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## Research Paper

# Creep-assisted slow crack growth in bio-inspired dental multilayers

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## ABSTRACT

Ceramic crown structures under occlusal contact are often idealized as flat multilayered structures that are deformed under Hertzian contact loading. Previous models treated each layer as linear elastic materials and resulted in differences between the measured and predicted critical loads. This paper examines the combined effects of creep (in the adhesive and substrate layers) and creep-assisted slow crack growth (in the ceramic layer) on the contact-induced deformation of bio-inspired, functionally graded multilayer (FGM) structures and the conventional tri-layers. The time-dependent moduli of each of the layers were determined from constant load creep tests. The resulting modulus–time characteristics were modeled using Prony series. These were then incorporated into a finite element model for the computation of stress distributions in the sub-surface regions of the top ceramic layer, in which sub-surface radial cracks, are observed as the clinical failure mode. The time-dependent stresses are incorporated into a slow crack growth (SCG) model that is used to predict the critical loads of the dental multilayers under Hertzian contact loading. The predicted loading rate dependence of the critical loads is shown to be consistent with experimental results. The implications of the results are then discussed for the design of robust dental multilayers.

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## 1. Introduction

The study of the contact loading of dental restorations/crowns is often conducted on idealized, flat multilayered structures, that are deformed under Hertzian contact loading (Kelly, 1997; Lawn et al., 2000, 2004; Lee et al., 2002; Malament

and Socransky, 1999; Rekow and Thompson, 2007; Zhang et al., 2004). This often leads to the onset of clinically-relevant sub-surface radial cracking, due to the high stress concentrations in the sub-surface regions in the top ceramic layer (Huang et al., 2007b; Shrotriya et al., 2003). Such radial cracking is also consistent with the major clinical failure mode reported by Kelly (1997).\*

Abbreviations: DEJ, dento-enamel-junction; FEM, finite element method; FGM, functionally graded multilayer; SCG, slow crack growth; CASCg, creep-assisted slow crack growth

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The opportunity to reduce the stress concentrations at the bottom of the top ceramic layer in dental multilayers was inspired by nanoindentation measurements on the dento-enamel-junction (DEJ) (Marshall et al., 2001). Their studies showed that the Young's modulus varies across the DEJ. Inspired by the gradation in Young's modulus across the DEJ, Huang et al. (2007b) developed a finite element model of the bio-inspired functionally graded multilayer (FGM) of ceramic crown structures. Their results showed that, under Hertzian contact, the FGM structure results in lower stresses in the sub-surface region of the top ceramic layer.

Subsequent work by Niu et al. (2009) and Rahbar and Soboyejo (2011) also showed similar reductions in stress concentrations in the top ceramic crown layer by using FGMs. These were later confirmed by the experimental work by Niu et al. (2009) and Du et al. (2013), who conducted contact loading experiments on actual FGM structures that were fabricated with nanocomposite layers. These had ~20–40% greater critical pop-in loads than flat conventional dental multilayers without FGMs. Also, since the FGM structure has a higher stiffness than the conventional single adhesive layer, it serves as a stress buffer to the top ceramic layer, by dissipating stress from the top ceramic layer into the FGM.

The pop-in loads in the bio-inspired and flat conventional dental multilayers have been predicted using a slow crack growth (SCG) model. This is a power law model that describes the stable crack growth behavior (Wiederhorn, 1974) in the top ceramic layers. It was adopted by researchers (Huang et al., 2007a; Lee et al., 2002; Niu and Soboyejo, 2006; Zhang et al., 2004) to explain subsurface radial crack growth in the top ceramic layers. It was also used to predict the critical pop-in loads in dental multilayers under Hertzian contact loading.

However, although the SCG model predicts the contact damage due to the slow crack growth in the top ceramic layer, it treats the middle adhesive layer and substrate layers as linear elastic materials. Furthermore, there is clear evidence of viscous flow in the substrate and middle layers of dental multilayers (Huang et al., 2007a; Lee et al., 2002; Niu and Soboyejo, 2006; Zhang et al., 2004) that is not accounted for in the conventional dental multilayered structures.

Hence, it has been suggested that the differences between the measured and predicted critical loads (that were obtained from these systems) were due to substrate creep effects (Lee et al., 2002). Furthermore, the differences between critical loads of structures with and without FGM were observed to be greater at slower loading rates than at faster loading rates (Du et al., 2013; Niu et al., 2009). These differences suggest that creep effects should be considered in the modeling of layer properties.

In prior studies, Niu and Soboyejo (2006) modeled the viscoelastic deformation of dental multilayers with rate-dependent elastic moduli. The elastic modulus for specific loading rate was measured from stress–strain data that was obtained from compression tests at particular loading rates. The limitation of this method is that the rate-dependent elastic modulus can only be used in the simulation of the Hertzian contact under the same loading rate. Hence, in order to simulate the Hertzian contact under various loading rates, compression tests have to be performed at each of the loading rates.

In this paper, the viscosity of the each layer in model dental multilayers is measured using creep experiments. The measured viscous behavior is then modeled using Prony series (Bower, 2011). The Prony series fits to the experimental data are compared with those obtained from other models. A creep-assisted slow crack growth model is then developed. The model, which incorporates the combined effects of slow crack growth (in the top ceramic layers) and the viscosity of the adhesive and substrate layers, is used to predict the critical pop-in loads. The implications of the results are then discussed for the design of robust, bio-inspired, dental multilayer structures.

## 2. Materials and experimental methods

### 2.1. Fabrication of bio-inspired FGM dental structures

The bio-inspired FGM structure was fabricated using a steel plate mold with a thickness of ~4 mm and an inner diameter of ~9 mm. The substrate was a dentin-like soft material, Z100 restorative (3M ESPE Dental Products, St. Paul, MN), which is a clinically-used dental material. It was poured into the mold and then cured with UV light. The FGM was produced using nanocomposite mixtures of zirconia or alumina nanoparticles (Nanotek Instrument Inc., Dayton, OH) and an epoxy matrix, EPO-TEK 301 (Epoxy Technology Inc., Billerica, MA). After mixing, the nanocomposite material was deposited in the steel mold and then cured in a vacuum oven at a temperature of 65 °C. The deposition and curing process were then repeated to build up the multilayers.

The crown-like dental ceramic layer on top was fabricated from a medical grade 3 mol% yttria-stabilized zirconia rod (YTZP, Saint-Gobain, Colorado Springs, CO). It was pressed onto the last layer of the FGM before curing. Finally, the multilayered structures, with a diameter of ~9 mm and a thickness of ~5 mm, were cut and removed from the mold. They were then cleaned with distilled water and blow dried with compressed air.

### 2.2. Hertzian contact experiments

Hertzian contact experiments were performed on the fabricated dental multilayers (Fig. 1a). The tests were carried out in an Instron 8872 hydraulic mechanical tester (Instron, Canton, MA, USA). They were conducted in air at room temperature and a relative humidity of ~25%. The Hertzian contact tests were performed under load control with a hemispherical tungsten carbide indenter with a diameter of 20 mm. The tests conducted at clinically relevant loading rates between 1 N/s and 1000 N/s (Du et al., 2013). The loads and displacements were recorded by the computer attached to the Instron tester. The critical loads were then determined as the loads at which discontinuities in displacement were observed. These were also found to correspond to the onset of cracking, which could be heard clearly during the tests.

### 2.3. Creep experiments

The creep specimens were fabricated using the same steel mold that was described above. The nanocomposite materials for

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