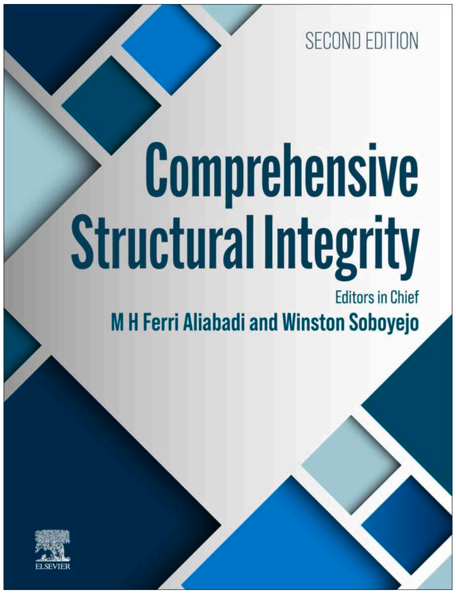


Du, Jing, Niu, Xinrui, Rahbar, Nima and Soboyejo, Wole (2023) Natural Teeth and Bio-Inspired Dental Materials. In: Aliabadi, Ferri M H and Soboyejo, Winston (eds.) *Comprehensive Structural Integrity, 2nd Edition,* vol. 9, pp. 66–89. Oxford: Elsevier.

http://dx.doi.org/10.1016/B978-0-12-822944-6.00093-1

Natural Teeth and Bio-Inspired Dental Materials

Jing Du, Department of Mechanical Engineering, Pennsylvania State University, State College, PA, United States
Xinrui Niu, Department of Mechanical and Biomedical Engineering, City University of Hong Kong, Kowloon, Hong Kong, China
Nima Rahbar, Department of Civil, Environmental, & Architectural Engineering, Worcester Polytechnic Institute, Worcester, MA, United States
Wole Soboyejo, Department of Mechanical and Materials Engineering, Worcester Polytechnic Institute, Worcester, MA, United States

Abstract

Dental restorations, including crowns, bridges, and implants, are designed to repair and restore damaged or missing teeth. All-ceramic restorations are widely used because of their biocompatibility and esthetics. However, their strength and fatigue life are not satisfactory and clinical complications including ceramic fracture and loss of retention are commonly observed. There are several choices of engineered materials for dental restorations. Ceramics are light and hard, but also brittle and fragile. Multilayered dental crown structures are subjected to contact loading due to occlusion and endure sub-surface stresses in the ceramic that can ultimately lead to sub-critical cracking in the ceramic and final failure of the whole multilayered structures. Such cracking is often aggravated by the mismatch and abrupt changes in the elastic properties from one layer to the other in the multilayers.Compared with manmade dental restorations, natural teeth exhibit superior mechanical resilience, including strength and durability, which can be attributed to the interpenetrating and graded microstructures and mechanical properties of the dental hard tissues and their interfaces, dentin-enamel junctions. The sophistication in the natural teeth provides the inspiration for the design and processing of layered structures with gradients. An adhesive layer that consists of functionally graded materials was designed and fabricated to mimic the dentin-enamel junction. It reduces the overall stress concentration in the dental multilayer structures and the stress level in the top ceramic layers and increases the critical loads of failure for the whole multilayered structures. Future work is needed to translate the bioinspired functionally graded multilayers to clinical applications and extend the concept to other dental restorations, such as implants and tissue-engineered structures.

Key Points

- Provide an overview of the microstructure and mechanical properties of dental hard tissues.
- Provide an overview of common dental materials and their mechanical properties.
- Provide an overview of the bio-inspired design of dental multilayer structures.

 doi:10.1016/B978-0-12-822944-6.00093-1

Introduction

Oral health is of great importance to people's general health and well-being. According to the World Health Organization (WHO), dental diseases are among the most common chronic diseases worldwide. For example, caries, which may lead to partial or total loss of teeth, influence almost 100% of adults and 60–90% of school children (World Health Organization, 2012).

Dental crowns have been commonly adopted as a treatment to repair the damaged tooth and recover dental function. According to the American Academy of Implant Dentistry (AAID), 5 million Americans have crown or bridge replacements for missing teeth (American Academy of Implant (AAID), 2016). The global market for prosthetic supplies is predicted to reach $4 billion by 2018 (American Academy of Implant (AAID), 2016). Ceramics restorations are widely used in dentistry and orthopedics because of their biocompatibility and esthetics. However, ceramic dental restorations have some commonly observed clinical complications, including ceramic fracture and loss of retention (Kelly, 1997; Zarone *et al.*, 2011; Le *et al.*, 2015).

There are several reasons why the strength and fatigue live of ceramic dental crowns are not satisfactory. Among the limited choice of engineered materials, none of the single materials can satisfy all of the requirements for dental crowns, which include many engineering aspects such as strength, longevity, biocompatibility, and esthetics. Metals are durable, but heavy, not biocompatible enough, and cause esthetic concerns. Ceramics are light, comfortable, and biocompatible, but fragile. Since no single class of materials can meet all of the requirements, multilayered architectures are often used to achieve the balance of properties that are needed for dental applications. Layered dental crown structures that are subjected to contact loading due to occlusal activity endure sub-surface stresses in the ceramic that can ultimately lead to sub-critical cracking in the ceramic and final failure of the multilayered structures. Such cracking is often aggravated by the abrupt changes in the elastic properties from one layer to the other in the multilayers.

In contrast to conventional dental restorations, including dental crowns, bridges, and implants, natural teeth exhibit superior mechanical resilience, including strength and durability. The mechanical properties of the dental hard tissues, such as their Young's moduli and hardness values, are not necessarily greater than those of the synthetic materials that are used in dental crowns. However, nature has constructed the natural teeth with astonishing sophistication in the underlying microstructures, which are interpenetrating and graded. It contributed to the superior overall performance of natural teeth (Francis *et al.*, 1995).

The microstructure and mechanical properties of natural teeth provide the inspiration for the design and processing of layered structures with gradients. Inspired by the dentin-enamel junction (DEJ), an adhesive layer that consists of functionally graded materials (FGM) was designed and fabricated. It reduces the overall stress concentration in the dental multilayer structures and the stress in the top ceramic layers and increases the critical loads of failure for the whole multilayer structures. This will be explored in depth in this article, including a combination of experiments and models.

This article is divided into 5 sections. Following the introduction in Section "Introduction", the composition, microstructure, and mechanical properties of hard tissues (enamel and dentin) in natural human teeth as well as their interface, DEJ, will be reviewed in Section "Resilience of Human Teeth". Some commonly used dental materials and their mechanical properties and applications in dentistry will be presented in Section "Dental Materials and Their Integrity", including some models that are used in the design of dental crowns. This is followed by Section "Bio-inspired Design of Dental Materials" in which a review of prior works on the bio-inspired functional graded dental multilayer structures is provided. Future directions are elucidated in Section "Future Directions", before summarizing the insights from this article in Section "Summary".

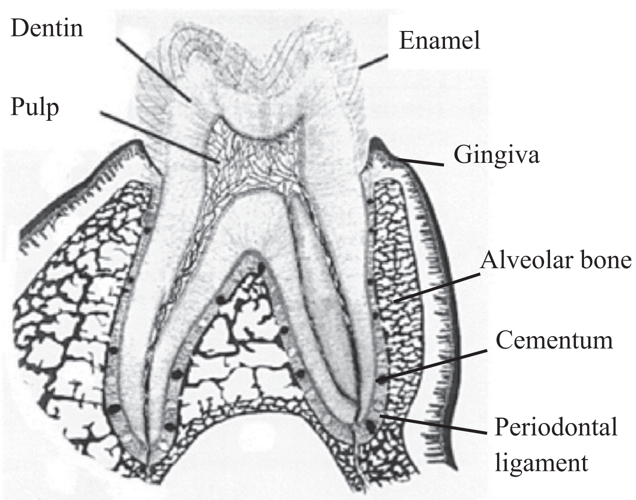


Fig. 1 Schematic of a tooth and periodontium. Adapted from Shrotriya, P., Wang, R., Katsube, N., Seghi, R., Soboyejo, W.O., 2003. Contact damage in model dental multilayers: An investigation of the influence of indenter size. Journal of Materials Science: Materials in Medicine 14 (1), 17–26. Available at: http://www.ncbi.nlm.nih.gov/pubmed/15348534.

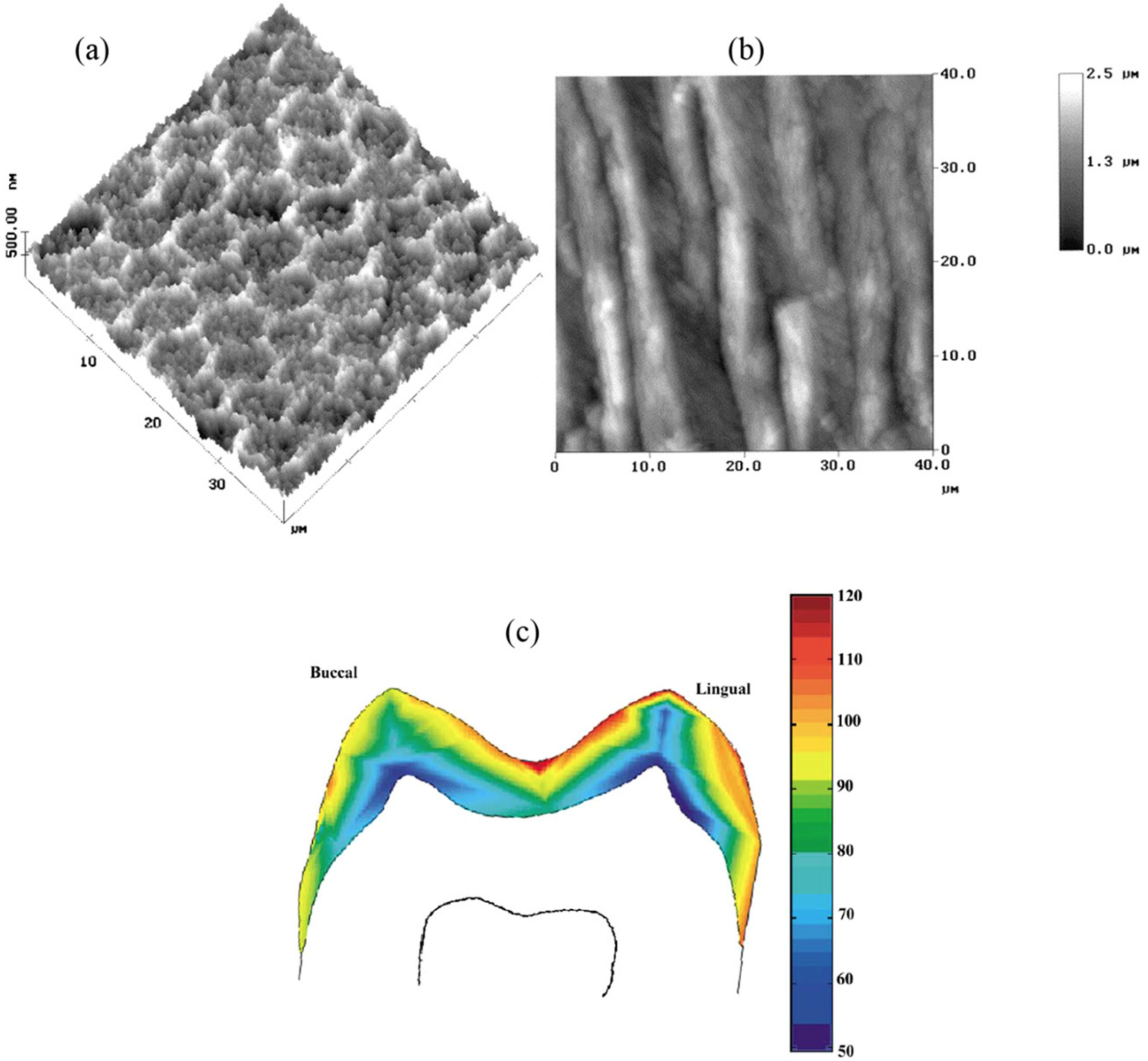


Fig. 2 Atomic force microscope images of enamel rods obtained on (a) an occlusal section and (b) a longitudinal section (c) Young's modulus distribution in enamel measured using nanoindentation technique (unit: GPa). Reproduced from Habelitz, S., Marshall, S., Marshall, G., Balooch, M., 2001. Mechanical properties of human dental enamel on the nanometre scale. Archives of Oral Biology 46 (2), 173–183. Available at: https://doi.org/10.1016/S0003-9969(00)00089-3. Cuy, J.L., Mann, A.B., Livi, K.J., Teaford, M.F., Weihs, T.P., 2002. Nanoindentation mapping of the mechanical properties of human molar tooth enamel. Archives of Oral Biology 47 (4), 281–291. Available at: https://doi.org/10.1016/S0003-9969(02)00006-7.

Resilience of Human Teeth

Form and function are important considerations in restorative dentistry. Restorative dental materials are designed to repair and restore damaged or missing teeth and their supporting tissues so that they mimic the appearance and performance of natural teeth. The natural tooth has superior overall properties to artificial crowns. It is a remarkable example of nature's design of a complex and functional composite structure (Francis *et al.*, 1995). Understanding the microstructures and properties of each tissue in teeth and their surroundings can help us understand how they interact and coordinate to achieve their functions, including mastication, speech production, and giving shape to the human face. Also, the knowledge of the structure and properties of human teeth is necessary for the evaluation of the properties and performance of restorative dental materials and is helpful for the design of artificial materials for dental restoration.

As shown in **Fig. 1**, a human tooth consists of enamel, dentin, and pulp (Shrotriya *et al.*, 2003). Dentin and enamel are both hard tissues that consist of inorganic minerals and organic materials. Enamel is the most highly calcified tissue inside the human body. Its organic content is the least among all tissues. Enamel forms the hard outer shell of the crowns and enables effective mystification. Beneath the enamel, dentin is the main component of teeth. It is integrated with enamel through DEJ. In the center

Table 1 Physical and mechanical properties of the oral hard tissues of human teeth

Property	*Dentin*	*Enamel*
Density ($g \cdot cm^{-3}$)	2.1	2.96
Compressive elastic modulus (GPa)	18–24	60–120
Compressive strength (MPa)	230–370	94–450
Tensile elastic modulus (GPa)	11–19	
Tensile strength (MPa)	30–65	8–35
Shear strength (MPa)	138	90
Flexural strength (MPa)	245–280	60–90
Hardness (GPa)	0.13–0.51	3–6
Fracture toughness ($MPa \cdot m^{1/2}$)	3.08	0.8

Note: Reproduced from Sakaguchi, R.L., Powers, J.M., 2012. Craig's Restorative Dental Materials. Elsevier. Available at: https://doi.org/10.1038/sj.bdj.2012.659. El Mowafy, O.M., Watts, D.C., 1986. Fracture toughness of human dentin. Journal of Dental Research 65 (5), 677–681. Available at: https://doi.org/10.1177/00220345860650050901.

of the tooth, the pulp is an organic tissue that includes soft connective tissue, blood vessels, and nerves. It provides nutrients for the growth and survival of teeth. The root of the tooth is covered by cementum and connected to alveolar bone through the periodontal ligament (PDL). The periodontium, including cementum, periodontal ligament, alveolar bone, and the gingiva, provides support to the teeth.

Enamel

Enamel is the most highly mineralized tissue inside the human body. Mature enamel is a complex inorganic-organic composite (Ten Cate, 1980; Miles, 1967). By weight, it contains about 96% mineral, 1% fat and protein, and water. By volume, the organic component is about 3%, and water is about 12% (Sakaguchi and Powers, 2012). The organic component is mainly non-collagenous glycoproteins and the inorganic component (mineral phase) is mainly hydroxyapatite (HAP) (Ten Cate, 1980). The HAP crystals have a 40-nm wide hexagonal cross-section and may span across the whole length of the enamel, which has a thickness of several thousands of nanometers. Hence, the aspect ratio of the HAP crystals may be very high, which is uncommonly seen in manmade materials (Sakaguchi and Powers, 2012). These crystals group into enamel prisms or rods with 5-μm cross-section **Fig. 2**(a) and (b) (Habelitz *et al.*, 2001). The prisms or rods then form a closely packed array and extend from the outer enamel surface downward to the DEJ.

Some of the physical and mechanical properties of enamel are listed in **Table 1** (Sakaguchi and Powers, 2012; El Mowafy and Watts, 1986). Enamel serves as a hard and wear-resistant surface for the teeth. It frequently goes through demineralization and remineralization cycles. Therefore, the mechanical properties of enamel are not spatially uniform. It is harder at the occlusal area and cusps and softer near the DEJ. The spatial distribution of Young's modulus of enamel was mapped by nanoindentation and shown in **Fig. 2**(c) (Cuy *et al.*, 2002). As measured by the indentation methods, the hardness, elastic modulus, and apparent fracture toughness are all dependent on the distance from the DEJ. As listed in **Table 2**, the results showed that the mechanical properties increased with distance from the DEJ (Park *et al.*, 2008). The hardness of enamel is about 5 times higher than that of dentin. However, the enamel is a brittle material. It fractures along the weak paths parallel to the enamel rods (Rasmussen *et al.*, 1976). The complex microstructure of DEJ prevents cracks from propagating into the underlying dentin.

Dentin

Dentin is a complex connective tissue. Understanding the microstructures and properties of dentin can help us design long-lasting dental adhesive materials. The majority of the dentin is made up of a highly cross-linked collagen phase mineralized with HAP crystallites (Ten Cate, 1980). By volume, dentin consists of ~ 50% inorganic HAP crystals, 23% organic material (mostly collagen fibers), and 27% water (Ten Cate, 1980; Miles, 1967; Linde, 1984). The dentinal tubules are the unique structure for dentin. The structure of dentinal tubules can be observed from two directions in **Fig. 3** (Marshall, 1993). The upper part of the Scanning Electron Microscope (SEM) image shows the dentinal tubules surrounded by peritubular dentin and separated by intertubular dentin. Peritubular dentin is highly mineralized and barely contains organic components. Intertubular dentin is a composite of highly cross-linked type I collagen and HAP. The lower part of the SEM image shows the tunnels that contain fluid and odontoblast processes. They extend from the pulp toward the DEJ.

Some of the physical and mechanical properties of dentin are listed in **Table 1** (Sakaguchi and Powers, 2012; El Mowafy and Watts, 1986). Dentin provides mechanically tough support for the enamel. The cross-linked and mineralized collagen resists deformation in the structure. The highly mineralized walls of dentinal tubules and their short lateral extensions also contribute to the stiffening of the structure. Similar to the gradient of Young's modulus in enamel, the spatial distribution of the mechanical properties in dentin is not uniform, either. It is stiffer near the enamel and softer near the root of the tooth. Though the stiffness of dentin is lower than that of enamel, the fracture toughness of dentin is higher than that of enamel. Dentin is toughened by its composite microstructure, which contains dispersed inorganic particles in a collagen matrix. Multiple interfaces between the

Table 2 Mechanical properties of the human enamel obtained from the indentation analyses

Material	HV_c *(GPa)*	E *(GPa)*
Human enamel (young)		
Inner	3.1	75
Middle	3.5	82
Outer	4.1	87
Human enamel (old)		
Inner	3.0	79
Middle	3.4	90
Outer	4.0	100

Note: Reproduced from Park, S., Quinn, J.B., Romberg, E., Arola, D., 2008. On the brittleness of enamel and selected dental materials. Dental Materials : Official Publication of the Academy of Dental Materials 24 (11), 1477–1485. Available at: https://doi.org/10.1016/j.dental.2008.03.007.

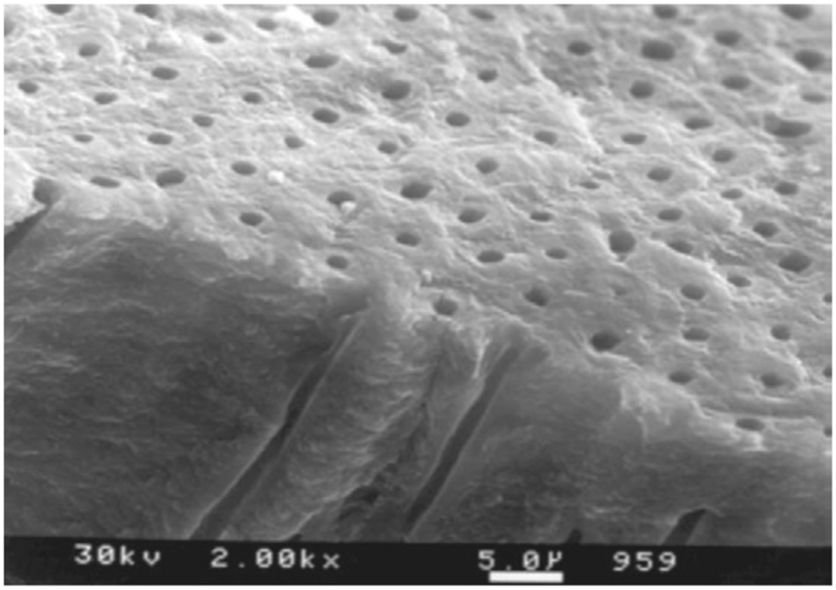


Fig. 3 Scanning electron microscope (SEM) image of dentin. Reproduced from Sakaguchi, R.L., Powers, J.M., 2012. Craig's Restorative Dental Materials. Elsevier. Available at: https://doi.org/10.1038/sj.bdj.2012.659.

organic and inorganic phases interfere with crack propagation and enhance its fracture toughness. An investigation of the fracture properties of dentin revealed that dentin is tougher along its thickness direction than it is laterally (El Mowafy and Watts, 1986).

Moreover, dentin is viscoelastic, which means the mechanical deformation of dentin is time-dependent and will not recover instantaneously after unloading. The deformation of dentin depends on the loading rate. This property can be attributed to the collagen matrix in dentin, which is a polymeric material. Usually, enamel and ceramic materials do not have viscoelastic behaviors (Sakaguchi and Powers, 2012).

Dentin-Enamel Junction

The interface between dentin and enamel is known as the dentin-enamel junction. As listed in **Table 1**, dentin and enamel have very different physical and mechanical properties (Sakaguchi and Powers, 2012; El Mowafy and Watts, 1986). Enamel is hard and brittle. Dentin is softer but tougher than enamel. They need to be integrated to be a biologically and mechanically compatible system. The integration of dissimilar materials is a difficult task. Yet, enamel and dentin integrate and function together harmoniously through their complex interface, DEJ (Francis *et al.*, 1995).

The microstructure of the DEJ is shown in the scanning electron microscopy images in **Fig. 4**(a) (Huang *et al.*, 2007b). It is distinct from that of either the enamel or dentin **Fig. 4**(a). The components from enamel and dentin interpenetrate through and intermingle within this interface (Rasmussen *et al.*, 1976; Lin *et al.*, 1993; Lin and Douglas, 1994). As shown in the schematic in **Fig. 4**(b), type I collagen fibrils in dentin approach each other in the DEJ and merge into coarse bundles before or after entering the enamel matrix (Lin *et al.*, 1993). The common mineral phase in enamel and dentin, HAP, is continuous across the junction. The DEJ interface is not smooth, but instead is a series of linked semi-circles, or scallops. This structure can increase the contact area,

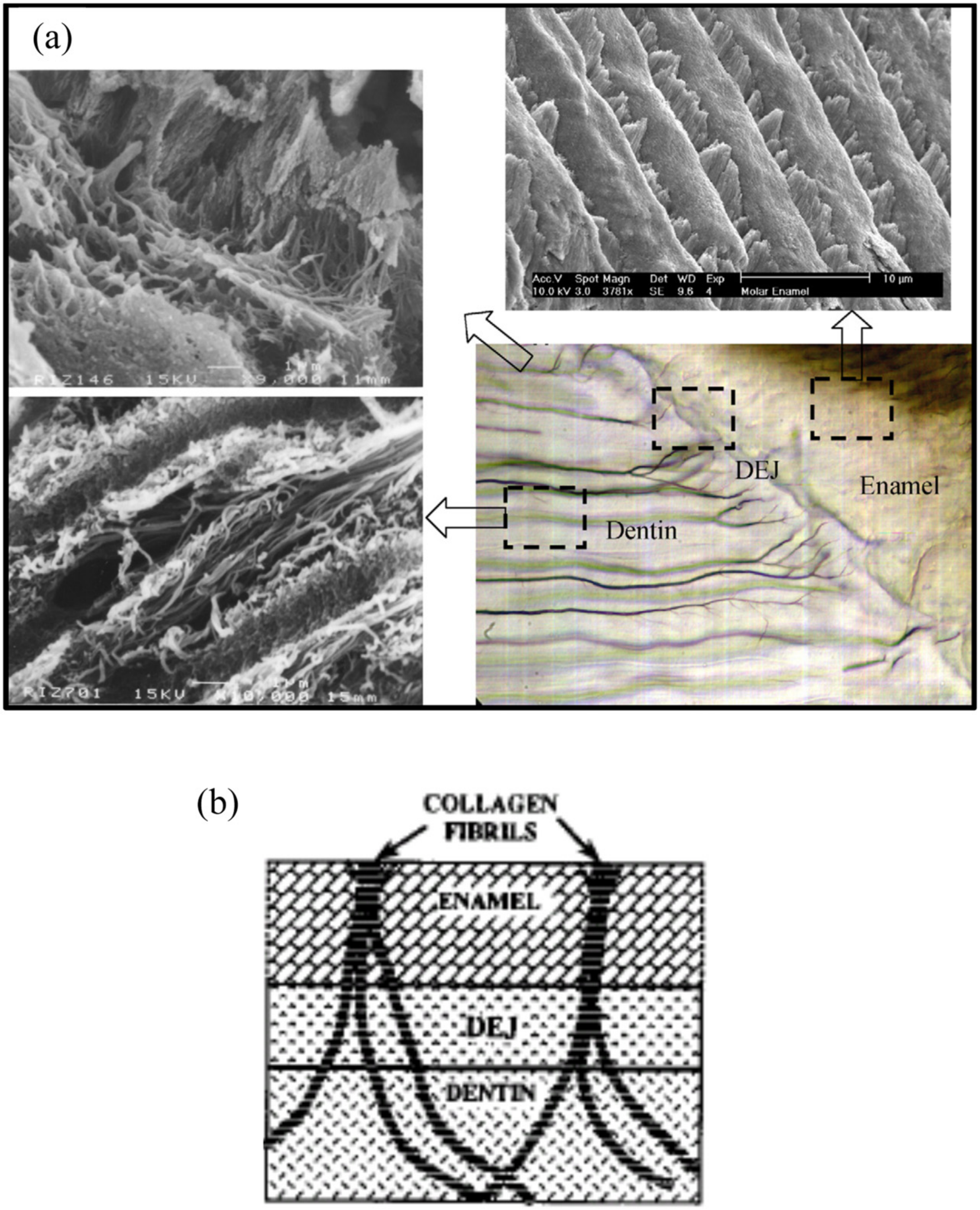


Fig. 4 (a) Microstructure of Dentin-Enamal-Junction (DEJ) under SEM (b) Schematic of the collagen bundles within the DEJ. Reproduced from Huang, M., Rahbar, N., Wang, R., *et al*., 2007b. Bioinspired design of dental multilayers. Journal of Materials Science: Materials in Medicine 18 (1), 57–64. Available at: https://doi.org/10.1007/s10856-006-0662-0. Lin, C.P., Douglas, W.H., Erlandsen, S.L., 1993. Scanning electron microscopy of type I collagen at the dentin-enamel junction of human teeth. Journal of Histochemistry & Cytochemistry 41 (3), 381–388. Available at: https://doi.org/10.1177/41.3.8429200.

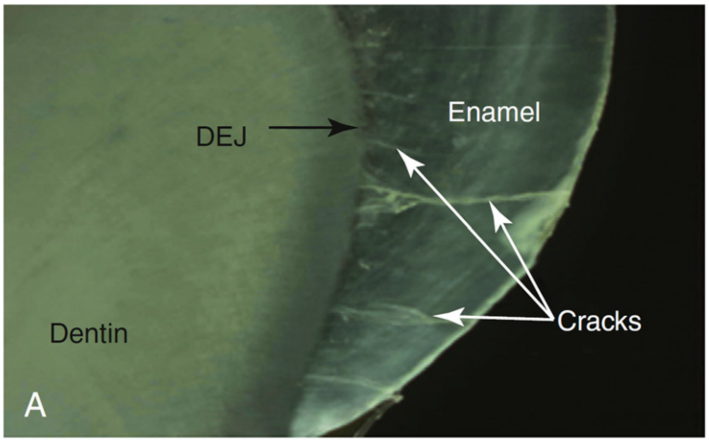


Fig. 5 An optical image of cracks in enamel. Reproduced from Marshall, S.J., Balooch, M., Habelitz, S., *et al*., 2003. The dentin–enamel junction – A natural, multilevel interface Journal of the European Ceramic Society 23 (15), 2897–2904. Available at: https://doi.org/10.1016/S0955-2219(03)00301-7.

and thus reduce the likelihood of dentin and enamel separating during function. It also imparts strength through stress transfer (Francis *et al*., 1995).

Enamel is brittle and fragile. Cracks can propagate readily through the enamel, but they seldom propagate into the dentin (**Fig. 5**) (Marshall *et al*., 2003). It is because in contrast to the artificial dental adhesive materials between manmade dental crowns and dentin, the natural DEJ rarely fails, except when it is affected by genetic disorders. The natural DEJ acts as the bond between dentin and enamel and also resists cracks that originate in enamel from penetrating the dentin. Therefore, the knowledge of the mechanical properties and toughening mechanisms of DEJ is useful in the design of robust artificial dental restoration materials and structures (White *et al*., 2005).

The Young's modulus of the natural DEJ area has been measured by the atomic force microscope (AFM) based nanoindentation techniques. The results show that, within the DEJ region, Young's modulus changes gradually from that of hard and brittle enamel (~ 70 GPa) to that of the softer and durable dentin (~ 20 GPa) (**Fig. 6**) (Fong *et al*., 2000; Marshall *et al*., 2001).

The fracture behaviors of DEJ have been investigated through several experimental techniques. Fracture testing has been performed on chevron-notched short-bar bovine DEJ specimens, in which a crack initiated at the vertex of the chevron in the enamel, went across the DEJ zone and propagated into the bulk dentin (Lin and Douglas, 1994). The results showed that there was an extensive plastic deformation (83%) associated with the fracture process in the DEJ zone. The fractography revealed the deflection of crack path in an area that was about 50–100 μm deep. The coarse collagen bundles within the DEJ may play a significant role in resisting the cracks (Lin and Douglas, 1994). The failure mechanisms of DEJ have also been investigated using micro-indentation tests. The results show that when the micro-indentation was performed near the DEJ, the DEJ did not undergo catastrophic interfacial delamination. Instead, the damage was distributed over a broad region. It occurred primarily within the adjacent enamel and sometimes in the adjacent dentin. However, the DEJ interface resisted delamination, as shown in **Fig. 7** (White *et al*., 2005). Results of other fracture tests once again demonstrated that it is extremely difficult to initiate cracks from the dentin side of the DEJ or propagate cracks from enamel to dentin through the DEJ (Marshall *et al*., 2001; Rasmussen, 1984; Xu *et al*., 1998). All of these may explain the fact that the multiple full-thickness cracks commonly found in the enamel in intact teeth do not typically cause a total fracture of the tooth by extending the cracks into dentin (Lin and Douglas, 1994).

Dental Materials and Their Integrity

Dental Materials

For a very long time in human history, people have attempted to fully or partially repair or replace teeth. Several thousand years ago, materials such as ivory, bone, wood, metals/alloys, and animal teeth, were used because they looked or felt like natural human teeth (Ratner *et al*., 1996). Nowadays, a wide range of materials is used in preventative and restorative dental medicine, including metals, ceramics, polymers, and composites.

Metals and alloys are used in almost all aspects of dental medicine, including dental restorations, implants, dental braces, and instruments in dental laboratories and clinics. Amalgam has been used for almost 200 years as a dental restorative material, especially as a dental filling. An amalgam is an alloy of mercury and one or more other metals, including silver, tin, copper, zinc, and palladium. Both noble alloys and base-metal alloys have been used for cast dental restorations. In noble alloys, the noble metal elements can be gold, platinum, palladium, iridium, ruthenium, and rhodium; the base metals include silver, copper, zinc, indium, tin, gallium, and nickel. Ranging from the softest to the hardest casting alloys, they can be used for inlays, onlays, crowns, and fixed and removable dental prostheses. Base metal alloys are also used extensively for a variety of restorations and instruments.

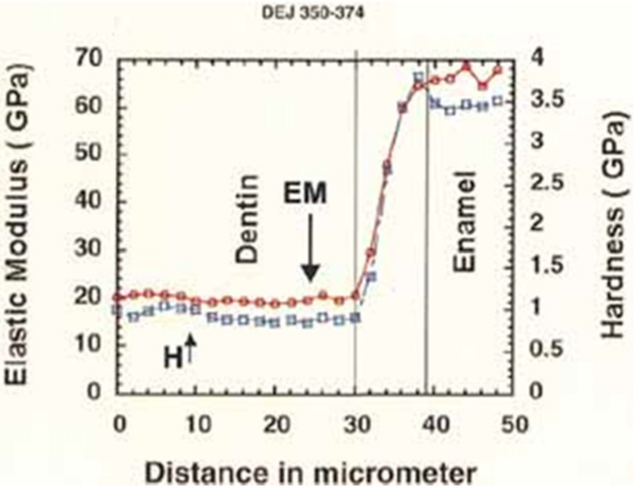


Fig. 6 Gradually changed hardness and elastic modulus over DEJ region. Reproduced from Marshall, G.W., Balooch, M., Gallagher, R.R., Gansky, S.A., Marshall, S.J., 2001. Mechanical properties of the dentinoenamel junction: AFM studies of nanohardness, elastic modulus, and fracture. Journal of Biomedical Materials Research 54 (1), 87–95. Available at: http://www.ncbi.nlm.nih.gov/pubmed/11077406.

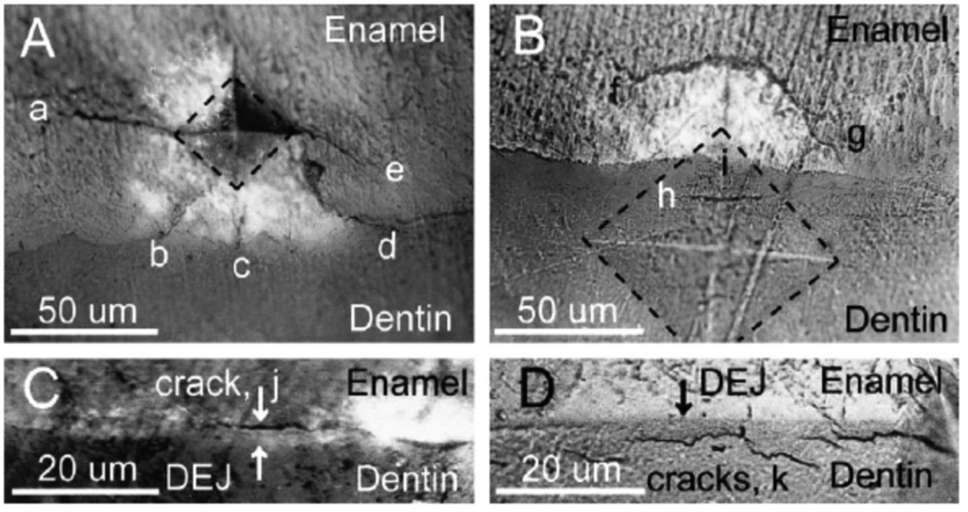


Fig. 7 Light micrographs of DEJ zone after indentation. Dashed rhombi indicate footprints of indentations, where letters a – k denotes cracks and capitalized letter A – D denotes the number of 4 images. A: Indentation centered on enamel side of optical DEJ; B: Indentation centered on dentin side of optical DEJ; C: Crack within enamel is close to and parallel to DEJ but does not involve optical DEJ itself (j); D: Series of interrupted cracks or partial tearing within dentin that are close to and slightly oblique to DEJ but do not involve optical DEJ itself (k). Reproduced from White, S.N., Miklus, V.G., Chang, P.P., *et al.*, 2005. Controlled failure mechanisms toughen the dentino-enamel junction zone. The Journal of Prosthetic Dentistry 94 (4), 330–335. Available at: https://doi.org/10.1016/j.prosdent.2005.08.013.

Cobalt-chromium alloys and nickel-chromium alloys have both been used in ceramic-metal restorations. Cobalt-chromium alloy, titanium, and titanium alloys have been used in dental prostheses. Titanium and titanium alloys can be used to make implants and crowns. Stainless steels, cobalt-chromium-nickel alloys, and nickel-titanium alloys have been used for orthodontic wires and endodontic instruments.

Ceramics were first used in dentistry in 1774 and effectively solved problems of stained, decayed, and terminally malodorous dentures (Kelly, 1997). More importantly, they are hard and biocompatible. However, despite these advantages, ceramic was not as widely used in prostheses as the original expectations (Rekow and Thompson, 2007). Early ceramics have different optical properties than natural teeth, were awkward to fit as prostheses, and had high failure rates. Efforts were made to improve the esthetics and fabrication methods for the purpose of dental practice (Kelly, 1997). Since the 1960s, the use of dental ceramics has been broadened to metal-ceramic restorations as they were being fused onto metal substructures (McLean, 1979). Later, the strength of dental ceramics has been improved through methods such as dispersion strengthening. The all-ceramic dental restorations, such as crowns and bridges, have then become more popular.

Table 3 Mechanical properties of selected dental materials

	Compressive Strength (MPa)	*Flexural Strength (MPa)*	*Diametral Tensile Strength (MPa)*	*Ultimate Tensile Strength (MPa)*	*Shear Strength (MPa)*	*Knoop Hardness Number (kg/mm^2)*
Amalgam	189	124	54	32	188	
Calcium hydroxide liner	8		1	2.3		
Feldspathic porcelain	149	65		25	111	460
Die stone	81	17	8	6		
Resin composite	225	139				
Zinc phosphate cement	110				13	38
Resin-modified glass ionomer		42–68				
Resin cement		66–121				
Acrylic denture resin					122	21
Cobalt-chromium alloy						391
Silicon carbide abrasive						2480

Note: Reproduced from Sakaguchi, R.L., Powers, J.M., 2012. Craig's Restorative Dental Materials. Elsevier. Available at: https://doi.org/10.1038/sj.bdj.2012.659.

At present, the commonly used dental ceramics include zirconia, alumina, and leucite. They are used in metal-ceramic restorations or all-ceramic restorations, partial or full restorations. Dental porcelain is a specific type of dental ceramic that is made from a mixture of kaolin, quartz, and feldspars and is used as veneers on crowns. Besides partial and full dental restorations, ceramics are also used in filling materials as particulate fillers for resin matrix composite. Zirconia is also used to fabricate dental implants.

Polymers are commonly used for dental applications such as tooth restorations, sealants, dental cement, orthodontic space maintainers and elastics (rubber bands), bases of denture bases, prostheses for palate defects, dental impressions, provisional restorations, root canal filling materials, and athletic mouth guards.

A composite is a combination of two or more materials with different properties. In dentistry, the most common composite is a combination of a polymer and ceramic particles, in which the polymer functions as the matrix and the particles are reinforcing materials. Polymer-ceramic composites, also known as resin composites, are used in sealants, fillings, crowns, veneers, provisional restorations, denture teeth, and cement. Glass ionomers have polymer or copolymer of carboxylic acids as their matrix and reactive fluoroaluminosilicate glass powders as fillers. They can release fluoride and can also physically and chemically bond to teeth. Therefore, they are used as filling materials when isolation is difficult and fluoride release is desirable, as well as dental cement.

Tissue engineering is a rapidly growing area with the goal of the restoration, maintenance, or enhancement of tissues. In periodontology and oral and maxillofacial surgery, bone tissue transplant materials are often used to repair defects. The advancement in tissue engineering demands the development of biological materials for scaffolds and the investigation of interactions between biomaterials and cells.

Integrity of Dental Materials and Structures

In order to withstand the rigors of dental applications in the oral environment, dental materials must meet a number of mechanical, chemical, thermal, and optical requirements. The key properties include their strength, fracture toughness, wear resistance, hardness, biocompatibility, and esthetics (Francis *et al.*, 1995). Some key mechanical Properties of selected dental materials are listed in **Table 3** (Sakaguchi and Powers, 2012).

Compressive strength can be obtained from compression tests of specimens with suitable height. It is especially useful for brittle materials that are weak in tension, such as amalgam, resin composites and cement. Flexural strength can be obtained by three-point bending or flexure test of beam specimens. The deformation obtained from the testing is also useful. Using the diametral compression test for tension or the Brazilian method, the ultimate tensile strength of a brittle material can be measured through compressive testing. The shear properties of dental materials can be determined by a torsional test. Shear strength is also important for the interfaces between two materials, such as ceramic-metal or implant-bone interface. In a push-out test, an axial load is applied to push one material through another and the shear strength can be estimated by the compressive force and the geometry of the specimen, assuming pure shear. Bond strength of dental adhesive materials to enamel and dentin can be tested by shear test, tensile test, or push-out test.

The fracture toughness of dental materials can be measured using a three-point bending test of single-edge notched beams. Fractographic analysis can help define the cause of failure, especially for brittle materials, such as dental ceramics. The fatigue strength can be measured using a fatigue test, in which multiple cycles of loading and unloading are applied. Tensile, compressive, shear, bending, and torsional fatigue tests can all be performed. The environment, such as temperature, humidity, saliva, and pH value, can all affect fatigue properties.

Hardness test is done by indentation using various indenters, such as Brinell hardness, Knoop hardness, Vickers hardness, Rockwell hardness, and Barcol hardness, which can be performed for dental materials. Nanoindentation techniques have been used to measure the Young's modulus and hardness of hard dental tissues. Dynamic mechanical analysis (DMA) can be carried out to measure the viscoelastic properties, such as storage and loss moduli, of polymers. Similarly, the rheology test provides the storage and loss shear moduli. The wear resistance of dental restorative materials can be gauged by two-body abrasion tests.

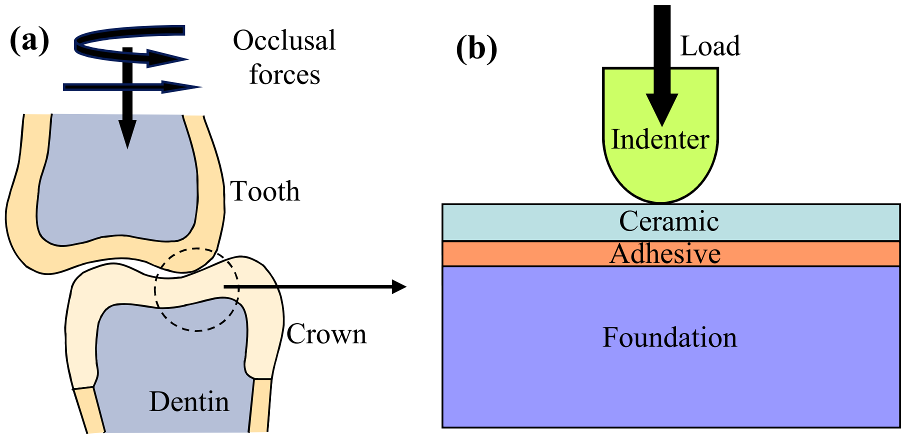


Fig. 8 Real occlusal activities and model. (a) Schematic of occlusal activities of teeth/crowns; (b) Simplified multilayered model structure under Hertzian contact.

The deformation and strain in the bone-tooth and bone-implant structures can be experimentally measured or numerically computed. The conventional strain measurement methods include strain gauges, (Akca *et al.*, 2007; Jantarat *et al.*, 2001; Popowics *et al.*, 2004; Asundi and Kishen, 2000a) photoelasticity, (Asundi and Kishen, 2000b, 2001) Moiré interferometry, (Kishen *et al.*, 2006; Wood *et al.*, 2003; Wang and Weiner, 1998) electronic speckle pattern interferometry (ESPI), (Zaslansky *et al.*, 2005, 2006; Zhang *et al.*, 2001) and digital image correlation (DIC) (Zhang *et al.*, 2009b; Tiossi *et al.*, 2011; Rossman *et al.*, 2017). They can measure the strain on the exposed surfaces. Mechanical testing coupled with micro-CT and digital volume correlation can measure the 3D full-field strain in bone-tooth and bone-implant structures (Du *et al.*, 2015a; Zhou *et al.*, 2020). Stress and strain in dental structures can also be calculated using numerical methods, such as finite element analysis. Combined with micro-CT, realist 3D finite element models can be built and the results can be compared with those measured using the above-mentioned 3D full-field method (Mao *et al.*, 2019; Su *et al.*, 2021).

Contact Damage of Dental Multilayers

The strong interest in improving the live time and the performance of posterior all-ceramic crowns has stimulated basic research on the contact damage of dental multilayers (Kelly, 1997; Rekow and Thompson, 2007; Huang *et al.*, 2005; Lawn *et al.*, 2000, 2004; Lee *et al.*, 2002; Malament and Socransky, 1999; Zhang *et al.*, 2004a; Zhou *et al.*, 2007). Dental multilayers are used in these studies as the engineering idealizations of dental crowns. The curved complex structures of actual dental crowns are simplified to flat multilayered structures with circular cross-sections that have similar sizes to real human teeth (Huang *et al.*, 2005; Zhang *et al.*, 2004a; Zhou *et al.*, 2007; Niu and Soboyejo, 2006; Niu *et al.*, 2008). The occlusal forces are simplified to Hertzian contact, contact between a hemispherical indenter head and the flat dental multilayers (Shrotriya *et al.*, 2003; Huang *et al.*, 2005; Lawn *et al.*, 2004; Zhang *et al.*, 2004a; Zhou *et al.*, 2007; Niu and Soboyejo, 2006; Niu *et al.*, 2008; Zhang *et al.*, 2004b). Schematics of actual occlusal contact between a tooth and a crown and the simplified Hertzian contact on a flat multilayer are shown in **Fig. 8** (Shrotriya *et al.*, 2003). The flat multilayered structures usually consist of a top layer of ceramic or glass with a thickness of 0.5–1.5 mm, a substrate layer of polymer or polymer-based composite with a thickness of 4–12 mm, and an adhesive layer in between with a thickness of ~100 μm. The diameter of the hemispherical Hertzian indenter varies from 0.8 to 20 mm (Shrotriya *et al.*, 2003). The Young's modulus of the top ceramic or glass layer can be between 70 and 400 GPa. The modulus of the substrate polymeric layer is about 18 GPa. If the adhesive layer is made of dental cement, its Young's modulus is generally between 3 and 5 GPa.

Fig. 9 shows some commonly observed damage and cracking modes in the dental multilayered structures under Hertzian contact loading (Niu, 2008). **R** denotes the sub-surface radial cracking that often occurs from the bottom surface (sub-surface) of ceramic layers (Kelly, 1997; Huang *et al.*, 2005; Zhou *et al.*, 2007; Niu and Soboyejo, 2006; Niu *et al.*, 2008; Huang *et al.*, 2007c). As shown in the finite element simulations by Huang *et al.*, stress concentrations occur in the sub-surface regime in the top ceramic layer because of the mismatch of Young's moduli in the multilayer structures (Huang *et al.*, 2007b). The Hertzian contact loading often leads to the pop-in of sub-surface radial cracks, which is consistent with the major failure mode of ceramic dental crowns observed in a clinical environment, as reported by Kelly (1997). These three-dimensional cracks initiate from the center of the bottom surface of the top ceramic layer and propagate upward towards the top surface of the dental ceramic and propagate along the radial directions at the same time. They can sometimes cause the bulk failure of the whole multilayer structure (Lin and Douglas, 1994; Rekow and Thompson, 2007; Tsai *et al.*, 1998; Thompson *et al.*, 1994). A photo of typical radial cracking at the initiation stage in a glass/adhesive/substrate structure is shown in **Fig. 10**(a) (Lawn *et al.*, 2002). An SEM image in **Fig. 10**(b) presents the morphology of the radial cracks that mainly propagate along grain boundaries, with some incidence of transgranular cracking in some of the grains.

Letter **C** in **Fig. 9** represents the cone cracks that initiate from the top surface of the ceramic layer and propagate towards the interfaces between ceramic and adhesive layers. As shown in **Fig. 11**(a), observing from the top view, they appear just outside of the contact area

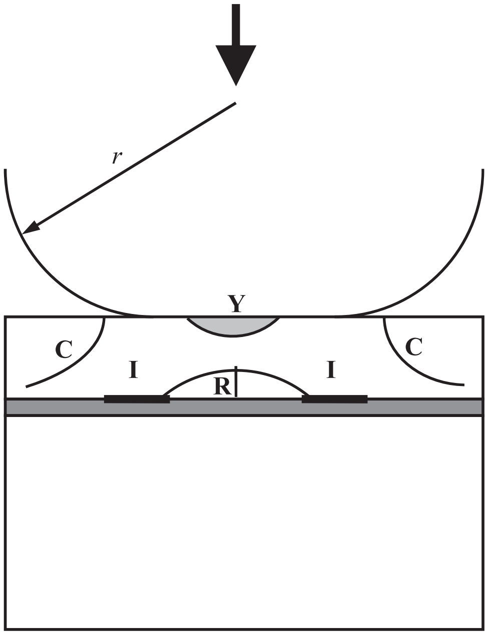


Fig. 9 Multiple failure methods in dental multilayer structures under Hertzian contact. R - Sub-surface radial cracking; C - Cone cracks; Y - quasi-plastic damage; I - Interfacial de-bonding. Fracture surface created by radial cracks. Niu, X., 2008. Contact Damage of Dental Multilayers. (November).

between the indenter and the ceramic/glass top layer. **Fig. 11**(b) shows the side view of these cracks in a glass/adhesive/substrate multilayer structure (Shrotriya *et al.*, 2003). Letter **Y** in **Fig. 9** represents the quasi-plastic damage that occurs right underneath the loading indenter. It is a form of yielding in response to the shear stresses (Lawn *et al.*, 1994; Lawn, 2005). Some typical photos of the quasi-plastic damage in indented glass-ceramics are presented in **Fig. 12** (Peterson *et al.*, 1998). Letter **I** in **Fig. 9** represents the interfacial de-bonding between the ceramic/glass top layer and the adhesive layers. An SEM image of interfacial cracks between zirconia ceramic and dental cement adhesive layers is presented in **Fig. 13** (Niu *et al.*, 2008). As shown in the figure, interfacial de-bonding is often associated with the propagation of radial cracks. Other minor failure modes may also develop in the multilayer structures, including median, outer, inner cone cracks and partial cone cracks, depending on the loading conditions and the environmental conditions.

Slow Crack Growth (SCG) and Viscosity-Assisted SCG

The power law slow crack growth (SCG) model was developed in the 1970s. It is an elastic model that describes the stable sub-critical crack growth behavior, (Wiederhorn, 1974) given by

$$\int_0^{t_R} \sigma(t)^N dt = D \tag{1}$$

where t is the time; $\sigma(t)$ is a time-dependent expression of the stress; t_R is the rupture time, at which sub-surface radial cracks occur. When the loading rate, $\dot{P}$, is constant, the rupture time, t_R, can be expressed as: $t_R = P_c/\dot{P}$, where P_c is the critical load. The parameter, D, is a time and load-independent quantity that is given by (Huang *et al.*, 2007a):

$$D = \frac{K_{Ic}^N\left(c_0^{1-N/2} - c_f^{1-N/2}\right)}{(N/2-1)v_0\beta^N} \tag{2}$$

where K_{Ic} is the fracture toughness; N is the crack velocity exponent, which is a material property; v_0 is the crack velocity; c_0 and c_f are the initial and final radial crack size; β is a crack geometry coefficient (Lee *et al.*, 2002; Huang *et al.*, 2007a; Dabbs *et al.*, 1982).

The SCG model was adopted by researchers to explain the occurrence of subsurface radial cracks in the top ceramic layers in dental multilayer structures (Zhang *et al.*, 2004a; Niu and Soboyejo, 2006; Zhang *et al.*, 2009a). When using this model, it is assumed that during contact damage in the dental multilayered structures, sub-surface radial crack growth occurs solely as a result of slow crack growth in the top ceramic layers (Lee *et al.*, 2002; Zhang *et al.*, 2004a; Niu and Soboyejo, 2006). When the initial crack length is very small, and the final crack length is much greater than the initial crack length, the crack propagation can be described by the SCG theory (Lee *et al.*, 2002; Huang *et al.*, 2007a; Dabbs *et al.*, 1982). The model can be implemented analytically or numerically. The dental multilayer structure shown in **Fig. 8** (b) can be simplified as a bi-layered structure when the middle adhesive layer is very thin. For the monotonic loading condition, when the bi-layer structure is loaded at a fixed loading rate, $\dot{P}$, the failure condition could be expressed as (Lee *et al.*, 2002)

$$P_c = \left[A(N+1)\dot{P}\right]^{1/(N+1)} \tag{3}$$

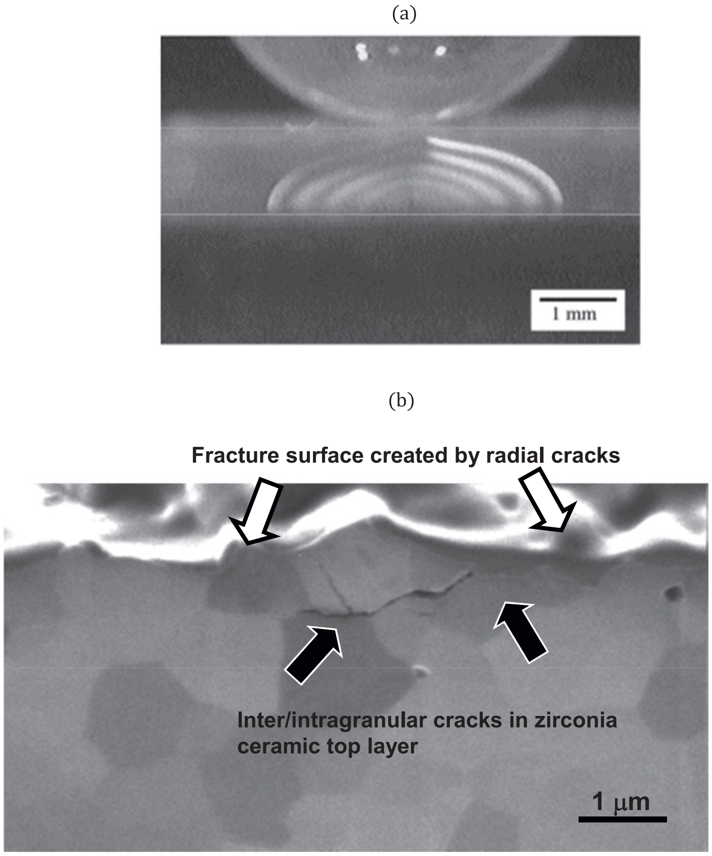


Fig. 10 (a) Image of radial cracks initiated at the sub-surface of top glass layer in glass/adhesive/substrate dental multilayer structure (b) focused ion beam (FIB) image shows inter/intragranular cracks in zirconia ceramic top layer. The microcracks are associated with radial cracks. Reproduced from Lawn, B.R., Deng, Y., Miranda, P., *et al*., 2002. Overview: Damage in brittle layer structures from concentrated loads. Journal of Materials Research 17 (12), 3019–3036. Available at:. https://doi.org/10.1557/JMR.2002.0440. Niu, X., Yang, Y., Soboyejo, W., 2008. Contact deformation and cracking of zirconia/cement/foundation dental multilayers. Materials Science and Engineering: A 485 (1–2), 517–523. Available at: https://doi.org/10.1016/j.msea.2007.09.014.

where P_c is the critical failure load; A is a load, time, and thickness independent quantity, and N is the crack velocity exponent. For the fatigue loading with constant frequency, the failure condition is given by (Lee *et al.*, 2002)

$$P_{max}^{N} t = 2AN^{0.47} \tag{4}$$

where $P_{\max}$ is the maximum load of the sinusoidal loading, and t is the time to failure.

The model can also be implemented using the finite element method (Du *et al.*, 2013). Using finite element models, the time-dependent stress, $\sigma(t)$, at the center of the sub-surface of the ceramic top layer in the dental multilayer structure can be calculated. The critical load versus loading rate measured by the Hertzian contact experiment can then be used to fit the value of D. Since D is only related to material properties and geometry, it should be consistent for all different loading rates. Therefore, with D known, Eq. (1) can be integrated to obtain the rupture time t_R and then the critical load P_c.

The SCG model captures the contact damage of dental multilayer structures solely contributed by the slow crack growth in the top ceramic layer. However, it treats all the materials as purely elastic and the viscous flow in the substrate and middle layers is neglected. In an effort to consider the combined effects of slow crack growth in the top ceramic layer and the viscosity-induced loading rate effects in adhesive and substrate layers, the rate-dependent Young's modulus assisted SCG (RDEASCG) model, was developed for the prediction of critical loads in multilayered dental structures under monotonic loading (Niu and Soboyejo, 2006).

Many biomaterials exhibit time-dependent viscoelastic or creep behavior. For metallic biomaterials, viscous behavior is often associated with diffusion or dislocation motion. Whereas for polymeric biomaterials, viscosity is associated with chain stretching, unfolding, or sliding (Soboyejo, 2003). The mechanical response due to viscous flow could be represented by models containing various combinations of springs and dashpots. **Fig. 14**(a) shows the Maxwell model that has a spring and a dashpot in series. The solution of the Maxwell Model can be expressed as (Soboyejo, 2003)

$$\frac{d\varepsilon}{dt} = \frac{\sigma}{\eta} + \frac{1}{E}\frac{d\sigma}{dt} \tag{5}$$

where ϵ is the strain, σ is the stress, E is the Young's modulus, η is the viscosity and t is the time. **Fig. 14**(b) shows the Voigt model

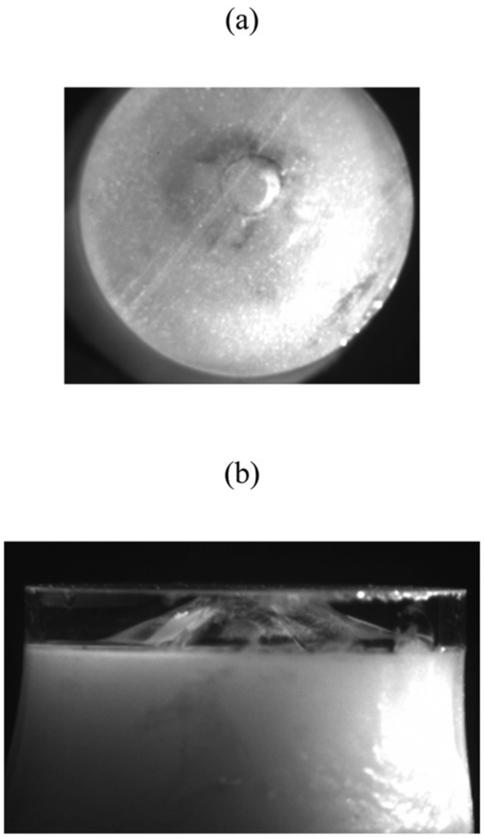


Fig. 11 Images of cone cracks in the of top glass layer in glass/adhesive/substrate dental multilayer structure: (a) top view and, (b) side view. Reproduced from Shrotriya, P., Wang, R., Katsube, N., Seghi, R., Soboyejo, W.O., 2003. Contact damage in model dental multilayers: An investigation of the influence of indenter size. Journal of Materials Science: Materials in Medicine 14 (1), 17–26. Available at: http://www.ncbi.nlm.nih.gov/pubmed/15348534.

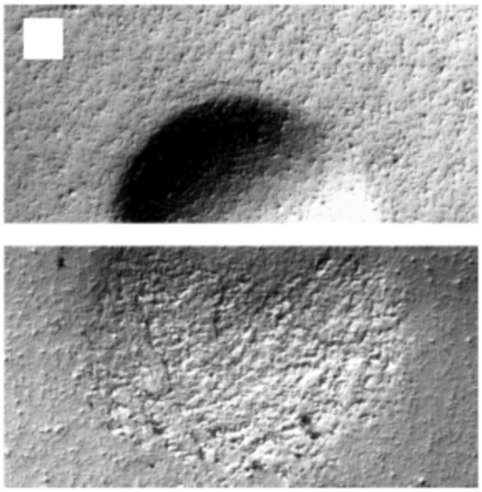


Fig. 12 Top and section views of the quasiplastic damage in bonded-interface specimens of coarse-grain micaceous glass–ceramic, from indentation with tungsten carbide sphere of 3.18 mm radius at 1000 N load. Fracture surface created by radial cracks. Reproduced from Peterson, I.M., Wuttiphan, S., Lawn, B.R., Chyung, K., 1998. Role of microstructure on contact damage and strength degradation of micaceous glass-ceramics. Dental Materials : Official Publication of the Academy of Dental Materials 14 (1), 80–89. Available at: http://www.ncbi.nlm.nih.gov/pubmed/9972155.

in which the spring and dashpot are combined in parallel. The response of the Voigt model is expressed as (Soboyejo, 2003)

$$\varepsilon = \frac{\sigma_0}{E}\left[1 - \exp\left(-\frac{t}{\tau}\right)\right] \tag{6}$$

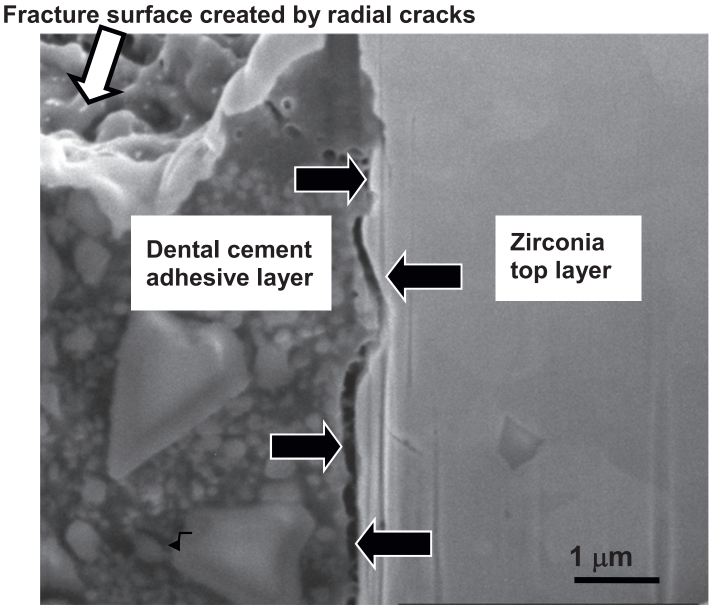


Fig. 13 SEM picture shows interfacial cracking between zirconia ceramic and dental cement adhesive layers, where solid arrows indicate interfacial cracks and hollow arrows indicate fracture surface created by radial cracks. Reproduced from Niu, X., Yang, Y., Soboyejo, W., 2008. Contact deformation and cracking of zirconia/cement/foundation dental multilayers. Materials Science and Engineering: A 485 (1–2), 517–523. Available at: https://doi.org/10.1016/j.msea.2007.09.014.

where σ_0 is the initial stress level and $\tau = \eta/E$ is the relaxation time. For material with more complex viscoelastic behaviors, three-element model (**Fig. 14**(c), four-element model, or even more complex Kelvin model can be used (Soboyejo, 2003). The 3-element model, also known as the Zener creep model, is expressed as (Soboyejo, 2003)

$$\frac{1}{E} = \frac{1}{k_1}\left\{1 - \frac{k_2}{k_1 + k_2}\exp\left[-\frac{k_1 k_2 t}{\eta(k_1 + k_2)}\right]\right\} \tag{7}$$

where t is the time, k_1, k_2 and η are the spring constants and dashpot constant shown in **Fig. 14**(c). For polymeric materials, the Prony series model can be used to describe their viscoelastic behaviors. Due to the heterogeneity of the polymeric structures, the relaxation times of polymers occur in a distribution (McCrum *et al.*, 1997). Prony series use a series of relaxation times to fully describe the creep or relaxation of polymeric structures, over an extended period **Fig. 14**(d). It is given by (Bower, 2011)

$$G(t) = G_0(1 - \sum_{i=1}^{N} g_i(1 - e^{-t/\tau_i})) \tag{8}$$

where $G(t)$ is the time-dependent shear modulus; G_0 is the instantaneous modulus; $\tau_i$is the relaxation time and g_i is the relaxation coefficient. When there is only one exponential term, the Prony series is equivalent to the Zener model.

The process of using RDEASCG mode to predict the critical loads in multilayered dental structures under monotonic loading is illustrated in **Fig. 15** (Niu and Soboyejo, 2006; Du *et al.*, 2015b). Using the above-mentioned models for viscous behaviors, the time-dependent stress, $\sigma(t)$, at the center of the sub-surface of the ceramic top layer can be calculated analytically or using numerical methods, such as finite element models. One of the critical loads obtained from the Hertzian contact experiments was then used to fit the value of D. Since D is only related to material properties and geometry, it should be consistent for different loading rates. Therefore, with D known, Eq. (1) was integrated to obtain the rupture time, t_R, and then the critical load P_c.

Under cyclic loading conditions, to model the viscosity-assisted fatigue behavior, a substrate creep effect (SCE) fatigue model was developed (Zhou *et al.*, 2007) to provide the relationship between the maximum load and the failure time that corresponds to the pop-in of the sub-surface radial crack in the top glass layer. The pop-in loads can be predicted from the equation shown below

$$P_m^N\left[log\left(\frac{CE_c}{k_1}\right)\left\{1 - \frac{k_2}{k_1 + k_2}\exp\left[-\frac{k_1 k_2}{\eta(k_1 + k_2)}\right]\right\}\right]^N t_f = H \tag{9}$$

where t_f is the time to failure, E_c is the Young's modulus of the ceramic top layer, N is the crack velocity exponent, C is a dimensionless coefficient, and H is a material- and geometry-dependent constant.

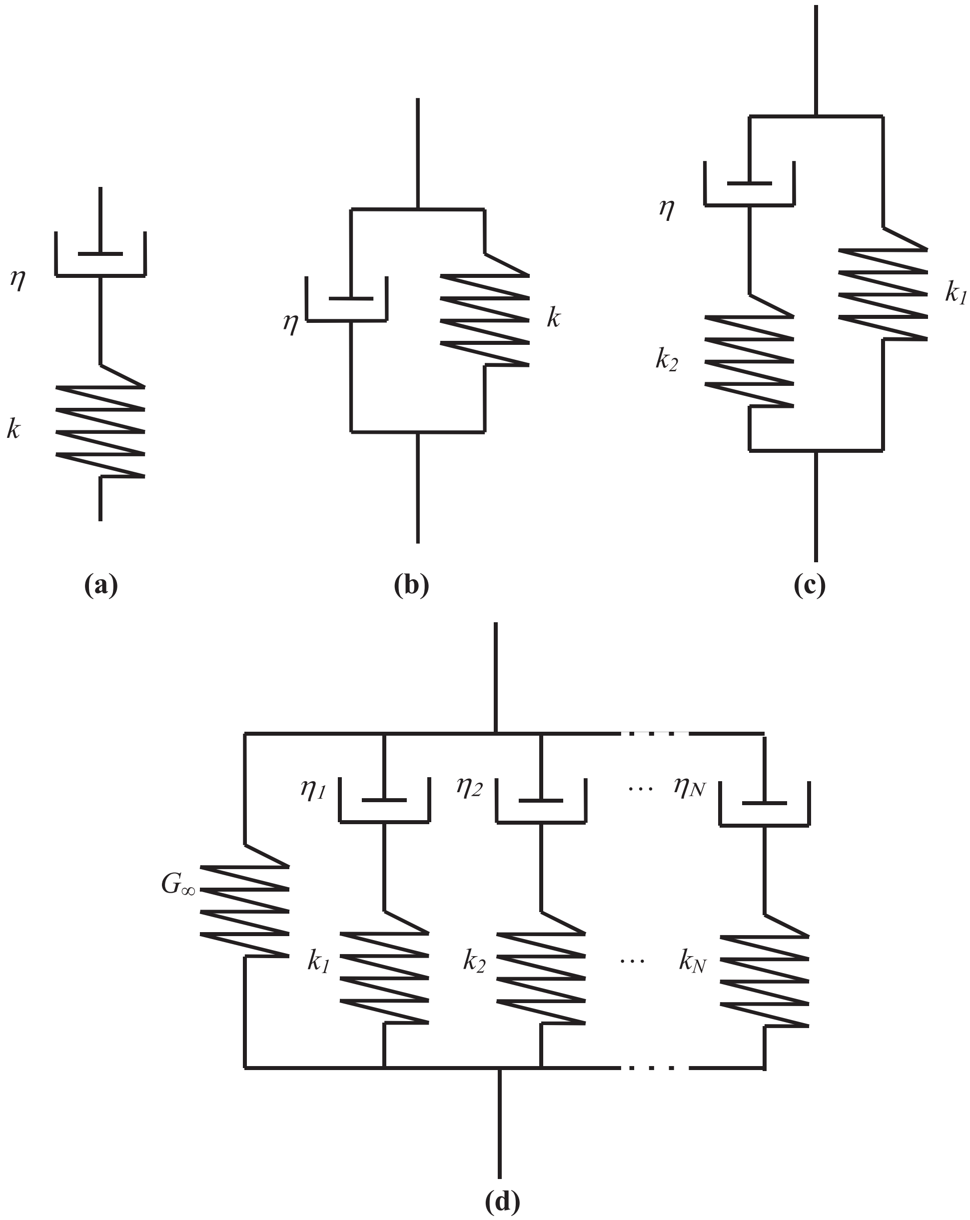


Fig. 14 Schematic of viscoelastic spring dash models: (a) Maxwell model, (b) Voigt model, (c) 3-element Kelvin model (Zener model), and (d) Prony series.

Bio-Inspired Design of Dental Materials

Motivation and Inspiration

Ceramics are brittle and generally have poor crack growth resistance, especially when initial defects are introduced during the fabrication processes. All-ceramic posterior crowns show poor performance (Rekow and Thompson, 2007). An earlier study shows that dental crowns continue to fail at a rate of approximately 3% each year (Burke *et al.*, 2002). The posterior crowns and bridges have the highest fracture rates because the stresses are greatest in them (Rekow and Thompson, 2007). Prior research has also shown that nearly 20% of the posterior all-ceramic dental crowns with resin retained ceramics fail within the first 5 years of service in the oral cavity (Malament and Socransky, 1999).

At the present stage, the strengths of synthetic ceramics for dental crowns appear to be comparable with or greater than some of the dental hard tissues, as shown in **Table 1** (Sakaguchi and Powers, 2012; El Mowafy and Watts, 1986). However, the strength values do not reflect the crack growth resistance. The reported values (Rosenstiel and Porter, 1989; Taira *et al.*, 1990) of fracture toughness for ceramic dental materials are still noticeably lower than those for hard tissues in natural teeth (Francis *et al.*, 1995). Also, some ceramic dental materials have been shown to be aggressive toward opposing natural teeth (DeLong *et al.*, 1989).

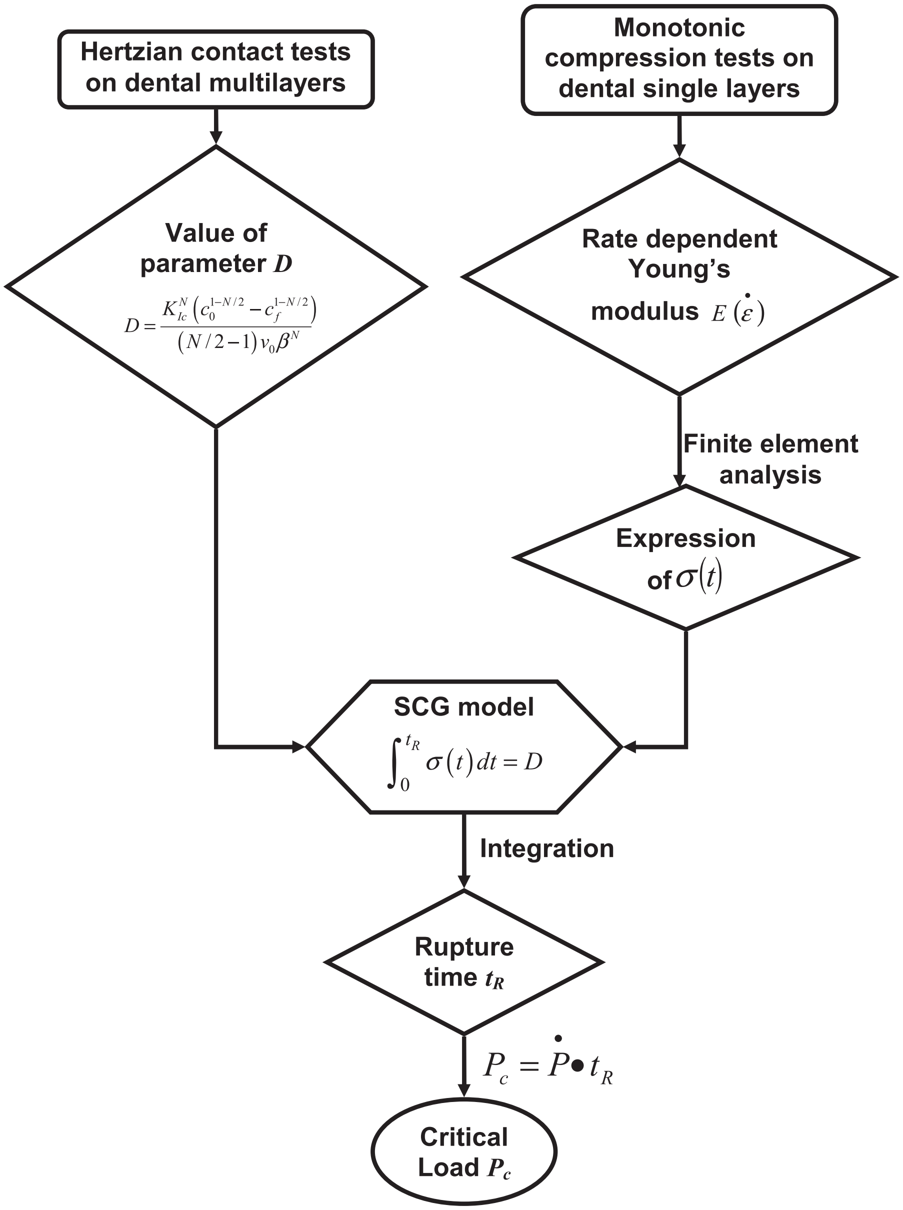


Fig. 15 Flow charts of the process of rate-dependent Young's modulus assisted SCG (RDEASCG) model. Reproduced from Niu, X., Soboyejo, W., 2006. Effects of loading rate on the deformation and cracking of dental multilayers: Experiments and models. Journal of Materials Research 21 (04), 970–975. Available at: https://doi.org/10.1557/jmr.2006.0114. Niu, X., Yang, Y., Soboyejo, W., 2008. Contact deformation and cracking of zirconia/cement/foundation dental multilayers. Materials Science and Engineering: A 485 (1–2), 517 523. Available at: https://doi.org/10.1016/j.msea.2007.09.014.

On the other hand, the financial drivers for developing damage-resistant crowns are high. Dental crowns generate over \$2 billion each year in revenues, with 20% of the units being all ceramic (Rekow and Thompson, 2007). The aging population is likely to drive the demand for dental crowns (Rekow and Thompson, 2007). Dental implants each require a dental crown on them and they account for about \$1 billion of revenue each year. The market for dental implants is estimated to be growing at a rate of 18% every year.

Overall, the natural tooth has superior properties to artificial dental crowns (Francis *et al.*, 1995). Hence, the knowledge of the structure and properties of natural human teeth is very important for the design of artificial dental crowns. Gradients exist in the microstructures and properties of the dental hard tissues and their interfaces. The mechanical properties, such as Young's modulus and hardness of enamel increase with the increasing distance from the DEJ (**Table 2**) (Park *et al.*, 2008). It is harder at the occlusal area and cusps and softer near the DEJ **Fig. 2**(c) (Cuy *et al.*, 2002). The dentin is stiffer near the enamel and softer near the root of the tooth. Enamel and dentin integrate and function together harmoniously through their complex interface, DEJ. The components from enamel and dentin interpenetrate through and intermingle within this interface (Rasmussen *et al.*, 1976; Lin *et al.*, 1993; Lin and Douglas, 1994). Within the DEJ region, the Young's modulus gradually reduces from that of hard and brittle enamel to that of the softer and durable dentin (**Fig. 6**) (Fong *et al.*, 2000; Marshall *et al.*, 2001).

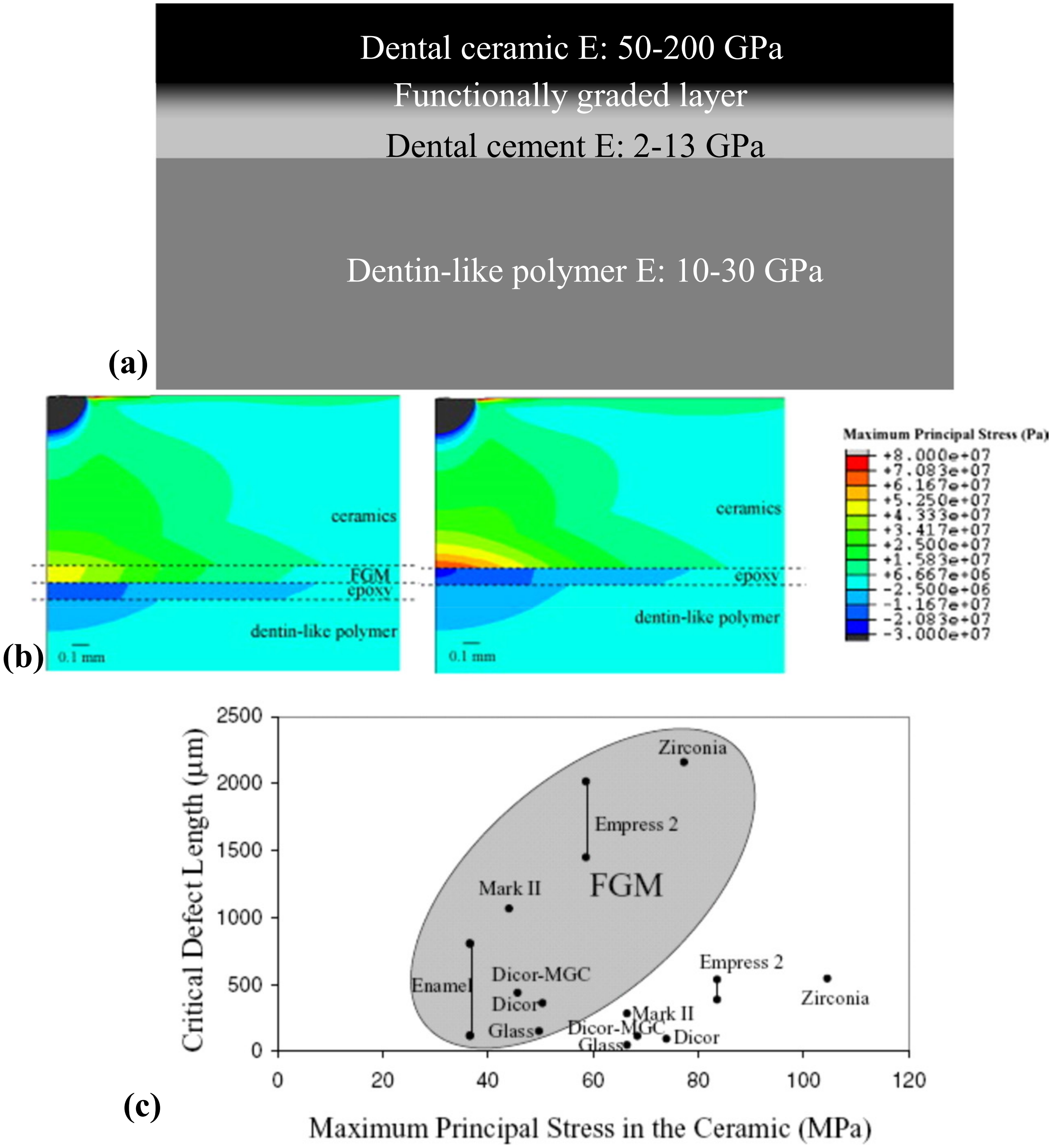
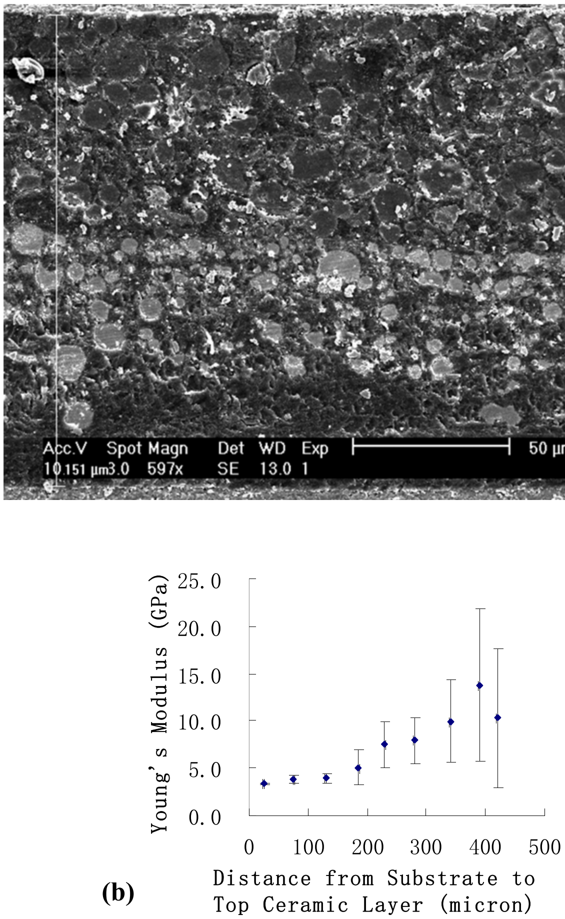


Fig. 16 (a) Schematic of a model dental multilayer structure with functionally graded layer between the cement and the ceramic layers. (b) Maximum principal stress in dental multilayer structures with FGM and without FGM. (c) Critical crack lengths versus maximum principal stresses in graded and non-graded multilayer structures. Adapted from Huang, M., Rahbar, N., Wang, R., *et al.*, 2007b. Bioinspired design of dental multilayers. Journal of Materials Science: Materials in Medicine 18 (1), 57–64. Available at: https://doi.org/10.1007/s10856-006-0662-0.

However, in commercially available dental restoration structures, gradients do not necessarily exist. They normally consist of a top ceramic layer, mimicking the enamel. The ceramic layer is supported by remaining dentin, which is mimicked using a ceramic-filled polymeric material in many dental multilayer studies. These two layers are bonded together by dental cement, which is usually a glass-filled resin-based material. In most dental crowns, the Young's modulus of the ceramic is between 70 and 400 GPa. The Young's modulus of the substrate polymeric layer is around 18 GPa, which is close to the Young's modulus of dentin. The dental cement generally has a Young's modulus between 3 and 5 GPa (Kelly, 1997). The mismatch in Young's moduli results in high stresses in the top ceramic layer and ultimately results in the sub-surface radial cracks in the top ceramic layer (Huang *et al.*, 2007b).

(a)



(b)

Fig. 17 (a) SEM image of the microstructure of functionally graded material. (b) Distribution of Young's moduli across the fabricated dental FGM layer. Reproduced from Niu, X., Rahbar, N., Farias, S., Soboyejo, W., 2009. Bio-inspired design of dental multilayers: Experiments and model. Journal of the Mechanical Behavior of Biomedical Materials 2 (6), 596–602. Available at: https://doi.org/10.1016/j.jmbbm.2008.10.009. Du, J., Niu, X., Rahbar, N., Soboyejo, W., 2013. Bio-inspired dental multilayers: Effects of layer architecture on the contact-induced deformation. Acta Biomaterialia 9 (2), 5273–5279. Available at: https://doi.org/10.1016/j.actbio.2012.08.034.

Concept and Design of Bio-Inspired Dental FGM

The concept of FGM arose in the mid-1980s as a way of improving the thermo-mechanical performance of aerospace structures. The gradation in the materials offers a way of maintaining the high strength of the inner core and high thermal resistance of the outer layer while eliminating the deleterious effects of a sharp interface (Rousseau and Tippur, 2001).

Inspired by both the prior work on FGMs and the graded structures and properties of natural teeth structure, Huang et al (Huang *et al.*, 2007b). Proposed to use a bio-inspired functionally graded material layer as a DEJ-like dental adhesive material in the dental multilayer structures. In their computational study, Huang *et al.* added an FGM layer with its Young's modulus gradually varying from that of dental cement to that of dental ceramic **Fig. 16**(a) (Huang *et al.*, 2007b). The results of finite element simulations **Fig. 16**(b) showed that adding an FGM adhesive layer can significantly reduce the stress concentrations in the sub-surface of the top ceramic layer. The reduction of stress concentration can potentially lead to increased resistance to sub-surface radial cracking. The critical crack lengths were estimated to be much greater than that in the commercially available dental crowns as well as that in the enamel-DEJ-dentin complex in natural teeth **Fig. 16**(b) (Huang *et al.*, 2007b). The result suggests the possibility of building synthetic, bio-inspired, functionally graded dental multilayers that have comparable or better durability than those of natural teeth.

Numerical simulations have also been carried out to study the effect of FGM architectures on the performance of the dental multilayer structure (Du *et al.*, 2013). The results of finite element simulations showed that as the thickness of the FGM increased, the stress concentration in the structure was reduced, and the maximum principal stress at the center of the sub-surface of the top ceramic layer decreased. Thicker FGM provides better support to the top ceramic layer because it has higher stiffness. However, when the dental multilayer was joined together by commercially available dental cement without FGM, the opposite is true, meaning the stress concentration increased with the increasing thickness of the adhesive layer (Du *et al.*, 2013). Simulations (Huang *et al.*, 2007b; Du *et al.*, 2013) have also shown that the linear gradient of Young's modulus in the FGM will provide balanced stress in the top ceramic layer and the FGM layer. The nonlinear gradient of Young's modulus in the FGM increased the

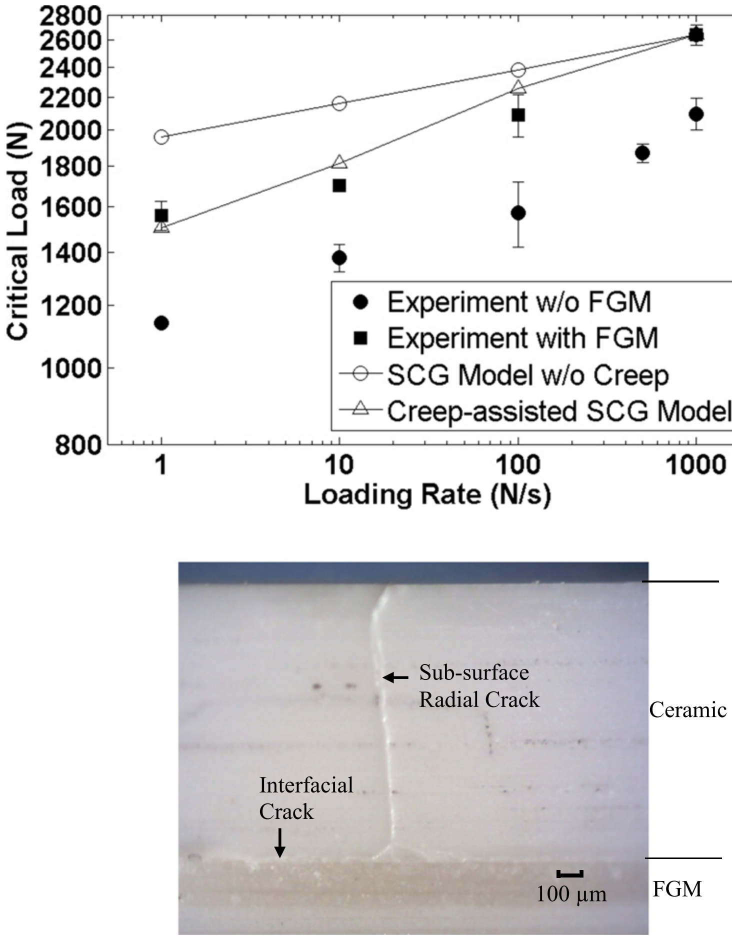


Fig. 18 (a) Comparison of the critical loads under Hertzian contact loading at various clinically relevant loading rates of the dental multilayer structures with and without FGM. (b) Failure modes of the dental FGM structure revealed by optical images of the cross-section after Hertzian contact tests. Reproduced from Du, J., Niu, X., Soboyejo, W., 2015b. Creep-assisted slow crack growth in bio-inspired dental multilayers. Journal of the Mechanical Behavior of Biomedical Materials 46, 41–48. Available at: https://doi.org/10.1016/j.jmbbm.2015.01.019. Du, J., Niu, X., Rahbar, N., Soboyejo, W., 2013. Bio-inspired dental multilayers: Effects of layer architecture on the contact-induced deformation. Acta Biomaterialia 9 (2), 5273–5279. Available at: https://doi.org/10.1016/j.actbio.2012.08.034.

stress in the top ceramic layer if the higher gradient is near the top ceramic layer and increased the stress in the FGM if the higher gradient is near the substrate (Du *et al.*, 2013). This suggests that the optimal architecture of dental FGM is around linear gradation, which is similar to the Young's modulus distribution in the DEJ in natural teeth.

Fabrication and Characterization of Bio-Inspired Dental FGM

A layer-by-layer deposition method was developed to fabricate the bio-inspired FGM structures using a polymer-based composite material reinforced by ceramic particles (Niu *et al.*, 2009). A steel plate mold containing holes was filled with a dentin-like composite material, which is a clinically used material for dental fillings, and then cured with UV light to become the substrate of the dental multilayer model structure. The FGM were fabricated using nanocomposites, a mixture of epoxy matrix and zirconia or alumina nanoparticles. After mixing the nanocomposite material, a wire-wound wet-film applicator rod was used to spread it across the steel plate. When the rod was pulled across the steel plate with a fluid film in front of it, the thickness of the fluid film was controlled by the threads on the applicator rods. After a layer had been deposited and spread, the plate was cured in a vacuum oven. The deposition and curing process was repeated multiple times to build up the multilayered structures. From bottom to top, the functionally graded nanocomposite layers contained ceramic particles with increasing weight fraction to increase the stiffness of the layers. The top layer with medical grade 3 mol.% yttria-stabilized zirconia was pressed onto the last layer of the FGM before curing (Du *et al.*, 2013; Niu *et al.*, 2009).

A typical SEM image of the fabricated dental FGM structure is presented in **Fig. 17**(a), showing its microstructure (Niu *et al.*, 2009). A total of 10 layers of nanocomposites with microscale layer thicknesses were used to construct the graded structure. Zirconia particles with increasing fraction were used in the bottom layers and alumina particles with higher modulus were used in the top layers. It reduced the possibility of particle clustering. The microsized ceramic particles are clusters of nanosized powders.

The nanoindentation technique was used to characterize the architecture of the FGM, i.e., the actual distribution of the Young's modulus on the cut and polished cross-section of the structure. The measured Young's modulus distribution is presented in **Fig. 17**(b) (Du *et al.*, 2013). The results suggested an almost sinusoidal variation in the Young's modulus across the FGM. The variabilities in the Young's moduli were partly attributed to the clustering and non-uniform particle distributions. The substrate was found to have a modulus of ~20 GPa, which mimics the Young's modulus of the remaining dentin in natural teeth. While the top zirconia ceramic layer had a modulus greater than 200 GPa.

Performance of the Bio-Inspired Dental FGM

Hertzian contact experiments were conducted to assess the performance of the dental multilayer structures with and without FGM. The tests were conducted at clinically relevant loading rates between 1 N/s and 1000 N/s. The low loading rates below 10 N/s correspond to the bruxism movements, while the higher loading rates correspond to the normal chewing actions. The critical load was determined from the discontinuity in the displacement as observed in the recorded load-displacement curves. Under any given loading rates, the dental multilayer structures with FGM exhibited ~20%–30% higher critical pop-in loads than those flat conventional dental multilayers without FGM **Fig. 18**(a) (Du *et al.*, 2015b; Niu *et al.*, 2009).

Following the cracking under Hertzian contact loading, the cut and polished cross sections of the tested samples were examined under an optical microscope to discover the failure modes of the dental FGM structure (Du *et al.*, 2013). **Fig. 18**(b) shows a typical sub-surface radial crack observed in the ceramic top layer as well as the interfacial cracking between the top zirconia ceramic layer and the FGM layer. The results suggested that critical load corresponded to the popin of radial cracks at the center of the subsurface of the top zirconia layer. The pop-in was associated with the propagation of radial crack into the top zirconia ceramic layer, along with partial interfacial cracking between the top ceramic layer and the FGM adhesive layer.

SCG model was used to investigate the crack growth and predict the critical loads. When all the materials were assumed to be linear elastic without any viscous behaviors, the critical loads predicted by SCG had good agreement with experimental measurements at higher loading rates, but the differences between SCG prediction and experimental measurements increased with lowering loading rates **Fig. 18**(a). These suggest that the dependence of the loading rate should be considered for the layer properties. Rate-dependent material properties were incorporated to consider the effects of the viscosity of the polymeric materials using the RDEASCG model (Niu and Soboyejo, 2006; Niu *et al.*, 2009) or the similar creep-assisted SCG model (Du *et al.*, 2015b). The predictions of the critical load were made by incorporating the viscoelastic behaviors into the FEM models and inputting the stresses obtained from the FEM simulation into the SCG model. The results show that the predictions from the creep-assisted SCG model were in closer agreement with the experimental results **Fig. 18**(a) (Du *et al.*, 2015b).

Other Bio-Inspired Dental FGM

There have been other efforts in making graded interfaces in various dental applications, with some of them having inspiration from the structures and properties of natural teeth. Efflandt *et al.* explored the bonding between bioactive glasses and dentin extracted from human teeth (Efflandt *et al.*, 2002). Their research suggested the exciting possibility of developing better dental adhesive material to bond to the dentin. Though using bioactive glasses in their bulk form is not a feasible option, the presence of bioactive glass may be beneficial to the bonding between dentin and the dental adhesives or tissue-engineered restorations (Efflandt *et al.*, 2002).

Zhang *et al.* produced a porous composite consisting of polysulfone (cellulose acetate) and bioactive glass particles that were fabricated using phase separation techniques (Zhang *et al.*, 2002). The composites had nonuniform structures with dense top layers and porous structures underneath. These porous composites may have potential applications as interfacial materials between soft and hard tissues, such as the interface between artificial cartilage and bone (Zhang *et al.*, 2002).

Francis *et al.* designed and processed a dentin-like material using alumina-glass or alumina-polymer composite and an enamel-like material using calcium phosphate-based coating. The interface between the above-mentioned material was created by deposition slurries of oxide or glass powder by a draw-down blade method, followed by drying and then higher temperature heating (Francis *et al.*, 1995). Microstructural analysis showed that a solidified eutectic CaO-A12O3-SiO2 (CAS) melt phase penetrated the man-made dentin-like layer below, created an interface similar to the DEJ in natural teeth, and provided strong bonding (Francis *et al.*, 1995). Compared with the natural DEJ, the DEJ-like interface developed in this study did not include collagen projections across the interface. Also, the fracture properties of the structures and the ability of the DEJ-like interface to interfere with or arrest crack propagation have not been investigated (Francis *et al.*, 1995).

Zhang *et al.* proposed to fabricate a graded interface between glass and ceramic using a glass-ceramic infiltration method (Zhang and Kim, 2009). A functionally graded glass/zirconia/glass structure was fabricated. The Young's modulus and hardness increased from the glass surface to the zirconia interior following power-law relations. The functionally graded structures exhibited improved damage resistance and esthetics compared to homogeneous yttria-stabilized tetragonal zirconia polycrystal plates (Zhang and Kim, 2009).

FGM can potentially be beneficial for dental implants, in terms of alleviating stress-shielding, enhancing osseointegration, and elongating the lifetime of the implants. The biomechanical behavior of an FGM dental implant and the interaction between the implant and the surrounding bone were investigated using the finite element method (Yang and Xiang, 2007). The computational results showed that the FGM in the implant effectively reduced the stress difference at the implant-bone interfaces where maximum stresses occurred. In another study, Traini *et al.* used a laser metal sintering technique to fabricate titanium alloy dental implants with a gradient of porosity from the inner core to the outer surface of the implant (Traini *et al.*, 2008). The FGM reduced the elastic modulus of the implant and reduced the difference between the elastic moduli of the implant and bone.

As for the general FGMs, there are a number of in-depth studies on the crack growth resistance and the fracture behavior of FGMs. Rousseau *et al.* studied the deformations at the crack tip that is parallel to the gradient direction of FGM and subjected to low-velocity, symmetric impact loading (Rousseau and Tippur, 2001). Paulino *et al.* have developed accurate graded elements for the modeling of FGM using FEM (Paulino and Jin, 2003; Kim and Paulino, 2003; Walters *et al.*, 2004). Such models could form the basis for the future work of computing crack driving forces and gaining a better understanding of fracture processes in FGM.

Future Directions

The study of the bio-inspired design of dental multilayers showed that bio-inspired FGM significantly increased the critical loads of dental multilayers under monotonic loading. However, there are still some challenges that must be resolved before the bio-inspired FGM can be potentially applied in actual dental crowns. The previous studies focus mostly on the performance of these structures under monotonic loading. Further work is clearly needed to explore the extent to which the FGM adhesives can improve fatigue lives. The idea of FGM also needs to be extended to two- and three-dimensional geometries, instead of only along the thickness direction (Huang *et al.*, 2007b). Also, in reality, tooth to tooth contacts **Fig. 8**(a) are far more complex than the Hertzian contact **Fig. 8**(b). Therefore, curvature effects on contact damage under Hertzian loading need further investigation. Then, the effects of complex loading conditions, such as shear contact loading or normal plus shear loading are to be studied.

Fig. 13 suggests that the pop-in of radial cracks is accompanied by interfacial cracking between the ceramic and adhesive layers. Hence, interfacial cracking should be considered as a possible cracking mode in future work. Also, environmental exposures that are relevant to occlusal conditions need to be explored. In the prior work, a model was developed to understand the mechanism of cracking induced by water absorption in dental multilayered structures (Huang *et al.*, 2007c). The effects of humidity and acidity on the fabricated structure should be examined in the future. Further work is needed to develop physics-based mechanics models that incorporate the effects of water diffusion and contact loading for the prediction of the fatigue lives of dental crowns. Future work is also needed to study SCG in the aqueous environments that are closer to those in the oral cavity, i.e., beyond distilled water.

Though not reviewed in-depth, some prior works already show the possibility of extending the concept of FGM to the design of other dental materials and structures, such as dental implants and tissue-engineered materials. This is also a possible direction for future work.

Summary

This article presents a review of the bio-inspired design of dental multilayer structures. Dental crowns generate a huge market and the need is still increasing due to the steady growth of the aging population. However, the lifetime of currently commercially available dental crowns does not live up to expectations. There is a need to improve the current design of dental crowns.

The structures and properties of hard tissue (enamel and dentin) and their interface DEJ are reviewed. Graded microstructure and mechanical properties exist in enamel, dentin, and their interface, DEJ. Particularly, the DEJ has a complex microstructure that resists crack propagation and a graded Young's modulus distribution which provides a buffer to the stress distribution.

Several commonly used dental materials and their mechanical properties are also reviewed. The models that are used for the research of dental crowns are also introduced. Artificial restoration crowns have a distinguished different feature than natural teeth. For the artificial all-ceramic dental crowns, instead of DEJ, they are bonded to the remaining dentin using a single layer of dental adhesive material that has a Young's modulus much lower than enamel, dentin, and dental ceramic. The mismatch of Young's modulus caused high stress concentration in the sub-surface regime of the top ceramic layer in the dental multilayer structures and hence resulted in the subsurface crack in the top ceramic layer. The failure mode is consistent with the major failure mode of all-ceramic dental crowns that were observed in the clinical environment.

Inspired by the microstructure and mechanical properties of natural teeth, the design of DEJ-like graded dental interfaces was proposed. Numerical simulations show that the FGM can reduce the stress concentration in the structure and reduce the maximum principal stress in the top ceramic layer, which is relevant to the sub-surface radial cracks. The effects of various FGM layer architectures, including the layer thickness and the gradients of Young's modulus, were also studied using simulation.

Using polymer-ceramic nanocomposite materials, the bio-inspired dental FGM structures were actually fabricated. The Young's modulus of the FGM was modulated by controlling the component and fraction of the ceramic fillers. Under Hertzian loading at any clinically relevant loading rate, the critical loads of these structures were tested to be ~20–30% higher than those of the conventional structures with no FGM.

The SCG model for the crack growth in ceramic provided a fracture mechanics theory of the crack growth behavior in the dental multilayer structures. The viscosity of the polymer and composite materials was considered by incorporating the rate-dependent Young's moduli into the SCG model. The predictions provided by the RDEASCG had good agreements with the experimental measurements.

For the potential application of bio-inspired dental FGM structures in dentistry in the future, further work is clearly needed to assess the clinical performance of such structures under cyclic loading conditions and in the oral cavity environment. The FGMs with 2D and 3D architectures also need to be explored. The complex geometries and loading conditions of the real dental

crowns should also be considered. The concept of FGM can potentially be extended to other dental materials and structures, such as dental implants and tissue-engineered structures for better integration of manmade materials and structures with the remaining biological tissues.

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