DENTAL MATERIALS XXX (2017) XXX–XXX

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Mapping the mechanical gradient of human dentin-enamel-junction at different intratooth locations

*Zhengzhi Wang**, *Kun Wang, Wanyin Xu, Xiaoyu Gong, Feiyu Zhang*

Department of Engineering Mechanics, School of Civil Engineering, Wuhan University, Wuhan, Hubei 430072, China

ARTICLE INFO

Article history:
Received 4 June 2017
Received in revised form
30 October 2017
Accepted 1 November 2017
Available online xxx

Keywords:
Dentin-enamel-junction
Mechanical gradient
Nanoindentation
Nanoscratch
Modulus mapping

ABSTRACT

Objectives. The local structures and mechanical properties within tooth dentin-enamel-junction (DEJ) regions have been focused for numerous studies. The reported results, however, remain inconsistent particularly on the functional width and gradient architecture of the DEJ. The current study aims at systematically determining the mechanical gradient of the DEJ at different intratooth locations such that a clearer understanding on the tooth properties and the potential correlations with the tooth function could be obtained.

Methods. We re-examined how mechanical properties such as elastic modulus and hardness transitioned from those of dentin to those of enamel using combined static and dynamic nanoindentation mapping techniques. A new mapping method and associated image processing procedures were developed to improve the measurement accuracy and resolution.

Results. A thin, sigmoidally-transitioned interphase layer of the DEJ was identified with an accurate functional width of 2–3 μm. The DEJ width and gradient architecture were found intratooth location-dependent, with the DEJ at the occlusal sites being wider and transitioning smoother than that at the cervical sites. Such different widths and architectures of the interphase layer at sites subjected to different types and magnitudes of loadings during mastication could promote more efficient stress transferring between enamel and dentin without compromising the overall stiffness of the tooth.

Significance. The presented study not only adds our understanding in the local mechanical properties within tooth DEJ regions, it could also further advance the development of DEJ-mimetic, functional gradient interphase for strong and ultra-durable jointing between dissimilar materials.

1. Introduction

As one of the most durable biocomposites found in nature, teeth have become a type of representative biomechanical complex that have been studied extensively in the past decades [1–3]. The remarkable damage resistance and structural integrity of the tooth crown have been primarily ascribed to its unique bilayer structure composed of the hard, wear-resistant, and highly-mineralized cover-layer enamel and the softer, tough, and less-mineralized core-layer dentin [4,5]. These two layers exhibiting dissimilar mechanical properties for dissimilar functionalities are delicately united by the so-called dentin-enamel-junction (DEJ) interphase layer [6]. The DEJ is reported as a gradient, transitional interlayer that enables efficient stress transferring between enamel and dentin and crack-arresting for flaws formed in the brittle

* *Corresponding author.*
E-mail address: zhengzhi.wang@whu.edu.cn (Z. Wang).

https://doi.org/10.1016/j.dental.2017.11.001
0109-5641/

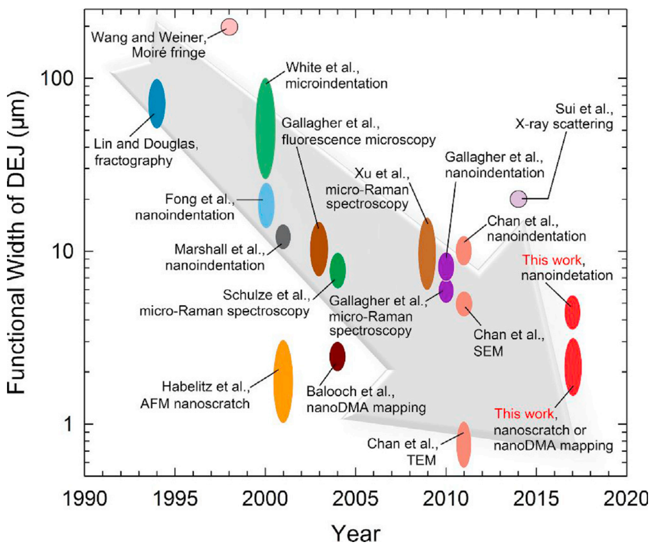

Fig. 1 – Chronological results of the functional width of human tooth DEJ measured in the past 30 years using various measurement techniques [14–26]. The grey arrow indicates the overall decreasing trend of the DEJ width over time.

enamel [7,8]. Therefore, the mechanical quality and properties of the DEJ per se play central roles in determining the performances and longevity of the whole tooth [9,10]. Understanding the structure-property-function relationships of the DEJ has thus long been a focus in the fields of dentistry, biomaterials, and biomechanics.

Despite considerable efforts have been devoted to assessing the local mechanical properties within the DEJ regions, little consensus has been achieved particularly on the functional width (spatial distance that the mechanical properties transition from those of enamel to those of dentin) and gradient architecture (the transitioning pattern of the mechanical properties) of the DEJ. As a result, knowledge of how dentin, enamel, and DEJ are three-dimensionally constructed and how DEJ contributes to the overall properties and functions of tooth still remains to be explored further. Fortunately, such challenging questions have become progressively promising with the advancement of various testing techniques such as the electron microscopy and small-scale mechanical tests including depth-sensing and atomic force microscopy (AFM) nanoindentations [11–13]. As summarized in Fig. 1 for the chronological results of the functional width of human tooth DEJ measured in the past 30 years using various methods [14–26], the overall trend indicates the width decreases as the time evolves mainly due to the improvements in measurement resolution and accuracy. Early studies based on microindentation tests and fractography or Moiré fringe estimations suggested a broad transition zone of ~100–200 μm for the DEJ [14–16]. This width dramatically decreased by over an order of magnitude (i.e. down to ~10–20 μm) with the advent of nanoindentation technique [17,18]. By differentiating the chemical compositions of the tooth dentin, enamel, and DEJ regions using micro-Raman spectroscopy and synchrotron X-ray scattering, the DEJ width was also reported ~10 μm [20,21,23,24,26]. Such techniques as nanoindentation and Raman spectroscopy, however, were still limited in their spatial resolution due to the inherent discrete sampling in tests. As such, continuous mechanical tests with the spatial resolution down to nanometers such as nanoscratch and nanoDMA (nanoscale dynamic mechanical analysis) modulus mapping were later adopted and yielded even smaller DEJ width of 1–3 μm [19,22]. Recently, direct observation of the crystalline structures in the tooth using Transmission Electron Microscope (TEM) gave thus far the smallest DEJ width of 0.5–1 μm [25].

These controversial results of the measured DEJ width have been intuitively explained by that, the higher the spatial resolution of the measurement, the smaller the sampling size and narrower the necessary step width to avoid interferences between adjacent measurements, and thus the less the sample mixing effect and the higher the measurement accuracy [7,24,25]. Also, considering the 3D, undulating, and multi-level scallop-shaped microstructures of the DEJ [6,7,13], the intratooth location-dependency could partly contribute to the huge variations of the measured functional width. However, this dependency has not been systematically investigated. Besides, due to the limitations of the sampling sizes and measurement noises in the nanomechanical tests, little information on the gradient architectures of the mechanical properties (e.g. hardness, elastic modulus, fracture toughness, etc.) across the narrow DEJ has been reported. For simplicity, linear transitions of these properties from dentin to enamel based on limited data points have been widely assumed for purposes of modeling and analyzing [14,16,18,24,27]. Such assumptions need to be validated by more accurate and systematic mechanical mapping tests within the DEJ regions.

To this end, the objective of this study is to systematically determine the mechanical gradients (functional width and gradient architecture) of the DEJ at different intratooth locations such that a deeper understanding on the local properties for dentin-enamel transition could be obtained. With the introduction of a new nanoindentation mapping method and image processing procedures, we are able to map out location-dependent, sigmoidal transitions of the elastic and plastic properties across the DEJ zone. The discrete nanoindentation tests are complemented by continuous nanoscratch and nanoDMA mapping measurements, combination of which provides a full, 3D picture of the mechanical gradient within tooth DEJ. These high-resolution nanomechanical tests yield a latest range of 1.5–6 μm for the function width of DEJ, depending on the measurement method and intratooth location. Such information clarifies the inconsistencies reported in the literature and potentially sheds new light on the tooth property-function correlations.

2. Materials and methods

2.1. *Sample preparations*

Five freshly caries-free extracted human third molars were collected after obtaining the informed consents of donors. Approval to testing these samples was granted by the Ethics Committee for Human Studies of the School & Hospital of