the center of the neck part (**Fig. 5-a**). Plastic flow occurs around these inclusions leading to an increase in the cavities' size that will coalesce to form a crack at the center of the specimen (**Fig. 5-b**). The crack grows in a parallel direction to its major axis and perpendicular to the applied stress. These cracks reach the surface of the specimen resulting in its fracture. This is known as cone and cup shape fracture. [4] (**Fig. 5-c)**

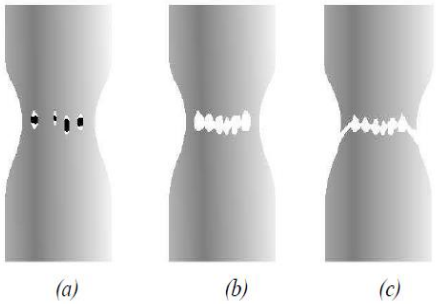


Fig. 5 shows the stages of the cup and cone shape in ductile fracture (a) small microvoids formation (b) coalesce of cavities to form crack (c) crack propagation

Fractographic Study (Microscopic examination)

A scanning electron microscope is used to study the microscopic features of fractured surfaces. It gives better resolution, higher magnification, and depth of field compared to an optical microscope. It gives more information that helps in analyzing the fracture mode, site of crack formation, and stress state.

Ductile fractured surface: The Fracture surface has elongated 'dimples' as if they had formed from numerous holes separated by thin walls. As the cavities approach the edge of the specimen, the crack changes direction and spreads along localized shear planes at an angle of 45 degrees to the tensile axis. As this is the direction of maximum shear stress under necking conditions, each dimple is one-half of a micro-void and may be elongated or C-shaped as seen in **Fig. 6 a and b**. [3]

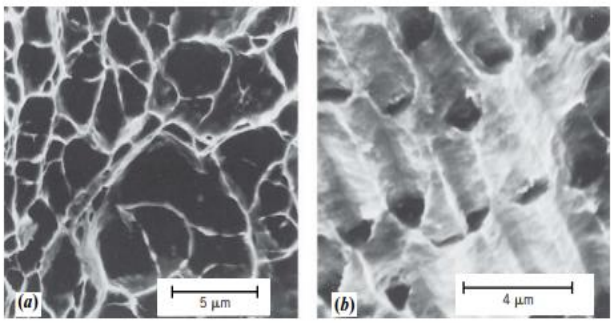


Fig. 6 Scanning electron micrograph of fractured surfaces. (a) spherical dimples characteristic of ductile fracture resulting from uniaxial tensile load 3300X, (b) parabolic shaped dimples characteristic of ductile fracture resulting from shear loading 5000X. [3]

Brittle fractured surface: an amorphous brittle material characterized by a shiny and smooth appearance in the site of fracture as in ceramics. Fracture of brittle material has different patterns; as in some steel pieces, a series of V-shaped takes place on the surface. It looks like river lines and is known by the chevron mark (**Fig. 7&8).** These river lines indicate the initiation point of fracture accordingly, crack propagation occurs in the opposite direction which helps identify the crack propagation direction and trace it when material fractures in a brittle manner, other brittle fracture surfaces display lines or ridges from the source of crack, forming a fan-like pattern that can be detected by necked eye as it has a noticeable coarse appearance. [3][4][5]

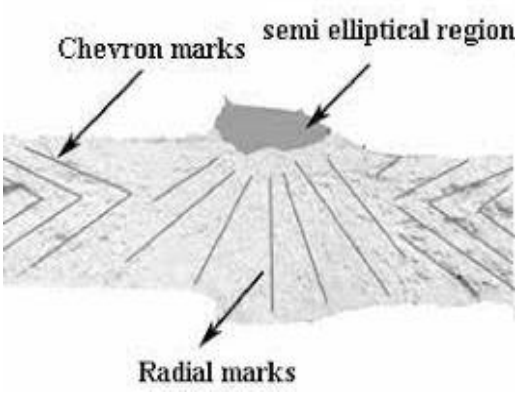


Fig. 7: The different patterns of brittle fracture

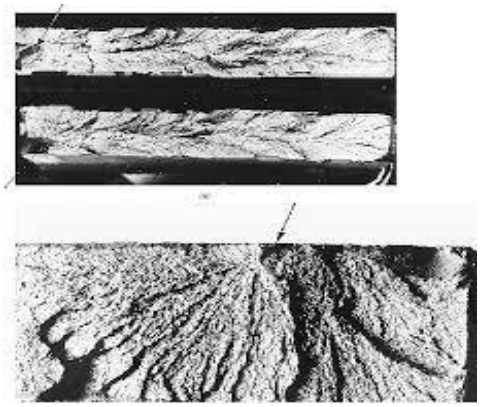


Fig. 8: The V-shaped 'chevron' marks and radial fan-shaped ridges are characteristic of brittle fracture. [3]

Most crystalline materials exhibit fracture separation along crystallographic planes which is known as cleavage fracture, in brittle fracture, the crack propagation is rapid and has a trans-granular shape usually although it can be inter-granular under embrittling conditions or weakening of the grain boundaries region.

The difference between trans-granular and inter-granular fractures is that trans-granular fracture passes through the grain forming a cleavage pattern, while inter-granular fracture takes place along the grain boundaries [3] **(Figs. 9&10**).

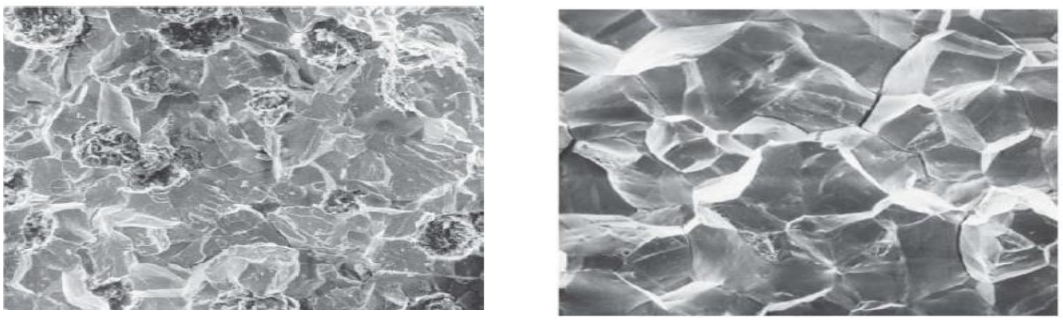


Fig. 9: Trans-granular and inter-granular fracture of crystalline brittle materials. [3]

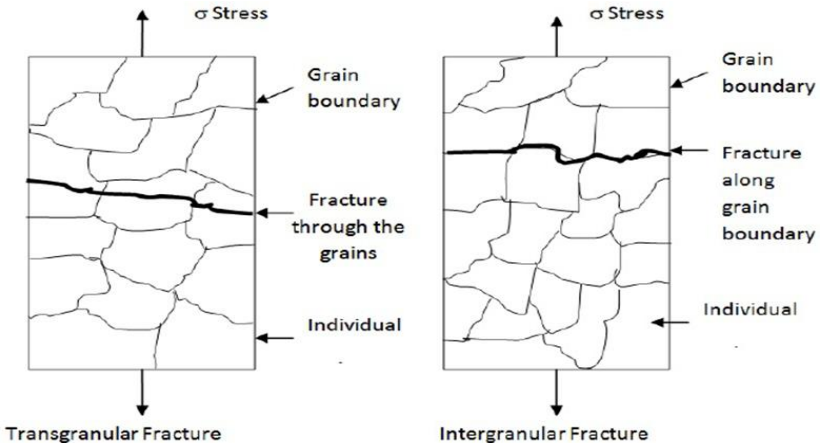


Fig. 10: Schematic cross-section profile showing crack propagation in the trans-granular and inter-granular fracture types

How to guard against brittle fracture

Several approaches can be considered to avoid brittle fracture occurrence. They can be summarized as follows: cavity design to withstand compressive stress more than tensile stress, elimination of sharp edges and notches in the cavity design and proper finishing to the restoration, avoid impact loading on brittle material.

Ductile To Brittle Transition

It has been reported that steel changes from ductile to brittle fracture when the temperature is around or below 4℃, once a crack originated at stress concentration it propagates. [3]

Pure metals have a definite transition temperature below which the material behaves ductile while above it behaves brittle. Most HCP metals and FCC as copper and aluminum tend to have no ductile to brittle transition. **Fig.11**

The transition temperature of low-strength steel (BCC) depends on the alloy composition and microstructure, the grain size refinement will lead to a decrease in the transition temperature and increase the strength as well as toughness. Both ceramics and polymers show ductile to brittle transition, ceramics develop transition at a high temperature above 1000℃. While polymer show transition at a narrow range of temperature below room temperature.[3] The Charpy impact test is used to determine the ductile to brittle transition by testing the impact strength. [3] **Fig 12**

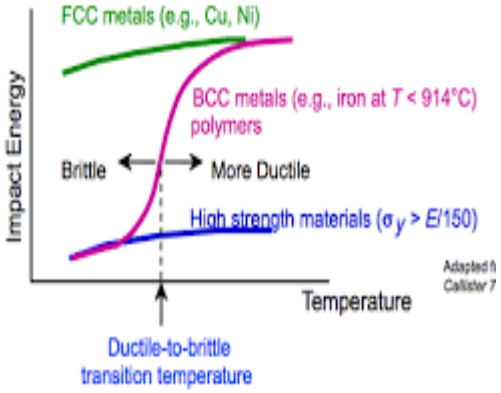


Fig.11 Schematic curves for the three general types of impact energy–versus–temperature behavior. [3]

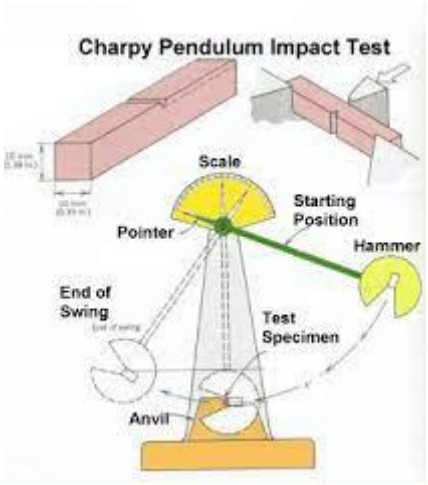


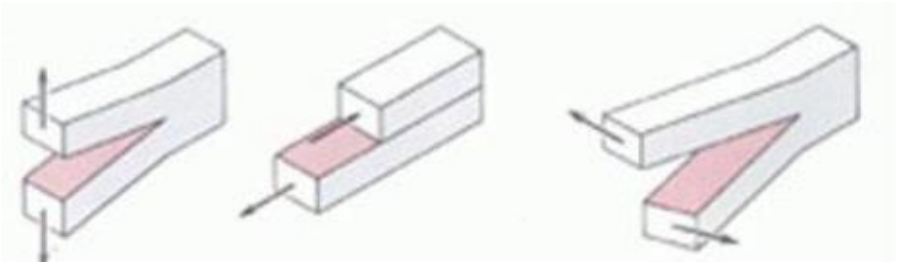
Fig.12 Charpy impact test

Fracture mechanism

The steps of fracture start with the crack formation and then its propagation in response to the induced stress. Cracks and flaws are present naturally in any material. It is a type of volume defect that occurs and nucleates after a time in service. This leads to the fact that the actual fracture strength is lower than the theoretical one predicted from the atomic bond energies. There are two types of flaws one present on the surface and the other located in the interior body of material. Cracks act as stress concentration factors accordingly, less force is needed to fracture the material. [3]

Modes of crack surface displacement

There are three modes of crack displacement that result from force leading to either opening, sliding, or tearing [3][6] (**Fig 13)**. The crack may be concentrated at the tip. This depends on crack orientation and geometry. In this case, the magnitude of the localized stress decreases as we go far away from the tip of the crack.



a b c

Fig.13: Modes of crack displacement (a) Mode I (opening or tensile), (b) mode II (sliding), and (c) Mode III (tearing)

Stress Raisers are flaws that can expand the area of stress. These stress raisers arise from surface cracks, defects, and surface roughness or from the internal as inclusion, pores that cannot be controlled or eliminated. Surface cracks are not preferred and should be avoided in dental procedures by proper finishing and polishing and glazing of dental porcelain. [3][7]

The maximum stress at the crack tip under tensile loading can be calculated from the following equation result from the crack propagation of the Griffith crack model:

$$6_m = 2 6_o \left(\frac{a}{\rho t}\right)^{1/2}$$

Where

6_o is the magnitude of the nominal induced tensile stress

ρt is the radius of curvature of the crack tip (**Fig.14**)

a represents the length of a surface crack or half of the length of an internal crack

for a long microcrack that has a small tip radius of curvature; we use this equation. [3]

$$k_t = \frac{\quad}{\quad} = 2\left(\frac{a}{\rho t}\right)^{1/2}$$

$$k_t = \frac{\quad}{\quad} = 2\left(\frac{a}{\rho t}\right)^{1/2}$$

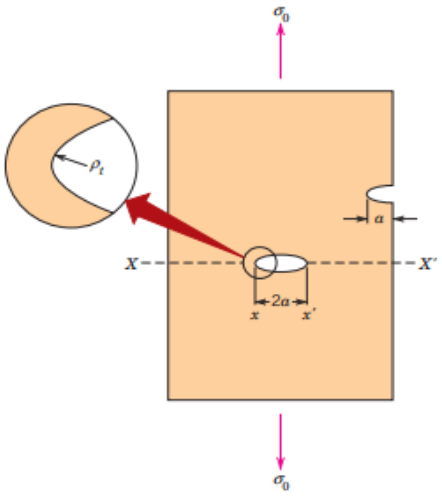


Fig. 14: The geometry of the surface and internal cracks. (3)

Fracture Toughness

Fracture toughness is a material property that can be defined as the ability of the material to be plastically deformed without fracture. It is the amount of energy needed to fracture the sample which contains a crack. It is more obvious in brittle material as the ductile material can be plastically deformed and redistribute the stresses. Therefore, brittle material has a lower fracture toughness value than ductile materials. (1, 3)

Fracture toughness (K_C) can be calculated from the following equation

$$K_{IC} = Y6\sqrt{\pi a}$$

Where

k_{IC} represents fracture toughness in mode I (shown in **Fig 13**)

Y dimensionless geometry factor

6 is the stress

a is the crack length (3)

Unit = stress times the square root of crack length

$MPa \bullet m^{1/2}$ or $MN \bullet m^{-3/2}$. (2)

Importance in dentistry

Fracture toughness describes the material resistance to crack propagation especially brittle materials when subjected to tensile loads that could lead to fracture. This can be prevented by several methods as designing a basis against these types of failure, modifying the material by adding fillers as in the case of resin composite to deflect cracks,

presence of crystalline phase in ceramics can deflect/ obliterates cracks, and the presence of tough zirconia particle that acts as a crack healer. [1]

Method of testing fracture toughness

There are several testing methods for measuring fracture toughness. Among these methods:

1. Single edge notched beam:
   a. Single edge pre-cracked beam
   b. Single-edge V-notched beam
2. Chevron notch method
3. Compact tension method.
4. Short rod method (tension test).
5. Double torsion method
6. Indentation method:
   a. Vickers's hardness or Koop's indentation.
   b. Nano-indenter

The most commonly used method for measuring the fracture toughness of metal, ceramics, and resin composite materials is the one presented in the standardized ASTM method. It is the single-edge-v-notched beam (SEVNB) test:

Single-edge-V-notched beam (SEVNB) test:

Rectangular pre-notched specimens prepared in a mold using the single-edge notched design according to ASTM Standards E399-90(1992) as shown in **Fig.15**.

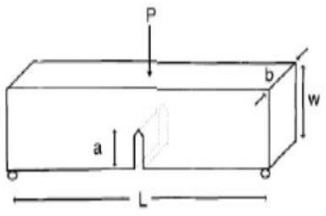


Fig.15 Specimen geometry for the determination of fracture toughness by single-edge V-notched method.

Testing Methods:

The specimens can be tested under 3-point or 4-point bending using the universal testing machine. In the case of the 3-point bending test, each specimen is supported on two parallel stainless-steel rods (10 mm in diameter) located at a certain distance from each other. The load was applied through a cylindrical stainless-steel rod at the middle as seen in **Fig. 16**. The load is applied with a certain crosshead speed (mm/min) until fracture. Both the load and the deflection are obtained from the load-deflection curve produced by the software program of the computer connected to the testing machine. Care should be taken on carrying the specimen that may be subjected to unexpected fracture before testing. [8] [9]

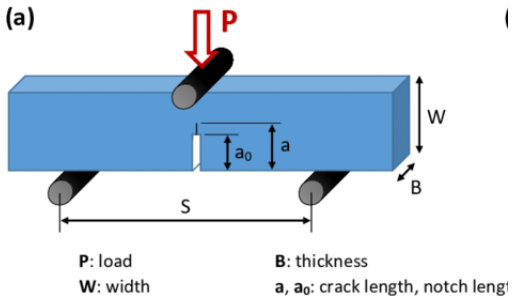


Fig.16 V-shaped pre-notched specimen subjected to 3-point bending.

Calculation of the fracture toughness (FT):

The fracture toughness of each specimen was calculated from the following equation.

$$K_{Ic} = [3\ P\ L\ a^{1/2} / 2\ b\ w^2] \times f\ (a/w)$$

Where:

P= Load at fracture in Newton (N)

L = Distance between the support in mm

a = Crack length in mm = W/2

b = Thickness of specimen in mm

w = Width of specimen in mm

f(a/w) = The value f (a/w) was obtained from ASTM slandered.

Ball-on three-balls method (B3B):

There is another method of testing the fracture toughness of ceramic materials known as "ball-on three-balls". It depends on placing a circular disk or a rectangular plate, which is supported by three balls in contact and loaded on the opposite side by a fourth ball of the same size. The loading ball is positioned centrally to the three supporting balls. **(Fig.17)** Then by Using a Knoop indentation almost semicircular surface crack is introduced as a starter crack in the center of the specimen, opposite to the loading ball. (**Fig.18**)[9]

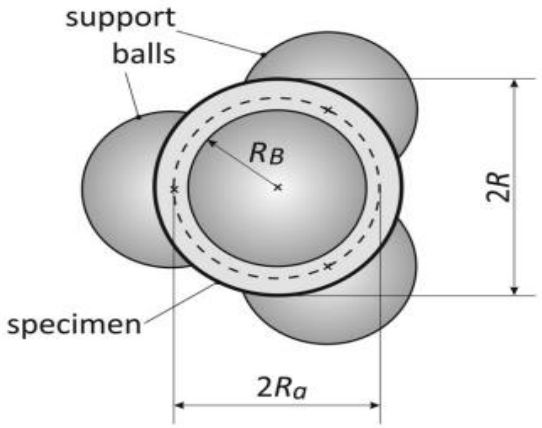


Fig.17 diagram showing Ball-on-three-balls test geometry with four equally sized balls of radius R_B. Specimen and loading ball is positioned centrally to the three supporting balls in contact. [9]

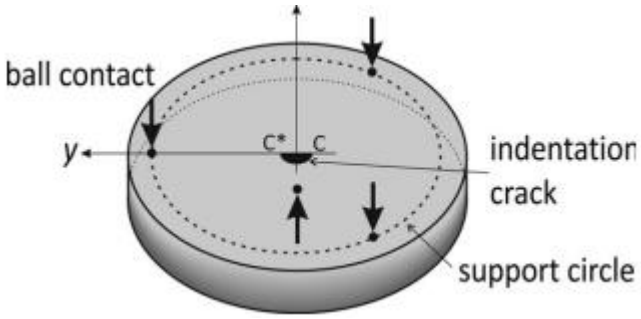


Fig.18 Diagram of crack position in B3B-KIc test [9]

Calculation of fracture toughness from the B3B method

$$K_{IC} = 6_{B3B}\ Y\sqrt{\pi a}$$

6_{B3B} the maximum tensile stress occurs in the center of the specimen opposite to the loading ball

a is the crack depth

Y is the load-independent geometric function that depends on the specimen and crack geometry as well as on Poisson's ratio of the tested material.

Several errors may occur during this test related to the crack size and shape, the position of the crack, and the inaccurate knowledge of Poisson's ratio of the material. [9]

Conclusions

- Cracks have a critical effect on materials' failure. They should be avoided especially those present on the surface of the dental material. There are two types of dental material; ductile (metal and alloys) and brittle materials like composite, ceramic, and amalgam.
- Ductile material shows a higher percent of elongation, and fracture toughness, than brittle material. Ductile material fractures away from the proportional limit while brittle material fractures at or near the proportional limit.
- Ductility can be measured with three methods which are: percent of elongation, percent of reduction, and cold bend test.
- Ductile fracture is characterized by necking while brittle fracture is characterized by crack propagation.
- Ductile fracture is more preferred than brittle ones because ductile fracture takes time and can be avoided while brittle fracture occurs without any warning and is characterized by rapid crack propagation.
- Fracture toughness is the material's ability to resist deformation in the presence of cracks.

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Assessment of chemical degradation in dentistry :a review

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Abstract:. Analysis of the reasons for structural degradation and failure of the employed materials is crucial in order to forecast how well the clinical performance of the dental materials will perform in the patient's mouth. The oral cavity is a challenging environment with pH and temperature variations, various stressors, and bacteria. The most frequent causes of dental material failure are poor material selection, poor design, or overuse. Damage might also happen while being repaired. It's crucial to prepare for failure, identify its causes, and take the appropriate precautions to avoid material failure**.**

Keywords: *Chemical degradation, dentistry, degrdation and aging.*

Introduction

It's important to predict the clinical performance of the dental materials used in the patient's mouth; therefore, it's essential to analyse the causes of structural degradation and failure of the materials used. The oral cavity is a harsh environment with fluctuations in pH and temperature, different stresses, and bacteria. Improper material selection, inadequate design, or misuse are the most common reasons for the failure of dental materials. Also, damage can occur during service. It's important to plan to avoid failure, assess the causes, and take the necessary preventive measures to prevent material failure.[1]

Dental material failure assessment can be classified into

1) Chemical degradation.
2) Biological degradation.
3) Mechanical degradation.
4) Optical degradation.
5) Thermal degradation.

1) Assessment of chemical degradation

A. Metal degradation

Tarnish is a staining of the surface caused by the production of hard and soft deposits. It doesn't cause material deterioration, but it is unpleasant and may be simply removed by polishing the metal. While, corrosion is a chemical interaction between the material (usually metals) and its surroundings, causing material degradation, so it's a far more significant problem. The corrosion process for metals in the oral environment is usually electrochemical, with the removal of electrons in an oxidation (anodic) reaction at the anode, converting the metal to a positively charged ion:

$M \rightarrow M^{n+} + ne^-$

Depending on the environment, a variety of reduction (cathodic) reactions at the cathode can occur. Metal ions take the electrons to produce metal atoms:

$M^{n+} + ne^- \rightarrow M$

When the environment is hostile; has strong temperature and pH fluctuations, all metals are susceptible to corrosive attack, which is a problem because it weakens materials, can cause fractures, and may also react negatively with the biological environment.[1]

Methods of testing metal degradation

Identification and quantification of degradation products from metals and alloys", there are 2 methods. Test solutions (electrolytes) to be used are either saline, artificial saliva, or artificial plasma. It's selected according to the environment to be surrounding the material in vivo. The sample shape can be circular or bar with a rectangular cross-section, but it is usually preferred to resemble the final shape to be used in vivo. The surface condition of the sample also should match that to be used in vivo.

1. Electrochemical test:

It is used to know the overall electrochemical behavior of the metal or alloy.

Procedure:

• Clean the specimen ultrasonically for 10 to 15 minutes in ethanol, then rinse with water and immediately transfer it to the test cell.

• Only the test surface should be in touch with the electrolyte, thus place it in a waterproof electrode holder. The sample is known as the working electrode. The counter electrode is either platinum in the shape of a grid, plate, or wire, or crystalline carbon of area 10 times or more than that of the sample.

Note: This difference in area is to ensure that there is continuous uptake of the ions from the solution at the cathode, so that ion saturation of the electrolyte does not occur, which leads to the termination of the corrosion reaction process.

• Avoid creating conditions (e.g. scratches) that could lead to crevice corrosion due to the formation of a crevice between the holder and the sample.

• Set up the test apparatus, and then fill the test container with the electrolyte. Temperature of the water bath should be maintained at 37°C.

• Oxygen level within the electrolyte is decreased before the beginning of the test by pumping nitrogen or argon.

• During the test period, continuous stirring of the electrolyte is done either by gas agitation or magnetic stirrers, to avoid concentration gradient.

• The breakdown potential (Ep) of the sample is determined by cyclic polarization. It's the electrode potential above which localized corrosion occurs.[2] (Figure 1)

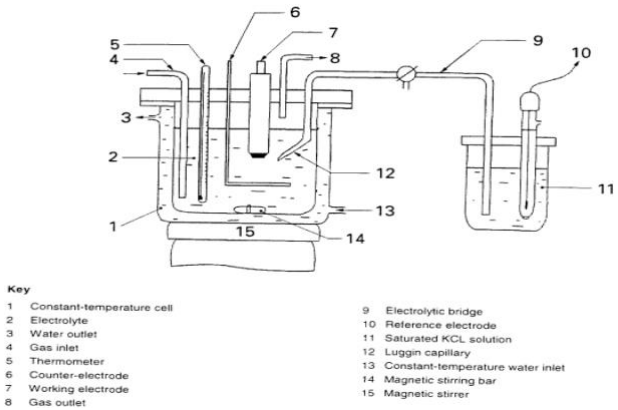

Fig. 1 A diagram of electrolytic test cell

2. <u>Immersion test:</u>

It is utilized to degrade the test material chemically in order to generate degradation products that can be analyzed.

Procedure:

• Calculated volume of the electrolyte of choice is poured in a glass container, which is placed in a water bath.

• Immerse the sample in the electrolyte, make sure it does not touch the walls of the glass container or touch each other if more than one sample is placed in the same container.

• The size of the glass container, as well as the number of samples to be used in one container, is determined so that less than 1 ml of the electrolyte is covering 1cm2 of the sample surface.

- Measure the initial pH value of the electrolyte at the start of the test.
- Close the container tightly, and keep at 37°C for 7 days.
- At the end of the test, remove the sample, then measure the pH value of the electrolyte again.
- Examine the surface of the samples under microscope of either low or high magnification, depending on the extent of details needed.
- The electrolyte undergoes qualitative and quantitative analysis using mass spectroscopy, atomic absorption spectroscopy, etc. Note: Both sample surface analysis and electrolyte analysis must be done together, as some corrosion products deposit on the sample surface and don't dissolve.[2]

B. Polymer degradation

Many polymers used in dentistry are sensitive to solvent absorption (mainly water), pushing the polymer chains apart and inducing swelling and slippage of chains, loss of soluble components, as well as affecting the matrix-fillers interface. Water acts as a plasticizer where it neutralizes the secondary bonds between the chains that prevent the slippage of the chains against each other. The end result is that the polymer gets softer, the glass transition temperature lowers, and the strength may be reduced. In dental materials based on polymers, unbound monomers and degradation products infiltrate into the pulp and other adjacent tissues, ultimately entering the circulation, giving possible biological risks as: being potential carcinogens and inducing immunological responses. Mastication forces result in increased material sensitivity to abrasive wear due to the softening of the polymer.[3]

Methods of testing polymer degradation

Identification and quantification of degradation products from polymeric medical devices", there are 2 tests.

The procedure is common; the difference lies in the temperature used and period of testing.

1. **Accelerated degradation test:**

- Temperature: higher than 37 °C but below the polymer's melting or softening range. Usually, 70±2 °C is employed.
- Test periods: 2 to 60 days for devices of more than 30 days usage. 2 to 7 days for devices of less than 30 days usage. Time can last until the material loses integrity if it is made of resorbable polymers. N.B.: the term Usage refers to the intended time the device should be serving in the intended environment (e.g the oral cavity). [3]

2. **Real time degradation test in simulated environment:**

- Temperature: 37±1 °C

• Test periods: 1,3,6 and 12 months for devices of more than 30 days usage. 4 time intervals within 30 days for devices of less than 30 days usage. Time can last until the material loses integrity if it is made of resorbable polymers.

These tests are to be done in order, where the accelerated test is done first, and if the analysis of the degradation products doesn't help in their risk assessment, real time test is to be done subsequently. Procedure:

• Sample is prepared. It should be taken in consideration to minimize or eliminate the parts that will not be in contact with the environment in in vivo conditions.

• The initial mass of the sample is recorded.

• The test solution to be used should be nearly the same as the in vivo environment of interest (e.g. artificial saliva).

• The sample is immersed in the test solution.

• Time and temperature of test depend on the selected test (accelerated or real-time). The pH value is selected to simulate the in vivo environment.

• The sample is then removed from the solution, rinsed, dried, and its final mass is measured.

• Since the rinse water could contain debris loosened from the sample, it should be added to the test solution for analysis of residual monomers and leached elements.

• High-performance liquid chromatography and mass spectroscopy are used for identifying and quantifying residual monomers and leached elements.[3]

C. Ceramic degradation

Ceramics are more resistant to electrochemical corrosion than metals, yet they are still subject to chemical corrosion, which can have a significant impact on ceramic strength. Ceramic failure is usually caused by crack propagation. At the crack tip, chemical interactions between the ceramic and the environment can have a significant impact on the pace of crack propagation. E.g.: Water or water vapor at the crack tip can create hydroxides by reacting with the Si–O–Si bond at the crack tip in a silica-based glass.

When environmental factors are paired with high levels of stress in the ceramic, the crack's rate of propagation is significantly increased. In such cases, the failure can be attributed to stress corrosion cracking. Ceramics are prone to chemical corrosion in the range of oral fluid pH. The dissolving potentials of ceramics in acidic beverages such as soda (pH 2.5-4) vary depending on the patient's saliva and diet buffering ability. Glass-phase ceramics can also be dissolved by basic items like antacids (pH 10-14).[4]

Methods of testing ceramic degradation

Identification and quantification of degradation products from ceramics", there are 2 methods.

1. **Extreme solution test:**

Most ceramics can be screened for probable breakdown products using an extreme solution test performed at a low pH.

Procedure:

• Sample to be tested is grinded to a suitable particle size (i.e. sample is in powder form), where it should be between 400 and 315 μm.

• Depending on the solubility of the particles, the initial mass is determined. For high solubility, 10 g should be used, while for low solubility 5 g are enough.

• A plastic container (e.g. polypropylene) is used for the test. Glass containers are to be avoided because they may contaminate the test solution.

• Test solution is buffered citric acid of pH=3.

• The container is placed in water bath of 37˚C for 120 hours. If the specimen totally dissolves before 120 hours, time should be recorded.

• Continuous shaking of the container is done mechanically.

• At the end of test period, the container is left to cool to room temperature.

• A filter paper of known mass is used to filtrate the remaining undissolved particles from the solution.

• The filtered solution is analyzed using inductively coupled plasma spectroscopy, atomic absorption spectroscopy or mass spectroscopy.

Rinse the filter paper having the remaining particles with water to remove the citric acid, dry, and then weigh them.

• The difference between the mass of the filter paper before and after filtering is the mass of the remaining specimen.

• Subtracting this mass from the initial mass of the specimen gives value of the mass of the dissolved part. [4]

2. **Simulation solution test:**

It uses a pH that is more commonly observed in vivo. It follows the same procedure as the extreme solution test, with slight differences:

• The test solution is Tris (hydroxymethyl) aminomethane hydrochloride (TRIS-HCI) buffer of pH=7.4, this resembles the normal pH level within the body.

• The sample could be put as a coating on a disc of dimensions 36x2 mm or could be grinded as in the extreme solution test.[4] (Figure 2)

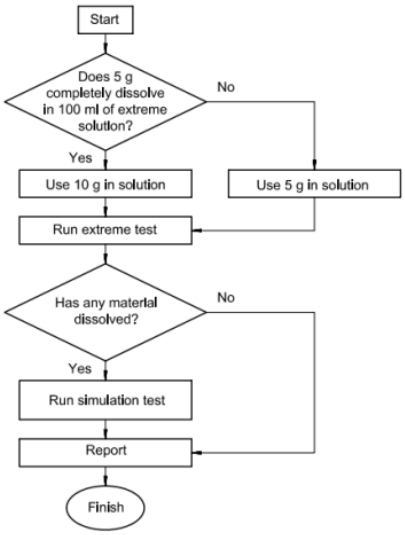

Fig. 2 Flowchart for testing sequence

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Assessment of biological and mechanical degradation in dentistry :a review

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Abstract:. To predict how well the clinical performance of the dental materials will perform in the patient's mouth, it is essential to analyse the causes of structural degradation and failure of the used materials. The pH and temperature changes, a variety of stresses, and microbes make the oral cavity a difficult habitat to live in. bad material selection, bad design, or overuse are the three most common reasons for dental material failure. Additionally, damage can occur while being corrected. To prevent material failure, it's imperative to plan for failure, understand its causes, and take the necessary precautions.

Keywords: *Biological degradation, dentistry, mechanical degrdation.*

Introduction

Analysis of the reasons for structural degradation and failure of the employed materials is crucial in order to forecast how well the clinical performance of the dental materials will perform in the patient's mouth. The oral cavity is a hostile environment that includes germs, changes in pH and temperature, and many stresses. The most frequent causes of dental material failure are poor material selection, poor design, or overuse. Damage might also happen while being repaired. To prevent material failure, it's crucial to plan to avoid failure, evaluate the causes, and implement the necessary preventive actions.[1-4]

1) Assessment of biological degradation

Restorative materials of any type may not attach to enamel or dentin with enough strength to withstand the stresses of contraction during polymerization, wear, or thermal cycling. This is known as microleakage. Along the unsupported border, the gap encourages material breakdown. The gap width widens as a result of this breakdown, allowing larger particles and molecules to enter the pulp chamber. Capillary motion may

suck bacteria, food debris, or saliva into the gap between the restoration and the tooth if a bond does not form or if debonding develops, causing pulp tissue infection. It also causes minor stains and poor aesthetics, which may necessitate early replacement.[5]

Methods of Microleakage testing

Microleakage testing has been employed to determine a restorative material's clinical performance. Over the years, many microleakage testing materials have been created and tested. There hasn't been any resolution on which testing method would produce the most accurate results. Simulating oral circumstances and providing a better quantitative picture of microleakage have been attempted.

1. **Direct observation:**

Direct inspection of restorations is the simplest way to assess microleakage, using no agent or tracer. Clinical observation is commonly performed to recognize macroscopic changes in a restoration's marginal integrity.

This can be done:

1) Tactilely with an explorer.

2) Visually by looking for discoloration in the surrounding enamel or a gap between the tooth and the restoration.

Clinical examination is frequently combined with photographic observation. To determine changes in marginal integrity, a macro photographic black and white record of each restoration is made at various time intervals.[5]

2. **Fluorescent dyes method:**

Because the fluorescent dye is non-toxic, it can be used for in vivo experiments both topically and systemically.

This dye:

1. Could be detected at low concentrations.

2. Sensitive to ultraviolet light.

3. Easy to photograph.

4. Allow for more consistent results.

5. Inexpensive.

Under UV light, the difference between the natural fluorescence of the tooth and the dye produced a contrast that made it easy to detect the course of dye penetration. As a result, new fluorescent dyes are now being used to tag

restorative products like cavity varnish and glass ionomer cements. The dye is quenched (i.e. the fluorescence disappears) by the zinc oxide eugenol cement, so it cannot be employed with it. The absence of the effect of pulpal hydrostatic pressure on the dye has been a source of criticism for laboratory testing (in vitro). In vivo testing yielded much lower mean microleakage scores than in vitro testing among human subjects. On the other hand, when in vivo teeth to be tested have undergone endodontic therapy, the outcomes were more similar to those of in vitro testing.[5]

3. **Radioisotope method:**

The use of autoradiography, specifically 45Ca (calcium 45), to identify microleakage has gained widespread acceptance. The radioisotope is injected into the specimens' edges. For proper contact between the specimen and the radiographic film emulsion, a flat surface is required. After that, the film is developed, and the radiolucency around the restoration is used to determine microleakage.

With the autoradiograph, even minute amounts can be identified, because the radioisotope can penetrate deeper than the dyes employed. The dye has a molecular size of 120 nm while the radioisotope has a molecular size of 43.2 nm. The radioisotope has two-hour exposure duration, unlike dyes which require 24h or more.

Note: Autoradiography is a technique that uses X-ray film to visualize radioactively labeled molecules or fragments of molecules.[5]

4. **Dye penetration method:**

The most popular procedure is to stain microleakage and nanoleakage with colored chemicals. The dye penetration method includes staining the sites of microleakage with contrasting dyes by immersion in a dye solution, and then examining the tooth–restoration interface for signs of staining. 0.5 % basic fuchsin, 2 % methylene blue (both known as organic dyes), and 50 % silver nitrate are the most frequently used solutions. An image analysis equipment connected to a stereomicroscope was used to analyze dye penetration. The actual length of dye penetration along the contact was measured using digital image microscopy.[5],[6]

Note: A stereo microscope is an optical microscope that allows you to see a specimen in three dimensions. Separate objective lenses and eyepieces are included among its components, so each eye has two independent optical paths. The use of silver nitrate is second to the use of organic dyes. Silver nitrate was chosen because the significant optical contrast of silver particles makes it easy to detect using microscopy. Silver nitrate staining is the most widely utilized agent for nanoleakage evaluation, because of its incredibly small diameter, which allows it to quickly

penetrate the interface zone. Silver nitrate molecules can become static after penetration, preventing additional penetration during specimen processing. This method requires immersing the specimens in a 50% silver nitrate solution for two hours in the dark. After rinsing the specimens to eliminate any silver ions on the surface, they are immersed in developing solution and subjected to fluorescent light for six hours. At this point, the silver ions absorbed in the specimens precipitate as silver particles. The microleakage specimens are then sectioned. The degree of leakage can then be determined in the same way as organic dyes. Regarding amalgam restorations, the silver staining procedure was tried, but the results were inconsistent. This was thought to have happened as a result of chemical interactions between amalgam components and silver ions. [5],[6]

The dye penetration assay has a number of benefits over other methods:

1) No radiation or reactive chemicals are used.

2) A variety of dye solutions are accessible, making the process exceedingly viable and repeatable.

3) Simplicity of use.

4) Inexpensive cost.

The drawbacks of organic dyes include:

1) Some dyes, such as basic fuchsin, can react with dentin, so researches have failed to clearly identify which dyes are appropriate for use.

2) The particle size of the dye utilized, which can impair the test's reliability.

3) Most dyes require a much longer exposure time than other procedures, such as radioisotope.

4) Unless images of the specimens were taken, there were no permanent records.

Because dye penetration varies from one location to another, assessing a single part of the tooth is not representative. Multiple surface scoring methods are thus preferred over single surface scoring methods because the findings are more indicative of the leakage pattern. To effectively assess leakage, it is advised that three segments be used for each restoration.[5]

5. Air pressure method:

Other tests were created to enable the quantification of results. The contact between restorations and cavity preparation walls was penetrated with air pressure. The presence of leaking was confirmed by the appearance of bubbles at the edges. The amount of air pressure required to demonstrate leakage is quantified. The region of leakage

cannot be identified because these specimens are examined under water, and photographic records are very challenging to collect.[5]

6. **Electrochemical method:**

The electrochemical approach was developed from endodontic research for application in restorative research. The technique allows for reliable identification of the onset of leakage and delivers quantified data over time. The procedure works by inserting an electrode into the root of an extracted tooth, bringing it into touch with the restoration's base. The restored tooth is isolated by nail polish (to prevent electrical leakage via the natural structure) and immersed in an electrolyte bath. Leakage is measured by monitoring the current flowing across a serial resistor when a voltage is introduced between the tooth and the bath.[5]

7. **Bacterial microleakage method:**

The investigation of microleakage using bacteria could be the most clinically useful microleakage test. Chromogenic microorganisms were used in a study with extracted teeth that had Class V amalgam or acrylic resin. They were incubated for seven to sixty days in a broth culture. Shavings of the dentin under the restoration were cultured at the end of the test period. Bacteria penetrated the acrylic resin more than amalgam restorations. To avoid cross-contamination with other germs, the procedure requires a sterile environment, so it's a quite meticulous procedure.[5]

8. **Secondary caries formation method:**

Bacterial culture or chemical systems are used in the secondary caries approach. This approach has the advantage of connecting artificial caries development to microleakage. The acidified gelatin gel approach has been found to develop lesions with histological characteristics that are similar to early caries.

Around the tooth, an acid-resistant varnish was applied, stopping 0.5 mm short of the restoration margin. The specimens were placed in a 20% gel solution, adjusted to pH 4.0 by the addition of 30% lactic acid, for 10 weeks, 24h after restoration placement. To prevent bacterial growth, thymol was added to the gelatin. Polarized light microscopy was used to examine ground slices of the teeth.[5]

2) **Assessment of Mechanical degradation:**

1) **Deformation**

a) **Compression testing**

Since most of the mastication forces are compressive, it's important to investigate material under compression. When an objected to compression, failure may occur due to complex stresses. Compression testing depends on the concept that axial force is applied at a constant strain rate to the cylindrical specimen at each end that causes failure in an opposite direction. The force is applied and resolved to forces of shear along a cone-shaped area at each end, and tensile forces at the central portion. If the test specimens are too short, force distributions become more complicated, and if it is too long, buckling may occur. The cylinder should have twice the diameter for satisfactory results. Compressive strength is useful for comparing brittle materials that can't be tested under tension. Compressive testing is useful in testing amalgam, resin composite, and cement.[7] (Figure 3)

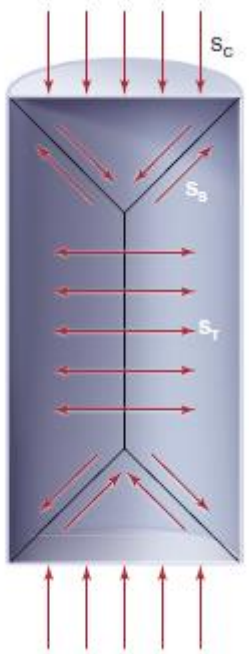


Figure 3: Compressive test

b) Tensile testing

i) Uniaxial tensile testing

Most restorative materials fail by tensile stresses due to the complex loading of their complex geometries. The typical shape of a specimen for tensile testing is like a dumbbell or dog bone with a center region (Gauge length) with a smaller diameter than the ends of the specimen. So this will concentrate the stress in the middle, and ensure failure in the middle. The test is technique sensitive and specimen alignment is critical to make sure that load loading is uniaxial. This test is not used to measure dental composite. It is used to measure the ductility of the material and is used with metallic materials. [8] (Figure 4)

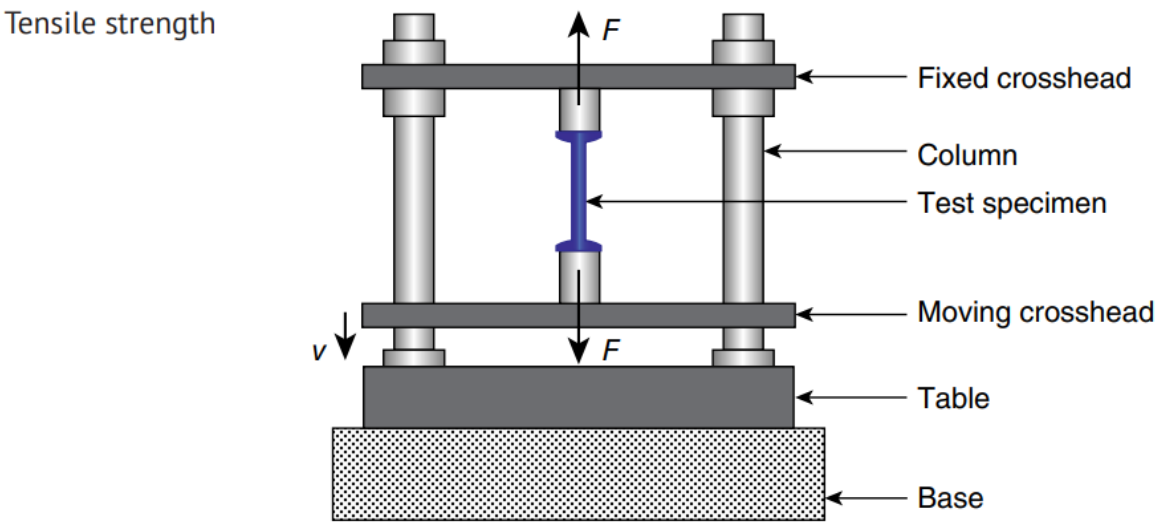


Figure 4: Tensile testing

ii) Diametral tensile testing (Brazilian test)

Due to the difficulty to perform uni-axial tensile tests for brittle materials due to specimen alignment and the difficulty to form dumbbell shape specimens, the diametral test was developed. It is used for dental composites and ceramics.[1]

It involves breaking a disk under compression diametrically till fracture. This compressive stress introduces tensile stresses to the material in the plane of the force application. The specimen production is critical to ensure that the specimen is uniformly loaded. This test is considered accurate when the specimen breaks uniformly. The failed specimen must be two halves, not a distribution of fractured pieces. [7] (Figure 5)

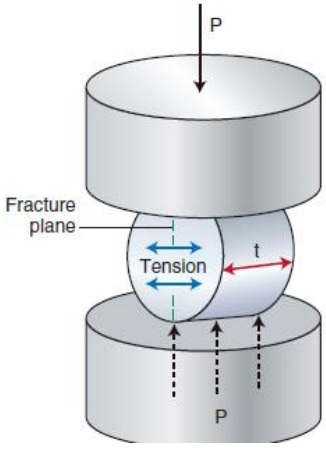


Fig 5: Diametral compressive testing

c) Flexural test

The flexural test is obtained when one loads a simple single beam supported at each end, with load applied in the middle. This test is called the three-point bending test. The maximum stress measured is called flexural strength. Four-point bending is preferred to three points. Four points loading uses two loading elements applying a uniform load to the beam to prevent V-Shaped buckling of the beam. It also prevents stress concentration in the midline when a single loading element is used. Also the three-point flexural test, the failure may not be in the midpoint directly, so a correction must be made. It is used with a long fixed partial denture spans or acrylic partial dentures. For brittle materials such as ceramics, it is preferred than diametral compressive tests because they stimulate stress distribution in dental prostheses and clap arms of removable dental prostheses.[1,7] (Figure 6)

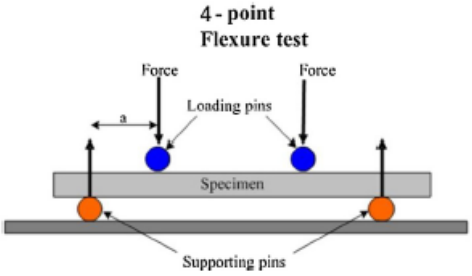


Figure 5 – Four-point flexural strength tests [61].

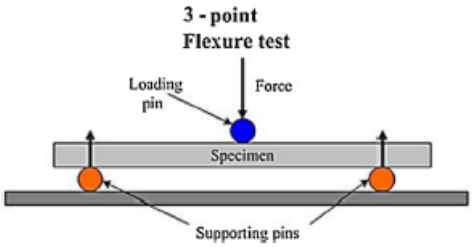


Figure 6: Three point and four point loadings

d) Torsion

Torsion is the variation of pure shear and rotation where the structural member is twisted. Torsion testing involves twisting a sample along an axis. A longitudinal sample is placed in a torsion tester and one end is twisted around the long axis till failure. Most endodontic files and reamers are subjected to twisting. Also, torsion is important on considering implant restorations and orthodontic wires.[7,9]

e) Buckling

This test is used with orthodontic wire or endodontic files. The idea of buckling depends on the application of forces in the axial direction resulting in lateral bending and deformation. A slender thin rod is more likely to buckle than a thick rod. Adequate buckling resistance is important for exploring canal orifices and negotiation of narrow canals. For measuring buckling resistance, the instrument handle was connected to a device, and the file or wire is loaded in an axial direction. The tip was restrained in a point. The maximum load was defined as the buckling resistance. [10] (Figure 7)

Figure 7: buckling

2) Failure due to static overload

The Fracture usually occurs in the form of ductile or brittle fracture. This is based on the ability to experience plastic deformation.

a) <u>Brittle fracture</u>

Brittle deformation occurs through rapid crack propagation without any appreciable deformation. The direction of the crack is perpendicular to the applied stress, and it occurs as a flat surface. Brittle fracture in the ceramic glass shows a shiny, smooth surface. [11]

<u>Microscopically</u>:

Brittle materials fracture in the form of a v-shaped marking (Chevron) near the center of the fracture that points back to the crack initiation site. Other brittle fracture surfaces contain lines radiating from the origin of the crack in a fanlike pattern. The fracture may be transgranular because the fracture crack occurs through the grain. The fracture surface may be grainy or faceted texture due to the change in orientation of cleavage planes from grain to grain. In some alloys, crack propagation occurs along grain boundaries which are called intergranular. [11] (Figure 8)

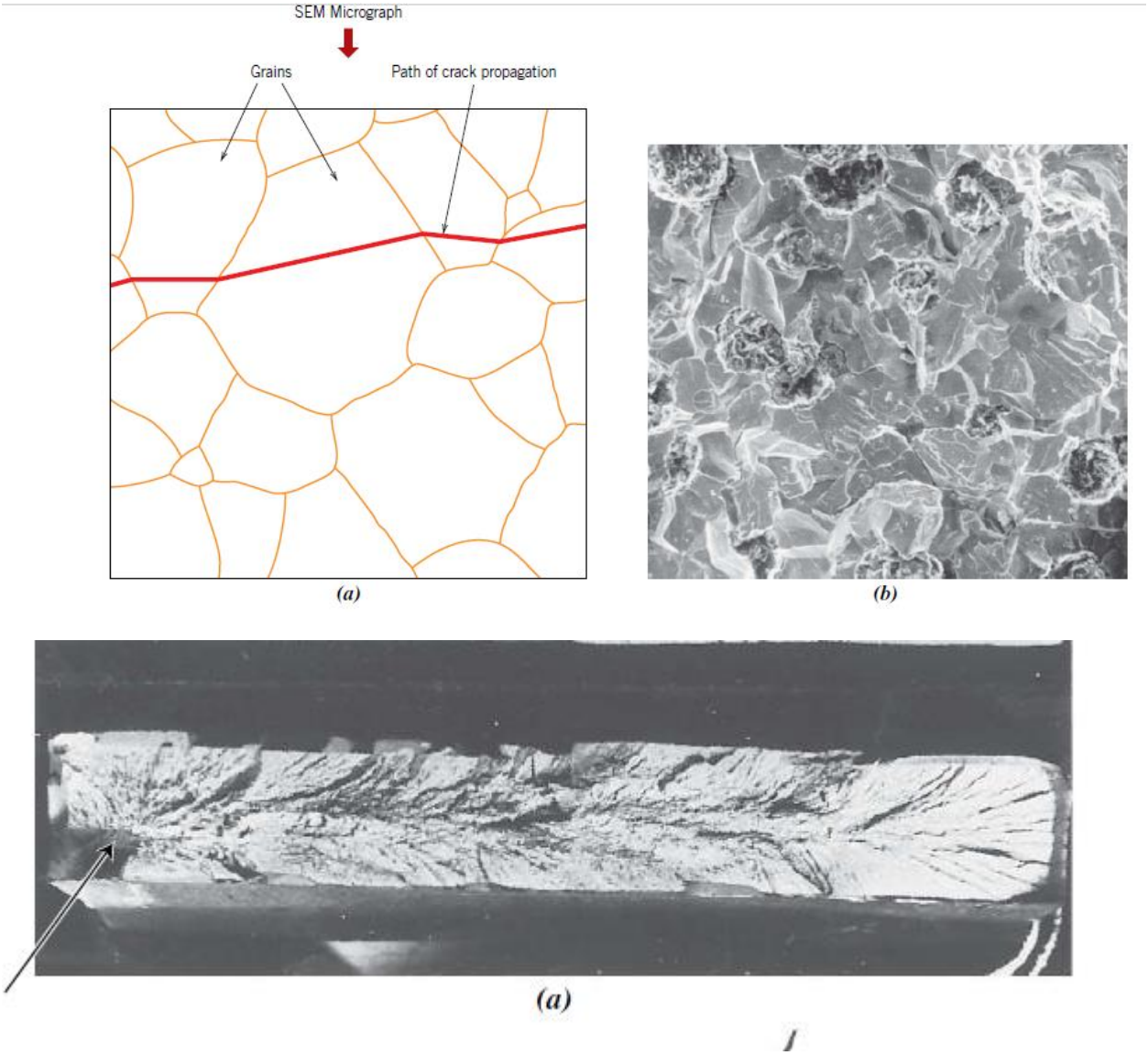


Figure 8: Brittle fracture

b) <u>Ductile fracture</u>

Ductile fracture is characterized slowly crack propagation forming necking down to the point of fracture. Ductile metals show a reduction in the cross-section before fracture. After necking, microvoids are formed in the interior of the cross-section, micro voids increase in size and coalesce to form an elliptical crack. The crack grows in a paralleled direction to its major axis. The fracture has a cup and cone fracture form. The central interior has an irregular and fibrous appearance. [11]

Microscopically:

When the cup and cone fracture is examined, it is found to consist of numerous spherical "dimples". Each dimple is half a micro void that formed and separated during fracture. These may be elongated or –shaped.[11] (Figure 9)

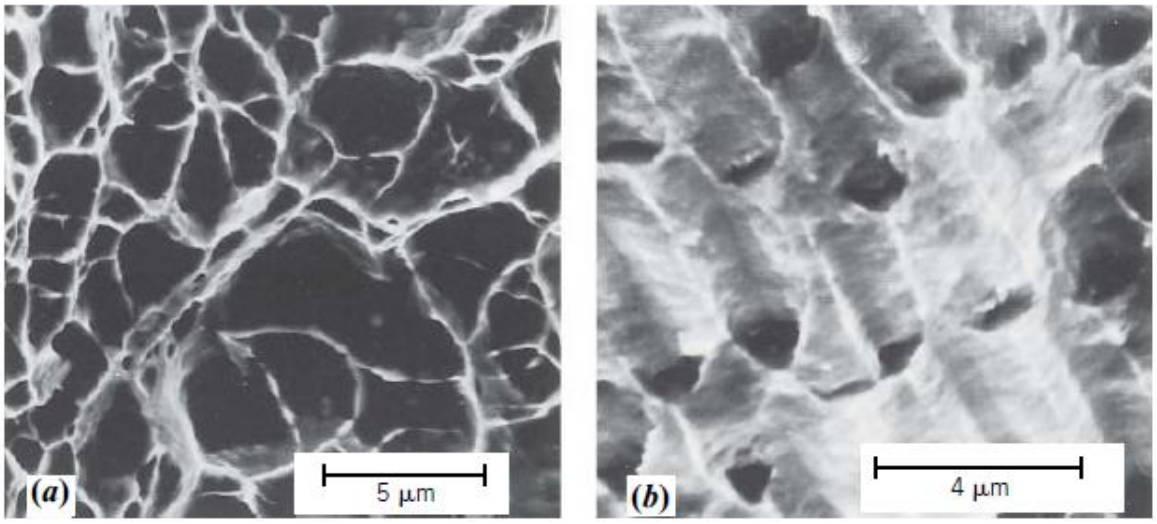


Figure 9: ductile fracture

- **Fracture toughness:**

Fracture toughness is used to describe the fracture resistance of the material. It is used to measure the resistance to brittle failure. Fracture toughness (Klc) is important in measuring amalgam, acrylic denture base, composite, ceramics, and orthodontic brackets. A fracture toughness test is performed using flexure bars with a notch at the tip with the crack of nanometer size tip.[11]

Fracture strength can be obtained according to Griffith's equation:

$$\sigma t = \frac{1}{Y} \frac{klc}{\sqrt{C}critical}$$

Where:

Y is the geometrical factor

Klc is the fracture toughness

Ccrit is the critical size of the crack.

i) Indentation method

Vickers indentation was made in the middle of a beam. The radial crack serves as a pre-crack in the test. This crack takes time to grow, so the beam is loaded after 20-30 minutes at load 19.6 N, and then the sample is subjected to

three-point bending set up till fracture. Specimens, where the fracture didn't originate from Vickers indentation, were excluded. [12]

Fracture strength was obtained from the following formula: f= $\frac{3WL}{2bd2}$

ii) Single-edge notched beam method

SEVNB method has been used for measuring the fracture toughness of ceramics due to its simplicity. The methodology involves a bar-shaped specimen that receives a narrow notch on one of the largest surfaces perpendicular to the specimen's long axis. The notch is produced by the diamond disc in a cutting machine. The notch root radius should be up to the same size as the major microstructural feature to validate the test, so it is not used with Yttria-stabilized zirconia. After notch preparation, the specimen is a fracture with the notch on the tensile side. [12] (Figure 10)

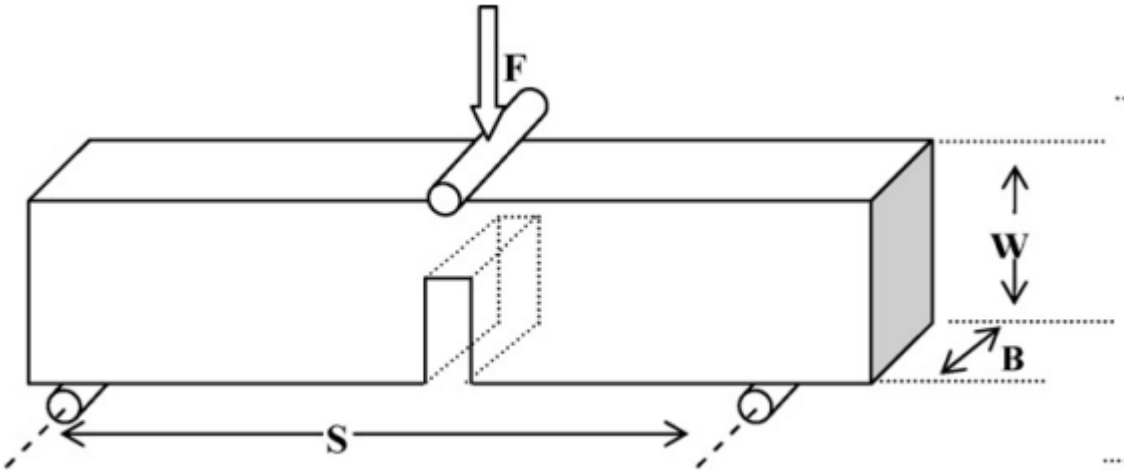


Figure 10: Single edge notched beam method

iii) Chevron notched beam method

Chevron notched beam requires a small amount of material for specimen preparation. The test can be performed with a short rod with the insertion of V shape chevron notch which is difficult to be achieved by cutting discs. During the test, a crack will develop from the tip of the chevron notch and progress as the load is increased till fracture. This method is useful in measuring fracture toughness for ceramics and composites. It can also be used with yttria-stabilized zirconia ceramics.[12,13] (Figure 11)

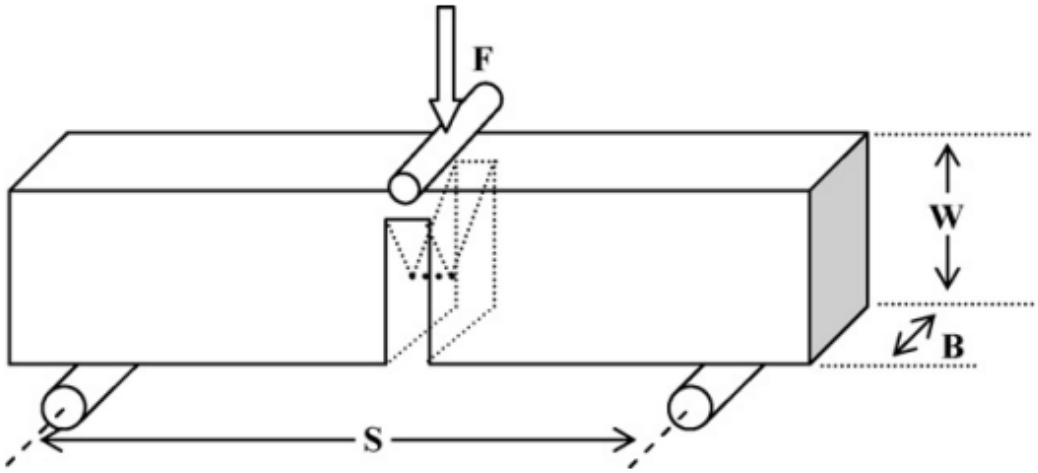


Figure 11: Chevron notched beam method

- **Impact testing**

Impact testing was used to measure the fracture of the material under high loading. The impact failure test is used to describe the behavior of the brittle material. Two standardized tests, Charpy and Izod to measure the impact energy. For both tests, the specimen is a bar or square cross-section, into which a V-notch is machined. The load is applied as an impact blow from a hammer at a fixed height. The main difference between Charpy and Izod techniques is the specimen support manner. Charpy and Izod are used to measure the ductile to brittle transition.[11] (Figure 12)

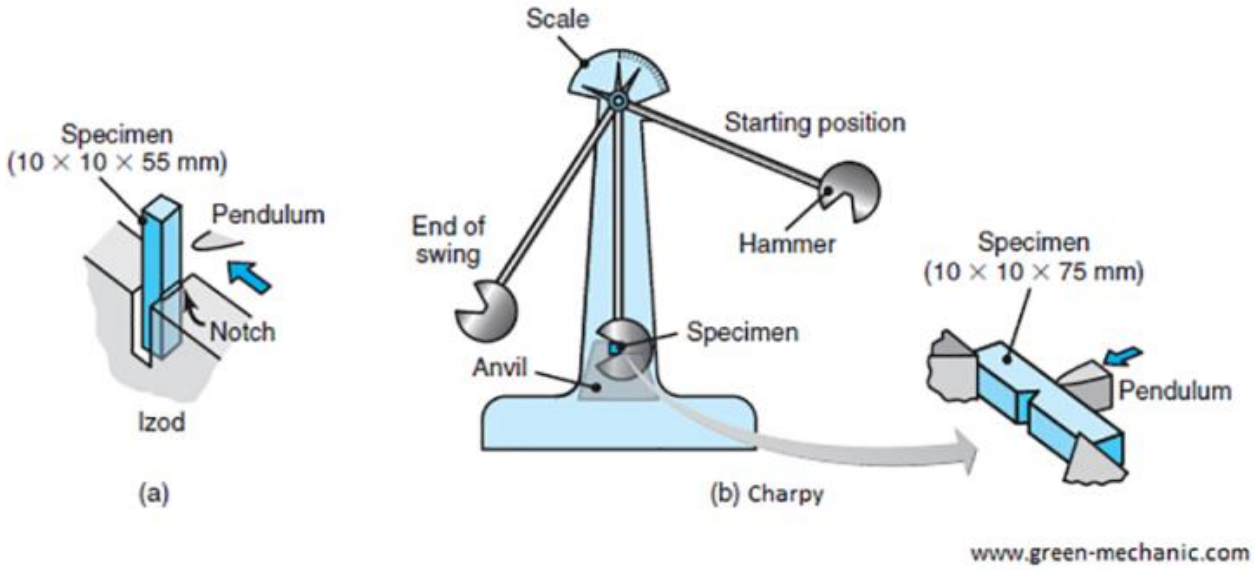


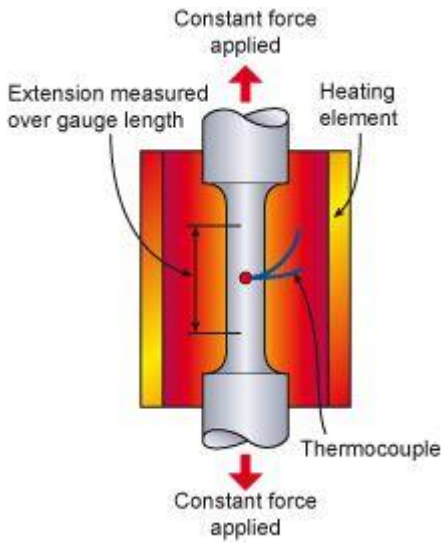
Figure 12: a) Izod test b) Charpy test

3) <u>Creep</u>

Creep is time-dependent permanent deformation when subjected to constant load or stress. Creep is observed when the temperature is greater than 0.4, the melting temperature. [11]

1) <u>Tensile creep testing:</u>

The Creep test is performed by a tensile specimen to whom constant stress is applied by simple methods of suspending weights from it. Around the specimen is a thermostatically controlled furnace. The temperature is controlled by a thermocouple attached to the specimen usually in the gage length. A constant tensile stress machine allows evaluation of tensile creep at 0-60°. A special loading arm, as the specimen lengthens by creep, and this reduces its cross-section, the moment arm shortens. Temperature control is achieved by heated water baths controlled by thermistors. This test is used with metallic materials.[14] (Figure 13)



(figure 13: tensile creep testing)

2) <u>Compressive creep testing</u>

The creep test is performed by applying compressive loading to eliminate the growth of cavities and their opening, so the creep rate in compression is slower than tension. Creep tests are better to be performed under uniaxial conditions such as tension or compression because the analyses of the uniform stress results are simple. According to ISO specification (ISO 1559), and ADA no.1 for amalgam testing creep refers to the deformation of amalgam under compressive stresses of 36 MPa of the specimen. The specimen of 4 mm in diameter and 7 mm in height were subjected to the compressive stress of 36 MPa for 4 hours at 37" C The change in length between one hour and four

hours shall be recorded after they had been stored at 1, 2, 4, 7 days. The reduction in length was measured using a disc transducer. Uniaxial compression testing is conducted with brittle materials. [15]

3) Creep rupture test

The creep rupture test is also called the stress rupture test. These tests are continued until the specimen fractures. The creep rupture test is a method of measuring the amount of creep material to withstand until it ruptures. This includes given stress and temperature at a definite hour. The specimen is heated using a furnace with a temperature-controlled device to ensure that the specimen temperature is maintained. The required load is applied by a system of dead weights. The length of the specimen is monitored using an extensometer attached to the specimen. Creep ductility of the material is obtained by comparing the length of the specimen after the rupture with the initial length. After a series of tests at different stresses, the time to rupture is measured as a function of the initially applied stress [16]

4) Fatigue

Fatigue strength is measured when the repetitive application of a small load to a material results in a fracture. Fatigue strength is measured by bending or twisting a test sample and counting repetitive stress cycles. Fatigue failure is an indication of failure on repetitive loads over a long period. A test apparatus should be designed to duplicate the service stress conditions (Stress level, time-frequency, stress pattern). The most common type of test is the rotating bending beam with alternating tension and compression stresses of equal magnitude as the specimen is bent and rotated. During rotation, the lower surface of the specimen is subjected to tensile stress whereas the upper surface experiences compression (negative stress). In service, conditions may call for conducting the fatigue tests using either uniaxial tension-compression or torsional stress cycling instead of a rotating bending beam. [11] (Figure 14)

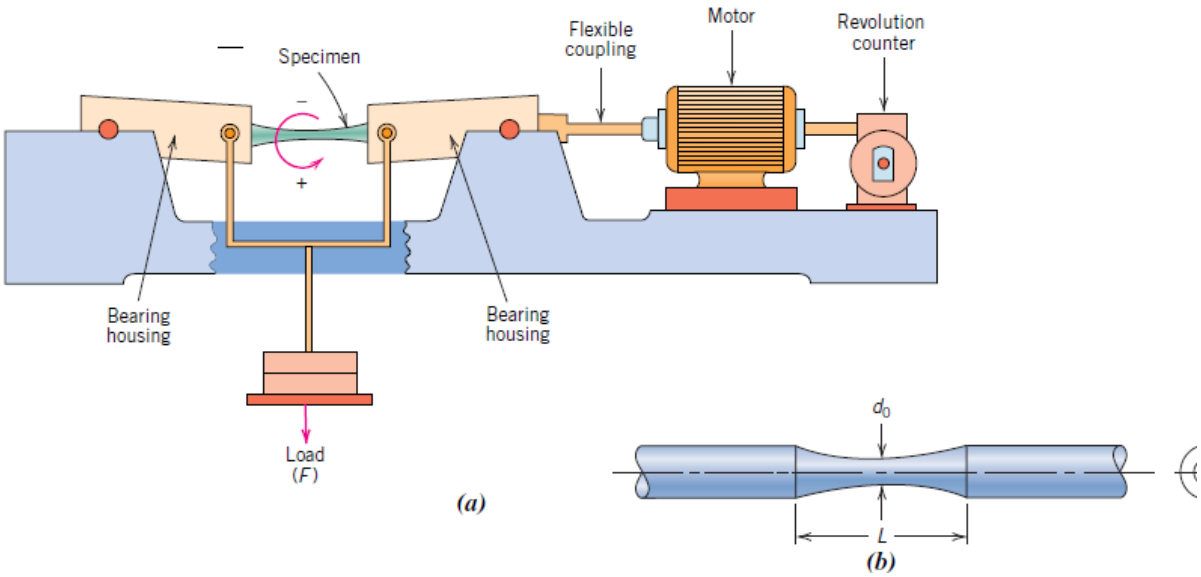


Figure 14: Rotating bending beam

Microscopic features:

Cyclic loading produces microscopic discontinuities resulting from the dislocation slip and cracks initiation sites. The region of the surface fracture appears as benchmarks and striations. This indicates the position of the crack at some point and as concentric ridges that expand from the crack initiation site. Benchmarks are of macroscopic dimensions. They are found as interruptions during the crack propagation stage. Each beachmarks represents a time over which crack growth occurred. However, fatigue striations are microscopic and can be seen by TEM or SEM. Each striation represents the advance distance of a crack during a single load cycle. Beachmarks and striations are fatigue fracture surfaces having similar appearances but from different origins and sizes. There may be thousands of striations within a single beachmarks.[11] (Figure 15)

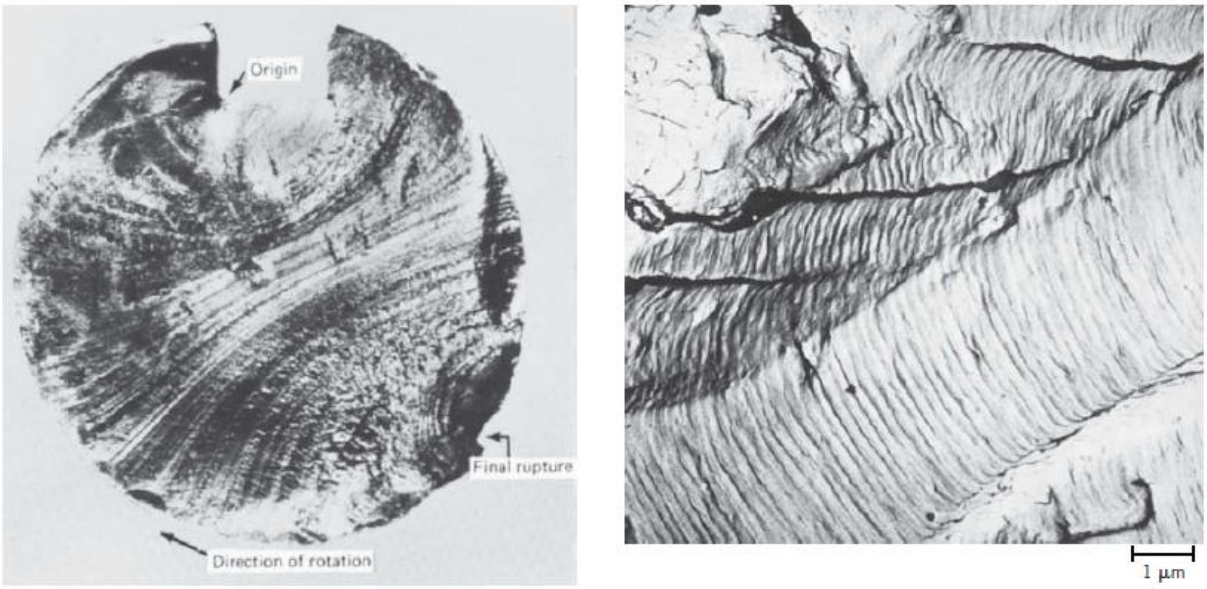


Figure 15: Beachmark and striations

5) Hardness

Hardness test is defined as a measure of how the material resists localized deformation. This can be done by applying a standard weight to an indentor. This produces a symmetrically shaped indentation that can be measured under a microscope for depth, area, and width. [1]

a) Knoop hardness testing

Knoop hardness testing was developed for ceramics, plastics, and thin metal sheets. In general knoop microhardness testing uses loads of no greater than 1kgf and the indentor is pyramidal shape. [7]

b) Vickers hardness testing

It has a diamond indentor to form a square-shaped indentation. It's used with brittle materials. It's used to determine the degree of polymerization of dental composite resin. [7] (Figure 16)

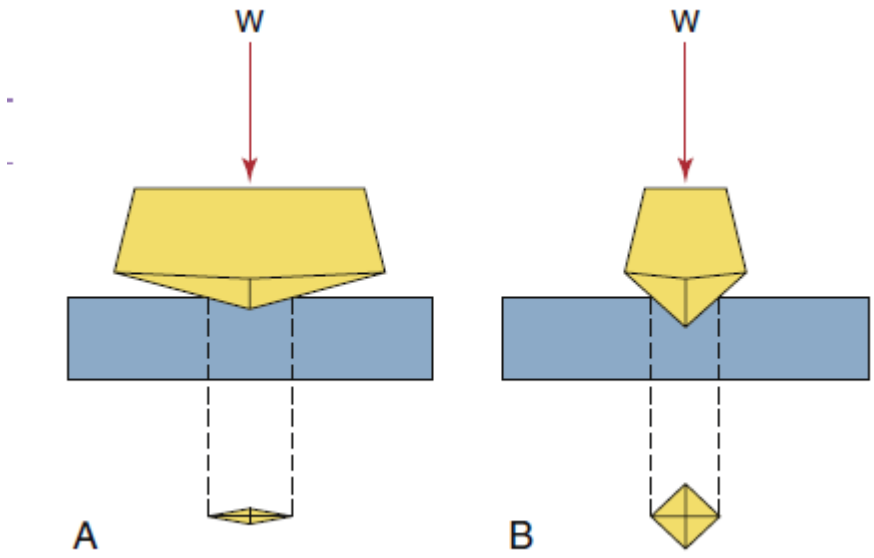


Figure 16: A) knoop hardness testing b) Vickers indentation

c) Rockwell hardness testing

Rockwell test is suitable with viscoelastic materials. It has a steel ball indentor. It applies a minor load of 3kg and then a major load exceeding 10 kg is applied. [7]

d) Shore A:

It is used with elastomer as rubber to measure the relative hardness. It is used with soft denture liners and mouth protectors. The instrument consists of a bluntly pointed indenter 0.8 mm in diameter that tapers to a cylinder of 1.6. If the indentor completely penetrates the specimen the reading is 0, and if no penetration, the reading is 100.[7]

6) Wear measurement

a) Pin on disc tribometer:

It is one of the most common methods for wear testing. The base of method is based on using a disc-shaped sample surface on a pin body in the form of a roller or non-rotating ball. At a chosen distance from the sample center, the PIN is stressed by a predetermined force. The disc starts rotating with a selected speed and number of rounds.[17] (Figure 17)

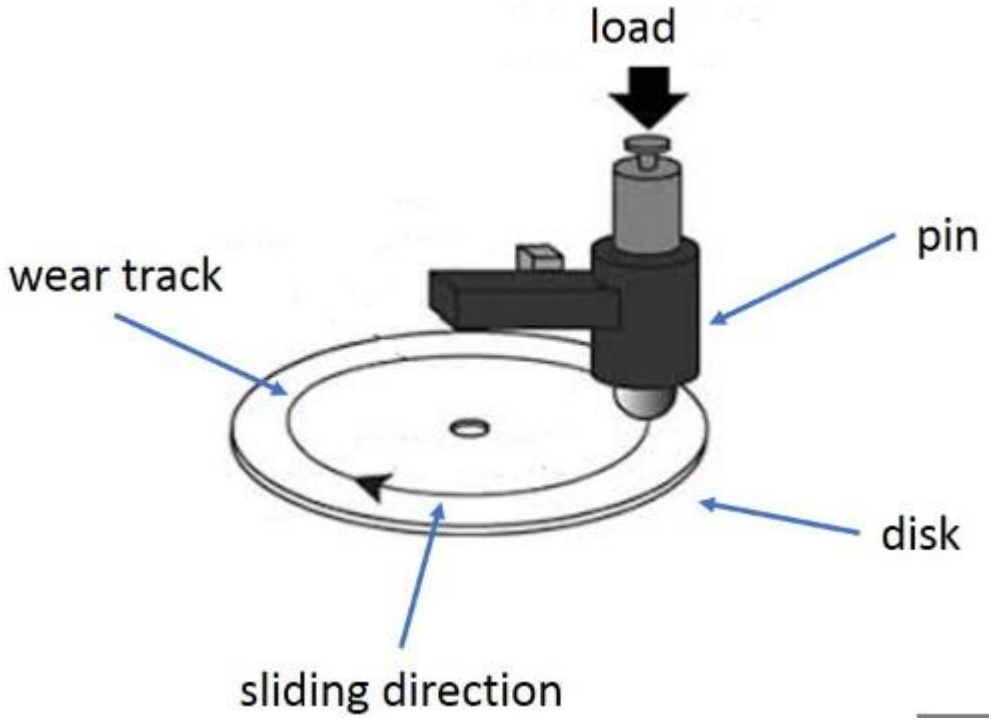


Figure 17: Pin on disc tribometer

b) Reciprocating tribometer

Similar to the tribometer, a pin or a ball is in contact with a flat specimen under a vertical force causing constant pressure, but it moves along a linear stroke alternately back and forth with a known frequency. It is commonly used with prosthesis material. It can be used with artificial saliva. [17]

c) Chewing simulator

Chewing similar is the only equipment to reproduce impact and sliding motion occurring during mastication. It can simulate vertical and horizontal movement by a definite stroke for horizontal sliding and a height for the descending movement. [17]

7) Tear strength

Tear strength measures the resistance of the material to tearing forces. It is an important property for polymers and impression materials. It is tested using a specimen crescent-shaped and notched. The tear strength is measured by dividing the maximum load by the thickness of the specimen. A high rate of loading will result in higher tear strength. [7]

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