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Type of the Paper (Editorial)

Biocermaics in endodontics

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Abstract: Bioceramics are materials which comprise alumina, bioglass, glass ceramics, zirconia, hydroxyapatite and other calcium phosphates. They have been used in dental field as bone filling materials, root repair materials, root-end filling materials, and as endodontic sealers. They have certain advantages as being biological compatible, inert, non-toxicity, dimensionally stable. They have a similarity to the natural minerals part hydroxyapatite which is the main component of hard dental tissues. Bioceramics are characterized by osteoconductive activity bioactivity. In endodontics, they can be generally classified into calcium phosphate; tricalcium; hydroxyapatite based; calcium silicate based or mixtures of both calcium silicate and phosphates. The shift from previously established bioceramics to newly advance formulations is too slow, and there is an urgent demand to boost clinical research.

Keywords: Ceramics; biocearmaics; endodontics.

Ceramics are defined as inorganic, non-metallic materials produced by the heating of raw minerals at higher degree of temperatures. Bioceramics are biocompatible ceramic materials or metal oxides with improved sealing capability, antibacterial and antifungal element is added to induce anti-microbial activity.

The mechanism of action of bioceramics depends on direct replacement of human tissues or being biodegraded to encourage the regeneration of natural tissues. They include alumina and, bioglass, glass ceramics, zirconia, calcium silicates, hydroxyapatite and other calcium phosphates.

Ceramics have been used in dental field as bone filling materials, root repair materials, root-end filling materials, and as endodontic sealers. They have certain advantages as being biological compatible, inert, non-toxic, dimensionally stable. They have a similarity to the natural minerals part hydroxyapatite which is the main component of hard dental tissues.

Bioceramics are characterized by osteoconductive activity bioactivity. In endodontics, they can be generally classified into calcium phosphate; tricalcium; hydroxyapatite based; calcium silicate based or mixtures of both calcium silicate and phosphates. Bioceramics utilized in endodontics mainly includes; calcium silicates; Portland cement; mineral trioxide aggregate (MTA). Mixtures of calcium phosphates, tricalcium phosphate and hydroxyapatite.

However, MTA was the gold standard endodontic bioceramic materials, enhancement in material should be done to overcome their disadvantages specially their physical properties and handling characteristics. Bioceramics now have a wide array of

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applications both in endodontics and restorative dentistry. A novel bioactive material is essential to ensure the selection of the most appropriate material in different clinical circumstances.

In conclusion, There is a need for extensive research to identify new bioceramics or changing the existing one to improve their beneficial properties. The development shoud be focused to provide a bioceramics easily to be handle, have excellent antimicrobial activity and alkaline pH. Bioactive property to be able to release calcium and phosphprus ions, promoting marginal adaptation and sealing ability, shorter setting time, biocompatibility, not cytotoxic, promote dentin remineralization. They can also be utilized in humid environment and are easily removed in cases of retreateemnt, have maximum adhesion to dentin, improved mechanical properties, not encourafe tooth discoloration.

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Type of the Paper (Review Article)

Antioxidants used in dentistry

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Abstract: Recently, many dental fields shift to the use of natural products such as antioxidants as it shows a huge effect in relieving several dental diseases.

Keywords: Antioxidants; dentistry.

Introduction

Many natural products rich in antioxidant activity are used in eliminating the reactive oxygen species and oxidative stress.

1. Reactive oxygen species

Most free-radical causing biological damage is known by Reactive Oxygen Species (R.O.S), which is an oxygen-derived molecule that comes from super-oxides and hydrogen peroxidase. They come from the surrounding atmospheric pollutants, UV light, and irradiation. It produces oxidative stress which plays a characteristic role in the formation of many diseases that may affect the oral cavity that can be prevented by using antioxidant products. (1)

2. Antioxidants

They are considered the latest interest in different fields as several factors are increasing the presence of the free radical, antioxidants are a substance known as a scavenger, it protects the body against the toxic effect of the free radical that may cause slow cell damage and harmful effect. Antioxidant materials have a wide spectrum of usage in many medical fields and clinical practice, especially in dentistry. Their usage can be assembled in the majority of the dental field in restorative, periodontics, endodontics, orthodontic, and oral maxillofacial surgery. (2)

2.(A). Mechanism of action

It has several mechanisms of action; the first form is the prevention which prevents the formation of free radicals, the second one is the chain breaking form by the termination of a chain reaction, the third one has a repair function. (1)

2.(B). Sources of antioxidants

Antioxidants can be synthesized normally in the body or obtained from multiple dietary sources, they may be endogenous obtained from enzymes, exogenous which come from outside the human body obtained from minerals, vitamins (A, E, C), Beta-carotene, lycopene, food (plant-based food) and herbal supplements. There are many compounds has antioxidant activity derived from plants as phenolic derivatives and flavonoids as well as terpenes, catechin, tannins, and catechins. (3)

Antioxidant products can be presented mainly in different dental products such as mouth rinses, toothpaste, and oral sprays. It was found that most dental materials such as resin composite filling, dental implants, bleaching agents, dental cement, metal, ceramic restoration, and intercanal medication may form free radicals. Therefore, there are several new approaches to adding antioxidants products in many dental materials. (4)

2.(C). Most common antioxidant products used in dentistry

Ascorbic acid (vitamin C) is a water-soluble antioxidant, which is found mainly in citric fruit like orange, lemon, grapes, and colored pepper. their deficiency may affect gingival health.

Vitamin c has several modes of action on the free radicals, scavenging, preventive form. Also, it is known as an oxygen scavenger antioxidant as it reacts with oxygen. (1)

α-tocopherol (vitamin E) is a lipid-soluble antioxidant, it is derived from unsaturated fats, olives, and oils. It presents normally in body plasma and is diminished in smokers. It can maintain cell membrane integrity. But It depends on another antioxidant for its action as ascorbic acid. It has an abundant role in chain breaking as an antioxidant. (1)

Carotenoids are a huge family of antioxidants. They are highly found in red, yellow fruits and vegetables such as carrots and pumpkin. They are classified as lycopene, α-carotene, β-carotene, and retinol. While Polyphenols are found naturally in vegetables, fruits, and green tea. They are Composed of huge compounds, most common (flavonoids, anthocyanins, catechin). They act as a free radical scavenger. (5)

Aloe Vera is a natural plant which has a medicinal effect, it is derived from the Asphodelaceae family and the genus 'Aloe'. Aloe Vera Barbadensis is the most common and useful species, showing a wide variety of uses, especially as an antioxidant in different dental applications. It is provided in many forms: gel, spray, added to toothpaste, mouth rinses, and several dental materials.

It blocks mainly the radical effect by donating a hydrogen atom. Aloe Vera shows wide application as an antioxidant after extraction due to its wound healing activity, used after tooth bleaching to eliminate the harmful effect of hydrogen and carbamide peroxide that may affect enamel and dentin, and it plays a significant role in treating aphthous stomatitis. (6)(7)(8)

Propolis is derived from bees and has various uses in dentistry in the relief of dentin hypersensitivity. in addition to that, It can be used as pulp capping material and contains caffeic acid phenethyl ester which affects bone healing. (9)

3. Antioxidant materials used in restorative dentistry

3. (A). Role of antioxidants on dental caries prevention

Researches and studies exhibit those cranberries specifically their Type A oligomers and their role in dental caries prevention, which is caused by Streptococcus mutans which is one of the most common bacteria that induce caries formation and propagation. (10)

Eugenol has been already used in controlling toothache, it contains an enzyme activator for antioxidant effect. (11) Additionally, natural polyphenols in tea in large volumes as Epigallocatechin-3-gallate play a role in minimizing dental cavities and plaque formation. (2)

Antioxidant has relevant effects on remineralization of the carious tooth, dentin hypersensitivity, pulp capping, and bond strength after teeth bleaching. (10)

3.(B). Role of antioxidants on remineralization of carious tooth

In vitro study based on grape seeds that contain Proanthocyanidin (PA) combined with fluoride done on a sampled tooth, by comparing it to another untreated tooth, the result shows higher remineralization power on carious enamel and dentine. This is because of ceasing the carious propagation by adding calcium and phosphate ions to be deposited on the demineralized enamel. (12)

According to Hiraishi et al. study, Hesperidin found in Flavonic acid enhances remineralization of dentin lesions compared to chlorohexidine, this is because it protects the collagen structure of dentin, the study based on several samples that subjected to low PH solution for 14 hours to ensure the demineralization, then incubated in a solution of hesperidin and another one of chlorohexidine for 2 hours, by observing the results reveal that the sample in hesperidin solution has the lowest mineral loss due to stopping the demineralization process. (13)

3. (C). Role of antioxidants on treating Dentin hypersensitivity

Many approaches are done to occlude the exposed dentin with a certain material to decrease the pain, based on the hydrodynamic theory that state that unprotected dentin cause sharp pain and unpleasant sensation to the patient when subjected to any stimulus as chemicals, and thermals. (10)

Dentinal tubules occlusion can be done by laser, dentin sealer, toothpaste containing desensitizing agents such as potassium fluoride, or varnish.

Some treatment modalities include using antioxidant products to reveal the reduction of dentin hypersensitivity in a short time, the result can be shown in two days to two weeks. (10)

Propolis shows a huge impact in desensitization of dentin, in vivo study was done on 40 different patients complaining of dentin hypersensitivity, by comparing the effect of propolis with potassium nitrate and sodium fluoride and another desensitizing agent. The visual analog scale (VAS) was used to record the baseline measurement for pain due to hypersensitivity. After 7 days, the final score was collected again after the application of the appropriate desensitizing agent. The result shows that all of them have a potent effect but propolis has a rapid tendency in stopping the dentin hypersensitivity. (14)

3. (D). Role of antioxidants as pulp capping material

Reports of a split-mouth study carried on pigs provided propolis that has an excellent pulp capping ability and high-quality formed dentin compared to calcium hydroxide. The first step done in the study is removing the tooth crown and sealing the cavity using the testing materials then by scarifying some of the pig on the 10th, 15th, and the 30th days, the sectioning teeth were detected under a light microscope by using H&E stain.

The result shows that propolis form high-quality dentin and the pulp remains vital, while calcium hydroxide shows a porous reparative dentin, although it is the most used pulp capping material it shows drawback of its solubility. (15)

3. (E). Role of antioxidants on the bond strength after tooth bleaching

In the past the bleaching process should be preceded by restoring the carious or broken teeth because free radicals interrupt the bond strength of the restoration, now restoring the tooth can be done after bleaching by 24 hours to 3 weeks. Because of the power of Sodium Ascorbate, α-tocopherol, and Proanthocyanidin in neutralizing the free radical produced from carbamide peroxide or hydrogen peroxide decomposition after the bleaching process. (10)

Several studies and research assure the success of antioxidant activity by adding Grape seed or pine bark extract solutions to bond material to enhance some of their properties by increasing its bond strength which usually decreases after the bleaching process.

Another experimental study was done in vitro by using anterior extracted tooth, then preparing it to a bleaching process, and comparing it with two control groups one with laser bleaching, then by the application of antioxidant product especially ascorbic acid, α-tocopherol, hesperidin before using a bonding agent and composite restoration. The results show great success after testing the shear bond strength, as they eliminate the oxidative effect of bleaching. (16)

4. Antioxidant materials used in periodontics

Human saliva considers a natural antioxidant compound, it is observed by studying salivary components that it is rich in uric acid, albumin, ascorbic acid, glutathione, and antioxidant enzymes. This facilitates the salivary functions in lubricating the oral cavity that led to a reduction in plaque and bacterial retention hence, decreasing dental caries formation, protecting oral mucosa, and maintaining oral health. (17)

An in-vitro study based on cranberry components shows their potent role in blocking the formation of Porphyromonas gingivalis which is a prominent pathogen that causes chronic periodontitis. the researcher used microplate assay and investigation of cranberry solution on the cell by using a scanning electron microscope. The result shows the ability of cranberry in reducing the quantity of p.gingivalis to aggregate in the periodontal site. (18)

Another experiment that depends on using vitamin E reveals its antioxidant effect on reducing periodontitis although it does not affect alveolar bone loss.

Also, Aloe Vera has an antioxidant effect on relieving oral ulcers and enhancing their healing. Therefore, maintaining good nutrients, vitamins, and antioxidant products in form of mouthwash and toothpaste can control the reduction of periodontal and gingival diseases. (19)

5. Antioxidant material used in Endodontics

Lycopene as well as Aloe Vera has a sedative effect as an antioxidant material therefore, it is used as dressing in root canal treatment and an intracanal medication.

In vitro study based on using a mixture of proanthocyanidin (grape seed extract) and lycopene to evaluate their effect when used as intracanal medication. Four samples of the mixture were used then the antibacterial property was tested using the agar diffusion method. The result proved that lycopene shows more antioxidant properties when compared to proanthocyanidin as intracanal medication. (20)

6. Role of antioxidants in preventing and treating oral cancer

Dietary antioxidants consider a preventive and therapeutic method of oral cancer. Research on animals stated the successful modalities of antioxidants intake in inhibiting oral cancer and decreasing oral carcinoma growth, Antioxidants have the ability of shielding lipids and other membrane molecules against oxidative damage by absorbing oxidants before they can cause tissue damage.

β-carotene and vitamin E have a significant role in controlling oral cancer, in 1960 vitamin E was the best option for treating oral leukoplakia by studying also lycopene and β-carotene it was found that they have a therapeutic effect on oral leukoplakia. (2) (21)

Also, Retinoids as well as aloe vera and has potent antioxidant activity on treating Oral Lichen Planus systemically or topically. (7)

There is a study to evaluate the effect of aloe vera on oral lichen planus (OLP) done on 21 female patients with OLP , they are grouped into three groups, the first two groups received the aloe vera gel with different concentrations on the lesion, while the third one has received the placebo and is considered as a control group, each group was instructed to use the aloe vera gel twice a day on the dry lesion after meals, baseline measurement of pain was taken before and after treatment using the visual analog scale, the size of the lesion was measured quantitatively using a calibrated grid, the result show improvement and reduction in the size of the lesion with the first two groups. (22)

7. Role of antioxidant material used in bone formation

Alveolar bone loss considers as one of the common diseases that affect the oral cavity and has a bad effect on different aspects, scientists use mice and small animals to study the effect of ROS and oxidative stress on the formation of alveolar bone loss and the initiation of peri-implantitis, which is formed in the subgingival region due to aggregation of anaerobic bacteria. (2)

Grape seed and propolis specifically caffeic acid phenethyl ester have a noticeable enhancement of bone formation and healing, therapeutic action on peri-implantitis in the animal study. (2)

In vivo study done on male rats with a bone defect, they are divided into three groups one for control and the other two groups with the testing materials, an incision is done after anesthetizing the rats, bone drilling was done with continuous irrigation then bone graft added with the testing materials and sutures were done in the periosteum and skin, followed by a single dose antibiotic injection, follow up is done and by scarifying seven rats on 7th, 15th and 30th day, the histopathological evaluation shows bone regeneration and healing enhancement from caffeic acid phenethyl ester. (23)

Also, eugenol is used in rapid wound healing, in paste form as a dressing material after tooth extraction. (11)

More research and studies are needed to elaborate the mystery of the side effect of antioxidants, the average dose, and the quantity to be used and added in dental material. Since high doses may have serious health complications such as teratogenicity and toxicity. Most of the studies done using antioxidants are in vitro or on animals, further clinical trials are needed to help their progress in the dental field. (2)(19)

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Creep in dentistry: a review

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Abstract: Materials are often placed in service in the oral cavity at elevated temperatures and exposed to static stresses. Deformation under these conditions is called Creep. Creep is normally undesirable and limit the lifetime of the material.

Keywords: *Creep, Creep in dentistry, Creep mechanism, creep testing.*

Introduction

Creep is a viscoelastic property of the material. It's a time-dependent permanent plastic deformation of materials when subjected to a constant load below its yield strength at a temperature greater than 0.4 the melting temperature. [1]Creep is observed in all materials, it's observed at a temperature greater than 0.4 the melting temperature.

Creep rupture is the fracture of the material due to the creep process.[1]

Creep Curve:

Creep curve has insights into the relative elastic, viscous and inelastic response of the viscoelastic material. 2 Creep recovery curve is obtained from data collected during load removal. In this curve, after the load is removed, there is an instantaneous drop in strain and slower strain decay to steady-state strain. The instantaneous drop is the recovery of the elastic strain. The slower recovery is the inelastic strain, and the remaining permanent strain is the viscous strain2 (Figure 1)

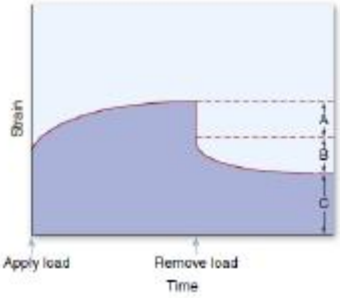

Figure 1: Creep recovery curve

Stages of Creep:

Creep consists of three regions; each has its own strain-time feature.

- Primary creep (Transient creep): In the primary stage, the strain rate is high, and it decreases with increasing time and strain. This is because of increasing the creep resistance or strain hardening.

- Secondary creep (Steady state creep): the creep rate is small and the strain increases slowly with time, so the plot is linear. This is the longest creep duration. Secondary creep is the dominant in most applications. This constancy is due to the equilibrium between softening and hardening.

- Tertiary creep: The strain rate increases rapidly until rupture, and the formation of internal cracks, cavities and voids.

Creep Mechanism:

1) Creep by dislocation slip

a) Glide

It occurs at high stresses and relatively low temperature. It involves motion of dislocation especially in metals where the density of dislocation is high. At stress below yield strength, no dislocation glide occurs. When the stresses are approaching the yield strength for long time, progressive dislocation occurs.

b) Climb

When dislocation glide reaches an obstacle, the stress is not enough for dislocation to overcome the obstacle, so dislocation climbs to a parallel slip plane. As the temperature increases, the atoms gain energy, so the vacancies in the metal increase. Vacancies should be at the site where climb is supposed to occur to allow vacancy-atom exchange.

2) Diffusion Creep

It occurs at high temperature and low stresses compared to the dislocation motion.

a) Grain boundary

The shape has changed from atoms becoming redistributed by diffusion. This type of dislocation occurs through the grain boundaries, it is referred to Coble Creep. Increasing the temperature increases the creep rate as the rate of diffusion become higher due to the high temperature. The activation energy for the grain boundary diffusion is low, and the cross sectional area for boundary diffusion is less than the bulk. So it occurs at low temperature and for samples with fine grain size.

b) Bulk diffusion

It occurs within the crystal lattice of the grain rather than the grain boundaries. It is called Nabarro-Herring creep. The cross sectional area via crystal lattice is high, especially if the grain is large. Bulk diffusion is temperature dependent, the activation energy is higher. This type of creep occurs at higher temperature and with large grains or single crystals.

c) Grain boundary sliding

In polycrystalline materials, the diffusion creep must be accompanied by grain-boundary sliding to reserve the material continuity. IR mainly occurs at grain boundaries in normal polycrystalline material undergoing diffusion creep.

How to measure Creep?

The major methods for testing creep are:

1) Tensile creep testing

2) Compressive creep testing

3) Flexural testing for creep

4) Impression (hardness) creep testing

5) Stress rupture test

The results of creep tests are plotted as strain-time curves. For metallic material, creep tests are conducted in a uniaxial tension having the same geometry as tensile test. However, for brittle materials, uniaxial compression tests are conducted as no stress amplification and crack propagation.

1) Tensile creep testing:

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.

The extension of the specimen is measured with an extensometer.The results of the creep test are plotted as strain versus time to obtain a curve.[3]

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 bath controlled by thermistors.

The test specimen is based on a standard tensile specimen, it should be proportional, and it should be machined to a tighter tolerance than the standard tensile test.

The specimen should be smooth, scratch-free.[4]

Testing is carried out in the air at atmospheric pressure. If the material reacts with air, they are tested in a chamber contacting an inert atmosphere such as argon or vacuum.

Creep failure occurs in three phases, a rapid increase in length which is known as primary creep where the creep rate decreases. Then it's followed by a period of constant creep rate known as secondary creep, and it is the longest period of the creep life. The third stage occurs when the creep life is exhausted, and the cross-section has been reduced. This stage fasts until the specimen finally fails.

The creep test measures the rate at which secondary creep occurs. Increasing the temperature of the stress has the effect of increasing the slope. The results are presented as the amount of strain expressed as a percentage produced by aplying a specified load.

2) Compressive creep testing

The creep test is performed by applying compressive loading to eliminate 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. [5]

3) Flexural creep testing:

Many creep experiments employ flexure tests. There are two common testing methods, three and four-point bending tests.

The specimens are rectangular and without notches. The four points bend setup where the applied force in the lower part is tensile, while the applied force in the upper part is compressive. As a result, calculable bending develops. The machine used is the universal testing machine.

The flexural strength for three-point test of the rectangular bar is applied through force acting on a lever of size L/2, and the force is supported or balanced at the two supporting points marked by arrows near the end of the rectangular bar.

Three-point bending mode is a pure modification of deformation, as the specimen is freely supported by fulcrum without a clamping effect. Constant stress is applied to the surface as the upper arm contacts the specimen, and the resulting strain is measured over time. Once the stress is removed, the recovery strain is measured.[6]

In the paper below, the creep was measured using DMA in three-point bending mode. The specimen is freely supported by fulcrum, a constant stress is applied to the surface as the upper arm contacts the specimen, and the strain is measured overtime. The testing conditions of DMA with a small preload, a controlled temperature of 23°C.

In another paper, the creep was measured using DMA, it used flexural deformation for creep measurement.

The specimen was mounted as cantilever beam on two arms, that forms a predefined displacement. A computer was used for instrument control, data collection and data analysis. It uses a driver signal to generate the deformation. When the driver signal was active, the specimen was subject to a constant stress, and the arm position data was collected as a function of time to measure the creep deformation. When the driver signal was turned off, the arm position followed the material recovery and the arm position data was collected as a function of time to monitor the recovery.

As the computer signal was based on fixing the initial flexural amplitude of deformation for creep measurement, the amplitude corresponding the driver was determined.The creep measurement was done at temperature 37 C

Creep data was carried out for 120 minutes, and the recovery data was collected for additional 210 minutes until the driver signal reached a constant value. The analysis of the data was done using DMA.

4) Indentation (Hardness test)

Indentation creep is the percentage increase in indentation depth under constant loading over a while. Since conventional creep testing needs many specimens to establish stress and temperature, indentation hardness is used as a substitute. Many ways have been proposed to test the time- dependent hardness. The indentor is circular with a flat end. [7]

The indentation creep was measured using the indentation length plotted versus the indentation time at constant load.[8]

The indentation length increases with the loading time and the applied load. The curve consists of two stages similar to an ordinary creep curve. The first stage is the increase in the indentation length with the loading time, with a decreasing rate. Followed by a steady-state where the indentation size increases linearly with time. During the hardness test, no fracture of the specimen, so it is no possible to record the third stage of the curve to what happens in an ordinary creep test. [9]

CIT = [(h2 − h1)/h1]*100%

Microhardness is measured by taking the depth of penetration of the indentor as the function of load. This allows the determination of the deformation under a load of indentations under increasing stable and decreas-ing load over time.A schematic diagram of the variation of the load over time is plotted. The load increase and the penetration

depth of the indentor was measure as a function of time (Δh). The load and the penetration depth were registered on the x-y graph. The measurement was made at the start of the creep, and after 10, 20, 30 and 60 seconds, and after 60 seconds up to 600 seconds.[9]

Indentation creep is used with the viscoelastic materials.

Nanoindentation creep test:

Nanoindentation provides a unique opportunity to measure the creep response of definite phases in complex microstructural materials that can’t be measured with conventional bulk techniques.[10]

Understanding the fundamental relationship between creep and microstructure is a way to develop improved materials.High temperature creep resistance is important in many applications of high temperature materials. [11]

To measure the indentation creep accurately, the test instrument must displays ultra-low thermal drift across the entire measurement.

Nanoindentation creep experiment includes creep test that are mostly made with three-sided pyramidal indenter.

5) Creep rupture test:

Creep rupture test is also called the stress rupture test. These tests are continued until the specimen fractures.

Creep rupture test is a method of measuring the amount of creep material withstand until it ruptures. This includes given stress and temperature at a definite hour.

Creep rupture test requires:

1) Method of heating the specimen to a constant test temperature

2) Method of applying a constant load

3) Method for measuring the increase in length

The specimen is heated using a furnace with the temperature controlled device to ensure that the specimen temperature is maintained. As creep a temperature dependent property, there is a relation between the temperature and the creep rate, so a small increase in the temperature leads to a large increase in creep rate.

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. [1]

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.

It is useful in specifying the design life of the components. Creep rupture test differs from other tests in that the creep test continues until rupture.

The material is fabricated to a known dimension. Then the material is placed under a load below yield strength. The temperature is elevated, and it reflects the temperature that presents in the application when the material is used.[4]

Factors affecting creep of materials:

1) Temperature

Creep is more pronounced at higher temperature. No creep occurs below 40% of the melting temperature

2) Time

Constant stress is maintained for long period of time.

3) Stress

Creep increases at higher load.

4) Alloy composition

Creep is dependent on grain size, and the grain size is dependent on the temperature.

a) If the material is exposed to high temperature (diffusion creep>dislocation creep): as it require coarse grain or single crystal. At high temperatures diffusion creep occurs.

b) IF material is exposed to low temperature function (dislocation creep>diffusion creep): fine grains are recommended to increase grain boundaries which act as obstacles, dislocations accumulate at the grain boundaries

(dislocation pile up), back stress is created that hinders any further movements, the material overcome theobstacles by the mechanism of climb.

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Corrosion and degradation of metals and alloys in dentistry

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Abstract: Metal alloys have high strength and are competitive among other materials used in dentistry. The choice of a material for dental application, depends on several factors like corrosion behaviour, mechanical properties including strength and fabricability, cost, availability, biocompatibility, and aesthetic values. For metallic materials, the corrosion behaviour is the most important property to be considered, due to the biocompatibility and cytotoxicity of the products of the corrosion process. We will discuss the different types of corrosion of metals and alloys in dentistry and how to minimize it.

Keywords: *Dental corrosion, metals, alloys, chemical failure, degradation of metals.*

Introduction

Corrosion is defined as "the destruction or deterioration of a material because of reaction with its environment". Also, "it is a chemical or electrochemical process through which a metal is attacked by natural agents, such as air and water, resulting in partial or complete dissolution, deterioration, or weakening." [2, 3]. Corrosion always come as a consequence to tarnish, which is "a surface discoloration on a metal or a slight loss of the surface finish or gloss due to the formation of thin films, such as surface oxides, sulphides, or chlorides". Fontana [3] and Jones [4] classified corrosion into eleven forms. Only nine forms occur in the oral environment.

Mechanism & Fundamentals of corrosion

Corrosion occurs by chemical or electrochemical means. Chemical corrosion or oxidation occurs when oxygen in the air reacts with metal without the presence of a liquid. Typically, chemical (dry) corrosion is not as damaging as electrochemical (wet) corrosion, but it is very sensitive to temperature. An electrochemical reaction involves metals, chemicals, and water that combine to form cells capable of generating electricity. The electrochemical cells contain four basic components: electrolyte, anode, cathode, and conductor. The electrolyte is the current carrying medium, such as water.

Definitions

1. Electrode potential: The difference in electric potential between an electrode and the [5]

2. High electrode potential (low dissolution tendency): If an element or compound has a positive standard electrode potential, it means it does not form ions as easily (be reduced and be an oxidizing agent). There is a greater tendency for reduction to occur.

3. Low electrode potential (High dissolution tendency): The more negative the value, the easier it is for that element or compound to form ions (be oxidised and be a reducing agent). There is a greater tendency for oxidation to occur.

4, Electrolyte: A medium that conducts electric current (through the movement of ions) as a result of a dissociation into positively and negatively charged particles called ions. This includes most soluble salts, acids, and bases dissolved in a polar solvent, such as water.

The anode (positive electrode) is in contact with the electrolyte, and corrosion will occur through an oxidation reaction that involves the formation of ions and the release of electrons. The cathode (negative electrode), no corrosion occurs because of a reduction reaction, which is the acceptance of electrons generated from the anode [5].

The conductor connects the anode and cathode to complete the circuit for current flow. In the wet corrosion process, all corrosion cells that are formed contain these four basic parts. Therefore, corrosion occurs from two simultaneous reactions: oxidation and reduction (redox) .

The most used metals naturally exist as compounds of oxides or sulphides, such as Fe3O4, Al2O3, or CuS. These compounds represent the thermodynamically stable form (lowest energy state).

Pure metals spontaneously react with oxygen, sulphur, or chlorine to revert to their lowest energy state, this is actually the process of corrosion. Gold and other noble metals are found in their pure form with lowest energy state, so they do not corrode [2].

Passivity

Passivity is defined as a “corrosion preventative mechanism where an oxidation layer forms a continuous film (passive layer) on a metal's surface that act as a barrier between the metal and the electrolyte, thus prevents further corrosion”. Active metals and alloys, lose their chemical reactivity and become extremely inert, because of the formation of a highly adherent and very thin oxide film on the metal surface, which serves as a protective barrier to further corrosion. It occurs in specific metals, such as Chromium, Aluminum, and Titanium [2, 5].

Re-passivation is repeating the process after a period of time due to the protective coating wearing down. The re-passivation potential is affected by the critical current density, pit current density and the thickness of the oxide film. The pit current density is “the current density below which pits stop growing”. While the critical current density is “the minimum current density needed to maintain the critical pit environment and prevent re-passivation”. The re-passivation potential decreases as thickness of the oxide film increases. Also, the re-passivation potential for a given metal film thickness increases when the electric current drops below the critical value of the pit current density. Similarly, the re-passivation potential will increase when the electric current is higher than the critical current density [30.]

Corrosion pathways

Three different electrolytic corrosive systems may exist in the oral cavity.

- The simplest cell consisted of a pure metal or single phase alloy electrode surrounded by tissue fluid and saliva.
- A cell with two electrodes that are physically separated, yet connected through saliva and tissue fluid may also occur in the mouth.

Both of these cells are unlikely to produce a high electron flow because of high resistance within the electrolyte.

- The electrodes are in direct contact or come into intermittent contact, this eliminates the high resistance of the two electrolyte systems and allows the possibility of a higher electron flow.

This is the electrolytic cell which is most likely to occur within a multiphasic alloy or where different alloys have been used to restore the same tooth. It is also likely to occur when different alloys come into the intermittent contact of mastication, when adjacent teeth restored with different alloys are in intimate contact, and When other alloys such as dental instruments or partial dentures contact a previously restored tooth.

Electrochemical potentials, current pathways, and resistances depend on whether there is no contact, intermittent contact, or continuous contact between alloy restorations [10].

a) Non-contacting Isolated Alloy Restoration

In case of a single metallic restoration, an eclectic current path between external surface exposed to saliva and interior surface exposed to dentinal fluids is formed.

Because the dentinal fluids contain a higher Cl concentration than saliva, so it is assumed the electrode potential of interior surface exposed to dentinal fluids is more active [11].

B) Nonisolated

For two restorations not in contact the extracellular fluid - saliva resistance, determines the extent to which the current will pass through. If the resistance is high, the current will not flow through the both. The interaction between the separated restorations will be minimized. Each restoration, though, will still generate its own current path loop [11].

Restorations Making Intermittent or Continuous Contact

a) Intermittent Contact

This occurs when two alloy restorations (one in the upper arch and the other in the lower arch) come into contact intermittently by biting. When the two restorations are in direct contact, a galvanic cell is generated with an associated galvanic current formed between the two restorations. The external current path can take a number of directions, with the least resistance path controlling. One entirely through extracellular fluids and the other partly through extracellular fluids and partly through saliva [11].

b) Continuous Contact

Another situation in which metallic restorations in the mouth are capable of generating galvanic currents involving two dissimilar metallic restorations in continuous contact, as in the combination of amalgam- gold alloy couples, between two amalgam restorations (one of conventional amalgam and the other a high-copper amalgam), two gold alloys with differences in noble metal content. Soldered appliances are also examples of dissimilar metals making continuous contact. Any multiphase microstructures are situations for galvanic corrosion to occur [11].

Different forms of corrosion

a. Chemical (dry) corrosion

Dry corrosion occurs in the absence of a liquid phase or above the dew point of the environment. In this case, a metal reacts with vapours and gases usually at high temperatures. These reactions can produce a tarnishing attack on exposed metals 15. In wet corrosion, a metal usually reacts with aqueous solutions or electrolytes.

b. **Electrochemical (wet) corrosion**

Wet corrosion causes the greatest amount of deterioration of materials. It requires the presence of water or other aqueous solutions (electrolyte) and a pathway for the transport of electrons [5].

b.1. Galvanic corrosion

This type of corrosion occurs due to the galvanic coupling of dissimilar metals such as metal restorations with different compositions, are placed in adjacent or opposing teeth. The less corrosion resistant metals become anode and corrode. When the two metals brought into contact, there is a sudden short-circuit formed that may cause what is called galvanic shock.

This galvanic current can occur also between restorations of similar alloys (as an old amalgam filling and a freshly placed amalgam restoration), because they will never have the same surface composition or structure. The old amalgam restoration will act as a cathode, and the freshly placed amalgam restoration will act as anode and corrode. This is because the old amalgam restoration has already undergone corrosion and oxidation and reached passivity (build up of oxide layer) which hinders corrosion progression. The high stability of the oxide film formed on the previously corroded old amalgam restoration serves as a further corrosion inhibitor [29].

Galvanic corrosion also occurs within the same filling material because the chlorine ion concentration in the interior surface of a dental restoration is seven times higher than that of saliva, so the inner surface of the restoration will have a higher electrochemical potential [2, 5]. The electrochemical cell is generated due to the difference in the electrode potential created by the two metals and the presence of electrolytes, as saliva and tissue fluids (the dentinal fluid, soft tissue fluid, and blood). Galvanic corrosion can occur in heterogeneous dental alloys' surfaces, having differences in electrode potential such as eutectic alloys, cored structure, impurities at grain boundaries, and solder joints [2, 5].

N.B: For galvanic corrosion to proceed, the production of electrons at anode by oxidation reactions must be exactly balanced by consumption of electrons at cathode. This is a very important in determining the rate of corrosion process. If the consumption of electrons at cathode is higher than production of electrons at anode the higher will be the corrosion rate and vice versa.

- High anode/cathode ratio (favourable) → decreased corrosion rate.

- Low anode/cathode ratio (unfavourable) → increased corrosion rate.

b.2. Crevice corrosion

This type of corrosion occurs when there are variations in the electrolyte composition within the system. Crevice corrosion occurs at the interproximal areas due to food debris and bacteria that alter the electrolyte composition, to be different from normal saliva. Also, Crevice corrosion results from differences in the oxygen tension. Irregularities such as pits, scratches and cracks in restoration are covered by food debris and mucin so the bottom of the defect becomes poor in oxygen concentration and acts as anode [2]. This is because the decrease in the oxygen concentration or hypoxia stimulates and increases the metal degradation or ionization. Also, the low oxygen tension causes reduction in the pH, which in consequence will increase the corrosion speed [31].

Crevices are unavoidable in surgical implants because implants never have simple shapes and are never exposed to a homogeneous environment. The local fluctuation of ions is significantly increased at crevice sites in the screw or plate. In the small space the liquid and oxygen exchanges are very limited and thus, the surface undergoes active corrosion and cause further deterioration of the implant material [2, 5].

b.3. Pitting corrosion

Pitting is a form of extremely localized attack that results in holes on the surface. Pitting corrosion is a type of corrosion that is similar to crevice corrosion, but it can begin on a smooth surface, as a consequence to localized breakdown of the passive layer that cause pitting. This type of corrosion can be initiated due to occlusal wear, acidic environment, or aggressive chemicals such as chloride [2, 6]. The rate of crevice and pitting corrosion is very rapid, because the area of the anode region is significantly smaller than that of the cathode. This unfavourable anode/cathode ratio causes rapid corrosion known as localized corrosion. Consequently, failure occurs much more rapidly than that expected from a uniform surface attack [2].

N.B: Uniform corrosion is defined as "a chemical or electrochemical reaction that proceeds uniformly over the entire exposed surface or over a large area".

b.4. Intergranular corrosion

Also termed intercrystalline or interdendritic corrosion. As the grain boundaries are known with its highly reactive nature, intergranular corrosion occurs adjacent to grain boundaries with little corrosion within the grains. This type of corrosion occurs due to the inhomogeneity. This type of corrosion is common in stainless steels.

It has been confirmed that prolonged heating of 18/8 stainless steel between 400 and 900 ◦C, during heat treatment or Welding, subjects the alloy to intergranular corrosion. This is due to the formation of small precipitate of chromium carbide along the grain boundaries. As a result, a chromium-depleted zone is formed adjacent to the grain boundary, which is known as sensitization/weld decay. So, the grain boundary region is now highly susceptible to corrosion [1, 7].

b.5. Selective leaching

Selective leaching is the removal of one element (the less noble component) from a solid solution alloy by galvanic corrosion process. Examples: leaching of Zinc in brass alloys (dezincification) in which zinc is selectively leached from a copper–zinc brass alloy. This process produces a weakened porous copper structure. Copper or Silver in Gold alloys, and Molybdenum in Nickel- Molybdenum alloys. Elements in an alloy that are more resistant to the environment remain behind.

There are two mechanisms for selective leaching: (1) Two metals in an alloy are dissolved; then one metal redeposits on the surface of the surviving elements. (2) One metal is selectively dissolved, leaving the other metals porous behind. The first system is involved in the dezincification of brasses, and the second system is involved when molybdenum is removed from nickel alloys [1, 2, 5].

b.6. Erosion-corrosion

Erosion corrosion is the acceleration or increase in rate of deterioration of a material due to the combined action of chemical attack and mechanical abrasion or wear. This type of corrosion arises because of the relative movement between a corrosive fluid and the material surface. It includes cavitation damage and fretting corrosion. Cavitation damage is not important in dentistry [5].

Fretting corrosion occurs in metal alloys that form a passive layer. The abrasive or wear action may erode the film, leaving an exposed metal surface. Erosion-corrosion is common in dental titanium implants [12]. During insertion of an implant, some degree of abrasion between fixing screws and implants or between tools and implants occur. Another source of abrasion on implant structures, is functional loading, which induces relative movement between the material (screw hole) and the screw head (i.e., the rubbing movement, disrupts the protective surface films). This action stimulates the chemical activities at the surface, leading to oxidation, active corrosion, or re-passivation [13].

b.7. Stress corrosion

Stress corrosion cracking is "the cracking caused by the simultaneous presence of tensile stress and a specific corrosive medium". Stress can accelerate the corrosion process because the internal energy is increased. Stress can be externally applied as restorations are subjected to heavy compressive, shear and bending forces during mastication. Also, it can be a residual stress as in cold worked structures due to permeant deformation, burnished restorations, or atoms at grain boundaries. Thus, an electrolytic cell is formed between the stressed and unstressed metal portions. For most metallic dental appliances, stress corrosion occurs mostly during fatigue or cyclic loading. Small surface irregularities act as sites of stress concentration (stress raisers), so the effects of fatigue and corrosion are combined [2, 5].

b.8. Hydrogen embrittlement

Also known as hydrogen assisted cracking or hydrogen induced cracking. It is the embrittlement of a metal by hydrogen. The atomic hydrogen is harmful to the toughness of iron and steel. It is a low temperature effect (near ambient temperature) because most metals are immune to hydrogen embrittlement above approximately 150°C. For hydrogen embrittlement to occur, a combination of three conditions are required: (1) the presence and diffusion of hydrogen atoms or ions, (2) a susceptible material and (3) stress 24.

Various metal alloys, as steels, experience a significant reduction in ductility and tensile strength when atomic hydrogen penetrates into the material, and diffuses interstitially through the crystal lattice. When these hydrogen atoms recombine in the voids of the metal matrix to form hydrogen molecules, they create internal pressure leading to crack formation (hydrogen induced cracking) [1, 15].

Copper alloys which contain oxygen can be embrittled if exposed to hydrogen. The hydrogen diffuses through the copper and reacts with inclusions of Cu2O, forming H2O, which then forms bubbles at the grain boundaries. This process can cause the grains to be forced away from each other causing embrittlement. Also alloys of vanadium, nickel, and titanium can absorb significant amounts of hydrogen. This can lead to large volume expansion and damage to the crystal structure leading to the alloys becoming very brittle [16].

N.B: Heating metals above 150°C causes the hydrogen within the metal to diffuse out through heat treatment, that's why hydrogen embrittlement is a low temperature effect.

b.9. Biocorrosion (Microbiologically influenced corrosion)

It is "the deterioration of the metal surface due to the direct or indirect activity of living microorganisms" [17]. This biological corrosion is influenced by the nature of their surrounding environment. An aggressive or electrochemically active environment can greatly increase the rate of corrosion of a metal. Biological corrosion is not considered actually a type of corrosion. But the activity of living organisms (bacteria, algae) can increase the corrosion process by affecting the anodic and cathodic reactions and the protective passive layer. It can also create corrosive conditions by producing deposits. Two types of microorganisms can cause this type of corrosion.

These are aerobic (oxygen using) and anaerobic (nonoxygen using). Anaerobic organisms are most responsible for corrosion of iron and steel [5]. Sulphate-reducing bacteria are the most important anaerobic bacteria that influence the corrosion of steels, Co-Cr-Mo and Ti- 6Al-4V alloys. These bacteria reduce inorganic sulphates to sulphides in the presence of hydrogen, where hydrogen is provided by the cathodic reaction or present in the general environment.

The reaction is according to the following equation:

$$SO4\ {-2} + 8H \xrightarrow{\quad} S\ {-2} + 4H2O$$

Iron then reacts with sulphides according to the following reaction:

$$Fe{+2} + S{-2} \xrightarrow{\quad} 2FeS$$

The presence of iron sulphide in rust is often an indication that corrosion was influenced by sulphate-reducing bacteria [17, 18, 19].

Factors affecting corrosion resistance of metals

Wide variety of factors influence the corrosion behavior of metals, including both the material and the surroundings. Changes in these variables can have an effect on the mode and rate of metal ion release. These factors include:

1. Presence of chloride and other ions

The presence of chloride affects the corrosion of metallic restorations and implants. Chloride ions can easily penetrate the passive film due to their high diffusivity. Leading to its accumulation at the metal/film interface. Chloride ions could further react with the metal substrate at the metal/film interface to form metal chloride. Rupture of the surface film due to volumetric expansion, because the molar volume of metal chloride is larger than that of the metal oxide in the surface film. Thus, pit nucleation occurs.

During the pitting process, the pH value of the electrolyte in the pit tends to decrease due to the formation of H+ ions. Once the pH value within the pits drops below a critical value, the repassivation behaviour of passive film is inhibited, and pits can further develop into stable pitting.

Fluoride ions are also very corrosive as chloride ion, and the corrosion ability of these ions is again enhanced when both are present together in the corrosive medium. Fluoride ions are very aggressive on the protective TiO_2 film formed on Ti and Ti alloys. When a Ti restoration is used, fluoride gels should be avoided because it creates an acidic environment that leads to the degradation of the titanium oxide layer and possibly inhibits osseointegration [20, 21].

2. Surface Topography

Most failures due to corrosion are known to initiate at the surface. The more homogenous is the surface, the higher is the corrosion resistance. Surface roughness offers a decrease in metal resistance to corrosion and increase the number of pits on the surface which act as nucleating sites for corrosion [20].

3. Dissolved oxygen, pH & bacteria

The corrosion behavior of metals is also influenced by oxygen content, pH, and presence of microorganisms. The effect of Oxygen Tension: The deposition of the biofilm leads to reduction in the amount of oxygen reaching the metal surface beneath the biofilm compared to the other areas. Areas of low oxygen tension act as anode, which undergoes corrosion releasing metal ions into the saliva.

These metal ions combine with the end-products of the bacteria, along with chloride ion in the electrolyte (saliva) to form more corrosive products like $MnCl_2$, $FeCl_2$ favoring further corrosion [22]. The PH influences the metal release in different ways, either directly through dissolution or indirectly by changing the extent of adsorbed complex agents or proteins. Generally, the release of metallic ions increases as the pH of the solution decreases. In case of base metal alloys [22]. Bacteria have great effect on the corrosion resistance of dental metallic material. The corrosion behavior of metals in the presence of Streptococcus mutans and its byproducts was found to be increased. This could be explained by the attachment of microbes on implants disturbs the passivity of passive metal. Furthermore, the formation of organic acids by bacteria lead to reduction in pH leading to a favorable environment for corrosion [22].

Factors Affecting rate of corrosion

1. Electrode Potential of individual metal. The more the reactivity of the metal, the higher will be the corrosion.
2. Relative position of anode to cathode in the Electromotive force of series: increasing the difference in electrode potential between anode and cathode will increase the rate of corrosion.
3. Increasing the anode/cathode ratio will decrease the rate of corrosion process as consuming the electrons at the cathode less than producing of electrons from anode
4. Type of electrolyte: normal saliva is weak electrolyte so the rate of corrosion will be lower when compared to rate of corrosion in acidic electrolyte.
5. Concentration of positive ions in the electrolyte. By increasing the ions in the electrolyte, the rate of corrosion is increased.
6. A rise in temperature (within a reasonable limit) increases the rate of corrosion [23].

Adverse effects of Corrosion Products

Most cast dental restorations are made from alloys or commercially pure titanium. It has been documented that metallic dental restorations release metal ions mainly due to corrosion. Those metallic components may be locally and systemically distributed and could play a role in the etiology of oral and systemic pathological conditions. The quality and quantity of the released cations depend upon the type of alloy and various corrosion parameters [24].

1. Allergic Reactions

Local and systemic allergic reactions to many metals have been observed. Of all the metals the most allergens are Ni, Cr, Co and Hg. While as Aluminium and Titanium are said to be rare allergens. These allergens usually cause allergic contact dermatitis which appears as swelling, redness, burning sensation, ulceration, and sometimes reaches necrosis [25].

2. Metallic Taste

The symptom of metallic taste has been reported and related to the release of ions from the metal. Patients with bruxism are likely to be more susceptible to metallic taste [25].

3. Discoloration of teeth

It has occurred mainly with amalgam fillings and metallic screw posts. With amalgams, Grey pigmentation is common in teeth with amalgam restorations. Corrosive amalgam discolors the dentine by formation of silver sulfide. While with the screw posts, copper and zinc were detected in both the dentin and enamel and the surrounding soft connective tissue. It was found also that Mercury, lead, and silver nitrate salts used in dentistry can cause a grey staining in teeth [26, 27].

4. Galvanic pain

It has been reported from the contact of dissimilar-alloy restorations. In this case, instantaneous current flows through the external circuit causing pain [28].

5. Oral lesions

Permanent irritation to the oral mucosa can be generated by the shape and location of the prosthesis in the mouth. Corrosion of a metallic prosthesis can change the smooth surface of the prosthesis to a rough & sharp surface which increases the irritation in the opposing tissues [11].

6. Bone Loss

Ti alloys contain significant amounts of alloying elements that affect osseointegration mainly due to corrosion products containing aluminium and vanadium. It was found that macrophages were present in the peri-implant tissue with the metallic ions. Thus, corrosion process plays an important role in implant failure. Moreover, presence of free titanium ions in the peri-implant tissue inhibits growth of hydroxyapatite crystals. These processes lead to local osteolysis and loss of clinical stability of the implant [21].

7. Metal Fracture

Corrosion can severely limit the fatigue life and ultimate strength of the material leading to mechanical failure and metal fracture [21].

Protection Against Corrosion

1. Increase noble metal content

As noble metals have higher electromotive potential when compared to any other metals used in the oral environment, it can be added to alloys to reduce corrosion. At least half of the atoms in the alloy should be noble metals (gold, platinum, and palladium) to ensure the resistance against corrosion. Palladium is found to be effective in reduction of silver sulfide formation for alloys containing silver [7].

2. In Base metal alloys

Passive metals develop a thin, adherent, highly protective film when they react with the environment. Chromium, titanium and aluminium are examples of such metals. Base metal alloys containing chromium are resistant to corrosion because of the passivity of chromium (not less than 20%) to form adherent oxide layer [7].

3. Polishing of restorations

Polished smooth metallic restorations like amalgam and cast metal minimizes corrosion [7].

4. In presence of two dissimilar metals

Increase anode/cathode ratio to decrease rate of corrosion process, as consumption of electrons at cathode is less than production of electrons at anode. Paint cathode surface by varnish coating [7].

5. Maintenance of oral hygiene

Conclusion

Materials used in the oral cavity are constantly exposed to fluctuating physical and chemical environment that includes temperature and components like saliva, salts, foods, acidic and alkaline liquids, and drugs. Plaques and food adhere to teeth and filling material which means several forms of bacteria and their products are always present inside mouth. Also, fluoride ions interfere in the titanium oxide formation which causes changes in the passive layer formation. This makes the oral environment a highly conductive medium for tarnish & corrosion.

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Failure of metals and alloys in dentistry

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Abstract: Failure of a material is generally to expect the conditions under which a solid material will fail under the action of external loads. The failure of metals is usually classified into ductile failure (yielding) or brittle failure (fracture). Metals can fail in a ductile or brittle manner or both dependent on the conditions such as temperature, type of stress, loading rate. Fracture is the separation of a body into two or more pieces in response to a stress that is static and at temperatures that are low relative to the melting temperature of the materia. In metals, there are two possible fracture modes, ductile and brittle. Fatigue failure occurs in structures subjected to dynamic and fluctuating stresses. Failure occurs at a stress level lower than the yield strength of the specimen. The term fatigue is used because this type of failure normally occurs after a long period of repeated stresses or strain cycling. Fatigue is estimated to be involved in approximately 90% of all metallic failures. Creep is a time-dependent permanent deformation of materials that are used at temperatures that are close to their melting points. creep is normally an undesirable phenomenon. for metals, it becomes important only for temperatures greater than about 0.4 of the melting temperature. In this article different types of metal failure will be discussed showing different mechanics, mechanisms and stages for fracture types, fatigue and creep..

Keywords: Failure; metals; alloys.

Introduction

1) Mechanical failure

Failure of a material is generally to expect the conditions under which a solid material will fail under the action of external loads. The failure of metals is usually classified into ductile failure (yielding) or brittle failure (fracture). Metals can fail in a ductile or brittle manner or both dependent on the conditions (such as temperature, type of stress, loading rate) [1, 2].

a. Fracture

Fracture is "the separation of a body into two or more pieces in response to a stress that is static (constant or slowly changing with time) and at temperatures that are low relative to the melting temperature of the material". In metals, there are two possible fracture modes: (ductile and brittle). According to the ability of a material to experience plastic deformation, ductile metals show considerable plastic deformation with high energy absorption before fracture. While in brittle fracture, there is little or no plastic deformation with low energy absorption [1,2].

Any fracture mechanism involves two steps crack formation and propagation in response to an imposed stress. The mode of fracture is highly dependent on the mechanism of crack propagation.

a.1. Brittle fracture

Brittle fracture mechanics (Stress concentration)

Fracture mechanics "is the field of mechanics concerned with the study of the propagation of cracks in materials". It calculates the driving force on a crack to characterize the material's resistance to fracture [3]. The measured fracture strengths for most materials are lower than those expected by theoretical calculations based on atomic bonding energies. This discrepancy is due to the presence of microscopic cracks or flows (stress raisers) that exist at the surface and in the interior of a material. These flaws affect the fracture strength because an applied stress may be concentrated and amplified at the tip. Stress amplification also may occur at macroscopic internal discontinuities as voids or inclusions, sharp corners, scratches, and notches [2, 3].

According to Griffith energy balance theory, there are two conditions necessary for crack growth: i. The bonds at the crack tip must be stressed to the point of failure. The stress at the crack tip depends on the ratio of its radius of curvature to its length. ii. The amount of strain energy released must be greater than or equal to that required for the surface energy of the two new crack faces [4].

The degree of amplification of stress depends on:

- Crack length
- Radius of curvature of the crack tip.

The maximum tensile stress could be calculated from this equation [2, 4]:

Where, σm = maximum tensile stress.

$\sigma 0$ = magnitude of the applied tensile stress.

ρt = radius of curvature of the crack tip.

a = the length of the surface crack or half of the length of the internal crack.

Therefore, for brittle fracture, cracks propagate very rapid, with very little associated plastic deformation. These cracks are unstable, and the crack continues propagation even without an increase in magnitude of the applied stress [2].

Mechanism of brittle fracture

For the majority of brittle crystalline materials, crack propagation is due to the sequential and repeated breaking of atomic bonds along specific crystallographic planes this process is known as cleavage. Cleavage fracture may be trans-granular or intergranular

i. Trans-granular cleavage [2, 6]:

The cracks pass through the grains, as the cleavage follows a well-defined crystallographic plane.

Microscopically, the fracture surface may have a grainy or faceted appearance due to the changes in orientation from one grain to the other.

ii. Inter-granular cleavage [2, 6, 7]:

In some alloys, crack propagation occurs along the grain boundaries known as intergranular fracture, fig. (3a). Inter-granular fractures usually happen after the occurrence of any process that weaken or embrittle grain boundary regions such as overheating of alloys. This weakening of the grain boundary is due to (1) attraction of impurities to grain boundaries which embrittle them, (2) growth of carbide diameter at grain boundaries or (3) environmentally assisted cracking at the grain boundaries.

Microscopically, scanning electron micrographs show the three-dimensional nature of the grains "rock candy appearance" which indicates inter-granular fractur.

Macroscopic features of brittle fracture surface [2]

Fracture surfaces of brittle materials have flat fractured surface without any signs of plastic deformation (necking) with grainy or faceted shiny texture, fig. (4b). V-shaped "chevron" markings form near the centre of the fracture cross section that indicate the site of crack initiation, fig. (4a).

Brittle fracture surfaces also contain lines or ridges that radiate from the origin of the crack in a fanlike pattern, fig. (5). Usually, these markings are coarse enough to be observed with the naked eye. While, in very hard and fine-grained metals, there is no visible fracture pattern can be observed.

a.2. Ductile fracture

Ductile fracture mechanics (Stress redistribution)

Unlike the behaviour around the crack in a brittle material, stresses are redistributed around the crack tip in ductile materials. Deformation occurs at the sharp crack tip results in blunting into a rounded groove. This occurs by sliding of rows of atoms past one another along the slip plane, where the bonds break one at a time and reform immediately with the adjacent atoms [9].

Therefore, ductile fracture is characterised by large plastic deformation in a propagating crack. Moreover, the procedure proceeds slowly as the crack length increases. Such a crack is often stable as it resists any additional crack propagation unless the applied stress increased [2].

Stages of Ductile fracture

First, after the necking begins, micro-voids are formed in the interior of the cross section. Then, as deformation continues, the micro-voids enlarge and coalesce to form an elliptical crack. The crack continues to grow in a direction perpendicular to the applied stress. Finally, fracture succeeds by the rapid propagation of a crack [2].

Macroscopic features of ductile fracture surface [2, 5]

In ductile materials, there are two macroscopic fracture profiles. First, for extremely soft metals (as pure gold), these highly ductile materials neck down to a point fracture, showing 100% reduction in area,. Second, for most ductile metals, where fracture is preceded by a moderate amount of necking.

Sometimes, ductile fracture has macroscopic features on the fractures surface termed a cup-and-cone fracture, where one of the surfaces is in the form of a cup and the other surface is like a cone.

Also, in ductile fractured surface the central interior region of the surface has fibrous, irregular and dull look, which indicates the occurrence of plastic deformation.

Microscopic features of ductile fracture surface [2, 5]

Microscopic examination, using scanning electron microscope (SEM), reveals more details regarding the ductile fracture mechanism. Studies of this type are termed fractographic studies. The scanning electron microscope is preferred over optical microscope for fractographic assessments due to its better resolution and depth of field.

The examination of the fibrous central region in a cup and cone fracture surface with SEM showed numerous spherical dimples which result from uniaxial tensile failure, fig. (10a). Each dimple is a one half of a micro-void that formed and then separated during the fracture process. Dimples can also be formed by shear loading on the 45°lip of the cup and cone fracture, where these dimples will be elongated or C-shaped (parabolic shape dimples), fig. (10b).

Ductile fracture is preferred to brittle fracture because, brittle fracture occurs suddenly and catastrophically without any warning (due to rapid crack propagation). While, in ductile fracture, the plastic deformation gives warning that the material will fail, so that, preventive measures can be considered. Also, more strain energy is needed to produce ductile fracture (because ductile materials are tougher) [2].

a.3. Ductile to brittle transition in metals

The ductile-brittle transition temperature (DBTT), nil ductility temperature (NDT), or nil ductility transition temperature is "the temperature above which a material is ductile and below which it is brittle". Both ductile and brittle behaviours are dependent not only on the material but also on the temperature (ductile-brittle transition) of the material.

Many steels exhibit ductile fracture at elevated temperatures and brittle fracture at low temperatures. These materials should be used only at temperatures above the transition temperature, to avoid brittle and catastrophic failure [8]. The ductile to brittle transition behaviour is normally found in low carbon steels. Yet, some metals do not go through a ductile to brittle transition, such as low-strength aluminium and copper alloys which remain ductile with decreasing temperature. On the other hand, high-strength steels and titanium alloys maintain a brittle even with increasing the temperature [2]. DBTT is not constant but varies according to previous mechanical and heat treatment, grain size and the nature and amounts of impurities. It can be determined by the amount of impact energy absorption through Charpy or Izod tests [2, 8].

Fatigue failure

1. Dynamic fatigue failure

Fatigue failure occurs in structures subjected to dynamic and fluctuating stresses. Failure occurs at a stress level lower than the yield strength of the specimen. The term fatigue is used because this type of failure normally occurs after a long period of repeated stresses or strain cycling. Fatigue is estimated to be involved in approximately 90% of all metallic failures. Fatigue is catastrophic in nature that occurs without warning. [2]

Fatigue failure process occurs by the initiation and propagation of cracks. The fractured surface is perpendicular to the direction of an applied tensile stress.

The applied stress may be axial (tension–compression), flexural (bending), or torsional (twisting) in nature.

Types of stress–time modes [9]

Reversed stress cycle: the amplitude is symmetrical about a mean zero stress level. Ie., alternating from a maximum tensile stress (s_{max}) to a minimum compressive stress (s_{min}) of equal magnitude.

Repeated stress cycle: the maximum and minimum tensile stresses are asymmetrical relative to the zero-stress level.

Random stress cycle: the stress level varies randomly in amplitude and frequency.

From the stress time cure we can determine different parameters that are used to characterize the stress cycle:

- Stress range: the difference between the max. stress and the min. stress.
- Stress amplitude: half the range of stress.
- Stress ratio: the ratio of minimum and maximum stress amplitudes.
- Mean stress: (min stress + max stress)/2

The S–N curve

The fatigue properties of a material could be determined by laboratory simulation tests. In which the specimen is subjected to cyclic stresses of relatively high max. stress (2/3 tensile strength) & number of cycles till failure is counted. Repeating this procedure on different specimens with decreasing the max stress level progressively. Data are plotted as stress (max stress or stress amplitude) versus logarithm of the number of cycles. [2]

Three parameters could be determined from this curve; fatigue limit, fatigue strength and fatigue life. Where, fatigue limit (endurance limit) is the limit below which fatigue failure will not occur. Some ferrous and titanium alloys show fatigue limit. For many steels, fatigue limits range between 35% and 60% of the tensile strength. While, most nonferrous alloys as aluminum & copper do not have a fatigue limit, so the S–N curve continues its downward trend with increasing N values. Thus, fatigue ultimately occurs regardless of the magnitude of the stress is. For these materials, the fatigue response is specified as fatigue strength. [2]

Fatigue strength is the stress level at which failure will occur for some specified number of cycles.

Another important parameter that is used to characterize fatigue behaviour is fatigue life (*Nf*). It is the number of cycles that causes failure at a specified stress level. Curves for the titanium, magnesium, steel alloys and cast iron display fatigue limits. While curves for the brass and aluminum alloys do not have such limits.

Low-cycle fatigue: is associated with relatively high loads that produce not only elastic strain but also some plastic strain during each cycle. Consequently, fatigue lives are relatively short, and occurs at less than about 104 to 105 cycles.

High-cycle fatigue: for lower stress levels wherein deformations are totally elastic, longer lives result. High-cycle fatigue is required large numbers of cycles to produce fatigue failure, fatigue lives are greater than about 104 to 105 cycles.

Stages of fatigue failure

The process of fatigue failure is characterized by three definite steps; crack initiation, in which a small crack forms at point of high stress concentration, followed by crack propagation, during which this crack advances incrementally with each stress cycle; and then final failure, which occurs very rapidly once the advancing crack has reached a critical size and the remaining material can no longer withstand the applied forces. [2]

First stage: Crack initiation

Crack nucleation sites include pre-existing surface scratches, sharp corners, and indentations. Or cyclic loading can produce microscopic surface discontinuities. Initiation of a new crack in smooth polished metals under cyclic load is caused by irreversible dislocation movement leading to intrusions and extrusions. [9]

Second stage: Crack propagation

The mechanism of crack propagation is explained by Plastic blunting and reshaping model: [9]

When reversed stress cycles are applied to a material with a present crack. When tensile loads are applied. At the crack tip, stress is concentrated in the slip zones along the plane of maximum shear stresses (45° to the crack plane) leading to plastic deformation at the tip. When load increases the slip zones at the tip broadens and tip blunting occurs.

When stress is reversed from tension to compression the crack tip sharpens once more by buckling and folding of the newly formed surface into a double notch resulting in striation formation.

Cause of striation formation: when crack closure occurs during compression, it cannot fully wipe out the blunting and the extension of the crack that happened during the preceding tension load, thus, net crack growth occurs during a fatigue cycle, leading to the formation of a striation. Where the distance between two striations is equal to the crack length increment.

Third stage: Final failure

When the crack size increases reaching a critical size, the remaining intact material can no longer withstand the maximum applied stress in a loading cycle. Unstable crack growth occurs leading to fracture.

R curve: describes how the material's resistance to crack propagation increases as the crack propagates. In ductile materials this occurs because plastic deformation dissipates energy which works to arrest a growing crack.[9]

Hysteresis Energy: during cyclic loading part of the applied energy is dissipated in the form of plastic deformation (slip at the crack tip) leading to an increase in the resistance to crack growth or what is called cyclic hardening. [9]

Fractography of fatigue surface

The region of a fracture surface that formed during the crack propagation step may be characterized by two types of markings termed beachmarks (clamshell) and striations. Both features indicate the position of the crack tip at some point in time and appear as concentric ridges that expand away from the crack initiation site(s), frequently in a circular or semi-circular pattern. Beachmarks are of macroscopic dimensions and may be observed with the unaided eye.

These markings are found for components that experienced interruptions during the crack propagation stage. Each beachmark band represents a period of time over which crack growth occurred. However, fatigue striations are microscopic in size and subject to observation with the electron microscope (either TEM or SEM). Each striation is thought to represent the advance distance of a crack during a single load cycle. Striation width depends on, and increases with, increasing stress range.

Factors affecting fatigue life

Mean stress level: By increasing the mean stress level, a decrease in fatigue life occurs.

Geometric design: It has a significant influence on fatigue behavior. Any notch or geometrical discontinuity can act as a stress raiser and initiation site. The sharper the discontinuity (i.e., the smaller the radius of curvature), the more severe the stress concentration is. The probability of fatigue failure may be reduced by avoiding these structural irregularities or by making design modifications by which sharp corners are eliminated.

Surface treatments: during machining operations, small scratches and grooves are inevitably introduced into the surface. This can limit the fatigue life. It has been observed that improving the surface finish by polishing enhances fatigue life significantly.

One of the most effective methods of increasing fatigue performance is by imposing residual compressive stresses within a thin outer surface layer. Thus, a surface tensile stress of external origin is partially nullified and reduced in magnitude by the residual compressive stress. Therefore, fatigue failure is reduced.

In shot peening residual compressive stresses are introduced into ductile metals mechanically by localized plastic deformation within the outer surface region. Small, hard particles (shot) having diameters within the range of 0.1 to 1.0 mm are projected at high velocities onto the surface to be treated. The resulting deformation induces compressive stresses to a depth of between one-quarter and one-half of the shot diameter. This results in an improvement in the fatigue properties.

In case hardening a technique by which both surface hardness and fatigue life are enhanced for steel alloys. This is accomplished by a carburizing or nitriding process. Steel alloy is exposed to a carbonaceous or nitrogenous atmosphere at an elevated temperature. A carbon or nitrogen rich outer surface layer (case) is formed by atomic diffusion from the gaseous phase. The improvement of fatigue properties results from increased hardness within the case and the desired residual compressive stresses which were induced by curburizing or nitriding process.[2]

Environmental factors may also affect the fatigue behavior of materials. as thermal fatigue and corrosion fatigue.

Thermal fatigue[2,14] is normally induced by fluctuating thermal stresses and mechanical stresses from an external source that should not be present. The origin of these thermal stresses is the restraint to the dimensional expansion / contraction that would normally occur in the material with variations in temperature. As a consequence of the thermally induced stresses and strains, fatigue may eventually occur. Moreover, Chemical effects of temperature as oxidation must also be considered.

The magnitude of a thermal stresses developed by a temperature change depends on the coefficient of thermal expansion and the modulus of elasticity. Which can be presented by the following equation.

Corrosion fatigue is type of failure that occurs by the synchronized action of cyclic stresses and chemical attack. Corrosive environments have a destructive influence and produce shorter fatigue lives. Even normal ambient atmosphere affects the fatigue behavior of some materials. Small pits may form as a result of chemical reactions between the environment and the material, which may serve as points of stress concentration and therefore act as crack nucleation sites. In addition, the crack propagation rate is enhanced as a result of the corrosive environment.

Several approaches to prevent corrosion fatigue exist. As, protective surface coatings application, selection of a more corrosion resistant material, reduction of the corrosiveness of the environment, reduction of the applied tensile stress level and impose residual compressive stresses on the surface of the member. [14]

Microstructural variables affect fatigue life greatly. It was found that the size of the grain affects the critical threshold stress that is required for crack propagation. By decreasing the size of the grain, grain boundaries increase which provide the extended topological obstacles to the slip movement. Moreover, the presence of impurities within the metal increases the obstacles to slip movement and raised the critical threshold stress that is required for crack propagation, thus increasing the number of cycles required to reach the crack critical size and increases the fatigue life.[15]

2. Static fatigue

3. Creep

Creep is a time-dependent permanent deformation of materials that are used at temperatures that are close to their melting points. creep is normally an undesirable phenomenon. for metals, it becomes important only for temperatures greater than about 0.4 of the melting temperature. [10]

Testing of creep is done by subjecting a specimen to a constant load while maintaining the temperature constant. Then deformation is measured and plotted versus time. In typical creep behavior of metals, upon application of the load, there is an instantaneous deformation, that is totally elastic. The resulting creep curve consists of three regions, each of which has its own distinctive strain–time feature.

Primary or transient creep, characterized by a continuously decreasing creep rate, the slope of the curve decreases with time. This suggests that the material is experiencing an increase in creep resistance or strain hardening thus deformation becomes more difficult as the material is strained.

Secondary creep, steady-state creep, characterized by constant creep rate thus the plot becomes linear. This is the stage of creep that has the longest duration. The constancy of creep rate is explained on the basis of a balance between the competing processes of strain hardening and recovery. Where, recovery is the process by which a material becomes softer and retains its ability to experience deformation.

Tertiary creep, there is an acceleration of the creep rate and ultimate failure. This failure is frequently termed *rupture* and results from microstructural changes as grain boundary separation, and the formation of internal cracks, cavities, and voids.

The most important parameter from a creep test is steady-state creep rate stage. Through this stage we can determine whether this material is suitable for long or short life applications. In case of short time applications time to rupture, or the rupture lifetime is the main design consideration. For its determination, creep tests must be conducted to the point of failure; these are termed creep rupture tests.

Factors affecting Creep of Metals

Temperature & stress both influence the creep of metals, effect of creep becomes noticeable when temperature is nearly 0.4 of the melting temperature. With either increasing stress or temperature, the following will be noted:

The instantaneous strain at the time of stress application increases
The steady-state creep rate increases
The rupture lifetime decreases.

In contrast to the influence of grain size on the mechanical behavior at low temperatures as it increases strength and toughness by hindering the dislocation movement. At higher temperature smaller grains permit more grain boundary sliding and diffusional creep, resulting in higher creep rates.

Mechanism of creep in metals

Depending on the temperature range and the stress level, different mechanisms are involved in the creep process. Diffusion, dislocation climb or glide, or grain boundary sliding can contribute to the creep of metals.

Diffusional creep [11]

Diffusion mechanism includes passive movement of atoms from high concentration to low concentration till equilibrium. Diffusional creep occurs at a relatively higher temperature and lower stresses compared to dislocation creep.

Nabarro and Herring mechanism

When a low tensile stress is applied on metal at high temperature, vacancies diffuse from grain boundaries that are under tensile stress towards grain boundaries under compressive stress. Diffusion passes through the grain itself. The atoms have slower jump frequencies but more pathways. The atomic diffusion leads to elongation of each grain in the tensile direction.

Coble mechanism

Based on diffusion of atoms in the grain boundaries instead of in the bulk.

In Coble creep the atoms diffuse along grain boundaries to elongate the grains along the stress axis. This causes Coble creep to have stronger grain size dependence than Nabarro–Herring creep, thus, Coble creep will be more important in materials composed of very fine grains.[19]

Generally, the diffusional creep rate should be the sum of Nabarro–Herring creep rate and Coble creep rate. Diffusional creep leads to grain-boundary separation, by formation of voids or cracks between the grains. To heal this, grain-boundary sliding occurs. The diffusional creep rate and the grain boundary sliding rate must be balanced if there are no voids or cracks remain. When grain-boundary sliding couldn't accommodate the incompatibility, grain-boundary voids are generated, which is related to the initiation of creep fracture.[19]

The strong sensitivity of Coble creep and Nabarro–Herring creep to grain size suggests that larger grain sizes are needed to improve the resistance to diffusional creep.

Dislocation creep[12]

Dislocation creep occurs at lower temperature and higher stresses when compared to diffusional creep.

Dislocation slip

Dislocation slip occurs along slip lines and planes at lower temperature when compared to diffusional mechanism. This is the same deformation mode as in conventional deformation at ambient temperature, and it does not depend on diffusion.

Dislocation Climb

When the applied stress is not enough for a moving dislocation to overcome the obstacle on its way via dislocation slip alone, the dislocation could climb to a parallel slip plane by diffusional processes, and the dislocation can glide on the new plane. This process repeats itself each time when the dislocation encounters an obstacle.

In which atoms move out of the slip plane. Higher temperatures than that required for dislocation slip and lower stress permit dislocations in a metallic material to climb. The dislocation climb occurs to bypass obstacle present in the same plane. These obstacles could be created by the pileup effect of multiple dislocations that increases the resistance to dislocation movement. Thus, further dislocations cant occur within this plane. Incase of elevated temperatures the increased energy permits dislocations to climb to a different plane. [18]

3. Grain-Boundary Sliding [16]

It is important to discuss the importance of grain boundary sliding phenomena. These are needed to prevent micro-void or microcrack formation due to the mass transfer associated with grain boundary or bulk diffusion. Hence, the diffusion creep rates must be balanced exactly by grain boundary sliding rates to avoid the opening up cracks or voids.

Note that the grain boundary sliding heals the crack/voids that would otherwise open up due to grain boundary diffusion. The sliding of the grain boundaries (due to an applied shear stress) must be coupled with diffusional accommodation to avoid opening up of cracks or microvoids. Diffusional creep and grain boundary sliding are, therefore, sequential processes. As with most sequential creep processes, the slower of the two processes will control the creep rate. However, large amounts of such sliding may lead, ultimately, to microvoid nucleation and creep rupture in the tertiary creep regime.

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