PROBING THE NANOMECHANICAL PROPERTIES OF TEETH

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ABSTRACT

Nanoindentation provides an excellent way of probing and relating the structure and mechanical properties of teeth at the submicron and nanometer scale. For example, a nanoindenter with a high resolution imaging capability can help to elucidate the mechanisms with which certain diseases or even cleansing and bleaching agents can damage or undermine the structural integrity of teeth at the micro- and nanometer scale. This will provide some information on how best to tackle these problems or even preventing it. This paper examines some of the nanoindentation research that has been done on teeth. In particular, we will demonstrate one example on the use of nanoindentation to investigate the effects of bleaching agent on the nanomechanical properties of teeth.

1 INTRODUCTION

A major function of teeth is mastication. This process can give rise to a tremendous amount of force being exerted on the teeth. Also in the process of dietary intake, teeth can be exposed to different types of liquids that may have effects on their mechanical properties and performance (Finke et al. [1]). Certain teeth-related diseases (Marshall et al. [2]) and dental treatment such as bleaching can also affect the mechanical properties as well as the nano and microstructures of teeth. As such, numerous research have been done to examine these effects on the physical properties of teeth.

Like most biological materials, teeth have structure that ranges from the micron to nanometer scale. Therefore an instrument that can probe at such small scale to determine the micro- and nanomechanical properties of teeth is needed. To date, the nanoindenter is one of the most suitable equipment to use. Nanoindentation is a technique that allows the mechanical properties of materials, such as the Young's modulus and hardness, to be determined at the submicron scale. Nanoindentation has already been employed in the study of metals, thin film and nanostructured and graded materials (Baker [3], Fougere et al. [4], Gouldstone et al. [5], Li and Bushan [6]). More recently, this technique has also been used to investigate hard biological tissues such as bones and teeth.

This paper presents some of the research work done on the nanoindentation of teeth. It shows the usefulness of this technique in studying the structure-property-function relationship of teeth at the micron and nanometer scale. In particular, example on the use of nanoindentation to determine the effect of a bleaching agent (30% hydrogen peroxide) on the nanomechanical properties of dentin and enamel is presented.

2 NANOINDENTATION

A nanoindentation system basically comprises a load-sensing piezoelectric transducer, a displacement sensing unit and an indenter tip. This tip is usually made from diamond due to its high elastic modulus and hardness. The geometry of the tip can be sharp such as the Berkovich, Knoop and Vickers tips, or blunt such as the spherical tip.

Figure 1 shows a schematic diagram of a typical plot of load against indentation depth obtained from a nanoindentation test. Figure 2 shows a schematic representation of the indentation process. The depth measured during indentation h is the summation of h_s (displacement due to elastic

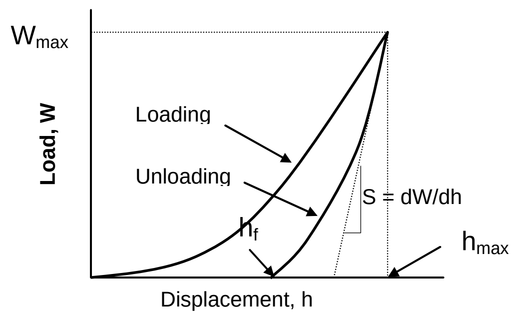

Figure 1: Schematic diagram of a typical load vs indentation depth curve

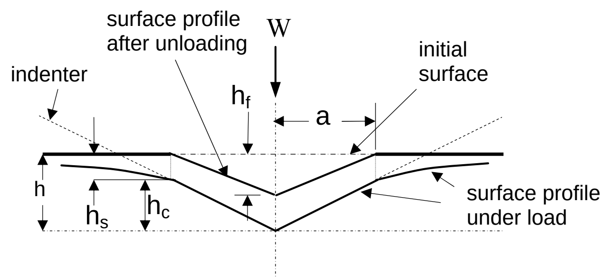

Figure 2: Schematic representation of the indentation process

deformation) and h_c (contact depth or the depth of indenter in contact with the sample under load). h_f represents the final depth of the residual indentation when the indenter is fully withdrawn. S is the contact stiffness (= 1/Compliance) and is the initial slope of the unloading curve (dW/dh). W_{max} and h_{max} are the peak load and displacement, respectively.

The hardness of the sample can be obtained by dividing the load by the projected area of indentation. It is the mean pressure that a material will support under load. From the indentation curve, hardness is obtained at maximum load using

$$\text{Hardness} = \frac{W_{\max}}{A} \tag{1}$$

where A is the contact area at maximum load. The elastic modulus of the sample, E_s is given by Oliver and Pharr [7] as

$$E_s = \frac{1-\nu_s^2}{\frac{2}{S}\sqrt{\frac{\pi}{A}} - \frac{1-\nu_i^2}{E_i}} \tag{2}$$

where E_i is the elastic modulus of the indenter, and ν_s and ν_i are the Poisson's ratios of the sample and indenter, respectively. Eqn. (2) is derived under the assumption that the material is homogeneous and isotropic.

3 NANO AND MICROSTRUCTURES OF TEETH

The tooth comprises mainly the hard enamel, the more ductile dentin and a soft connective tissue, the dental pulp.

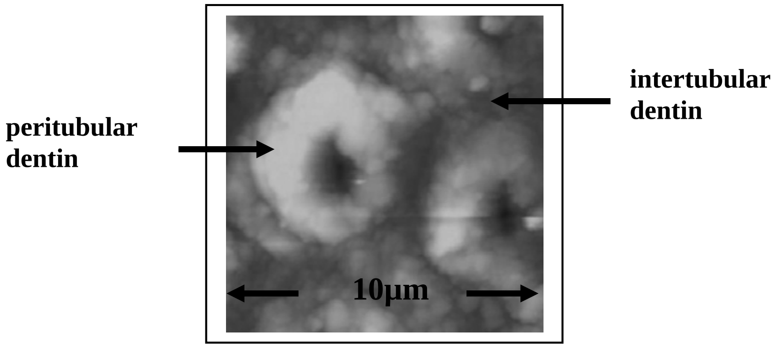

Figure 3: An AFM image of dentin

The enamel forms the crown of the tooth and is the most highly mineralized tissue. It consists of about 96% inorganic material, mainly hydroxyapatite crystallites with traces of organic material enveloping each crystallite. This very high inorganic content makes enamel very susceptible to demineralization in an acid environment created by bacteria and thus to dental caries. The high mineral content of enamel also forms much of its microscopic structure. When enamel is destroyed, it cannot be replaced or regenerated. Due to its exceptionally high mineral content, enamel is a very brittle tissue. In fact, it is so brittle that it cannot withstand the mastication forces without fracture unless it has the support of a more resilient tissue - dentin (Ten Cate et al. [8]).
Dentin forms the bulk of the tooth, supports the enamel and "compensates" for its brittleness. Dentin is a hard, elastic, yellowish white avascular tissue enclosing the central pulp chamber. It is approximately 70% mineralized with hydroxyapatite crystals by weight. Its organic component comprises mainly the fibrous protein collagen. Dentin is a sensitive tissue which is capable of repair as the odontoblasts can be stimulated to deposit more dentin as needed. The dentin is made up of intertubular and peritubular dentin. Kinney et al. [9] found that pertubular dentin is much harder that intertubular dentin. Figure 3 shows an atomic force miscroscopy (AFM) image of dentin.

4 NANOINDENTATION STUDY ON TEETH

4.1. Nanoindentation study of enamel and dentin

Cuy et al. [10] used the nanoindentation technique to investigate the mechanical properties of enamel over the axial cross-section of a maxillary second molar (M^2). They sought to disprove that tooth enamel is a homogeneous solid (Remizov et al. [11]). Their results showed that tooth enamel has varying Young's modulus and hardness throughout its mesial cross-section. Regions with the largest concentration of CaO and P_2O_5 have the highest hardness and Young's modulus. These results suggest that the mechanical properties of hydroxyapatite depend strongly on the degree of mineralization. Habelitz et al. [12] used nanoindentation to examine the mechanical properties of single enamel rods at different orientations at the cusp area. Hardness and Young's modulus values are greater for indents parallel to the rod axis and for indents made in the head area of the rods when compared to tail area and inter-rod enamel. These can be explained by the crystal anisotropy of the apatite crystals that make up the enamel rods.

Kinney et al. [9] used nanoindentation to determine the Young's modulus of intratubular and intertubular dentine and investigate if the values vary with the location of indent. It was found that the average modulus of intratubular dentine was 29.8 ± 8.9 GPa, and was not dependent upon the site within the dentine. It was also found that the decrease in dentine hardness can be attributed to changes in the hardness of intertubular dentine and not to an increase in the number of tubules as previously suggested (Pashley et al. [13]).

4.2. Effects of drinks and agents

4.2.1. Effects of drinks

Finke et al. [1] studied the hardness changes of polished human enamel surfaces after exposure to three different drinks. The three drinks tested were mineral water (pH 6.8), a prototype blackcurrant drink (pH 3.8) and orange juice (pH 3.8). The blackcurrant drink was touted to have low demineralization potential. 250 ml of the drinks were consumed over a 10-min period at 9.00, 11.00, 13.00 and 15.00 h. The high accuracy of nanoindentation made it possible to distinguish the effects between the three different drinks after just one day of intra-oral exposure. This has previously only been possible after at least 15 days of clinical trial (Hughes et al. [17]). There was no statistically significant difference between the hardness and Young's modulus of the area treated with water and the prototype blackcurrant drink. This can be explained by the addition of calcium to the blackcurrant drink. However, the greatest decrease in hardness and Young's modulus occurred in the teeth treated with orange juice.

4.2.2. Effects of cleansing agent

Marshall et al.[14] investigated the effects of $NaOCl_{aq}$ treatment on the microstructural and nanomechanical properties of dentin. $NaOCl_{aq}$ is used as a cleansing and non-specific deproteinizing agent in endodontic treatment. The samples were kept in a liquid cell filled with deionized water during nanoindentation. All samples showed an initial decrease in nanomechanical properties following early treatment with $NaOCl_{aq}$. Initially the indentations were done on the layer of collagen that has experienced demineralization. The nanomechanical properties are reflective of the collagen and underlying dentin surface instead of an isolated collagen layer. After the initial softening, the progressive removal of collagen resulted in a nearly linear increase in hardness and Young's modulus. After complete removal of the collagen, the porous surface had nanomechanical properties that were 70-80% of the values obtained for the unetched portion.

4.2.3. Effects of bleaching agent

Hairul Nizam et al. [15] evaluated the effect of a bleaching agent, 30% hydrogen peroxide, on the nanomechanical properties of dentin and enamel using the nanoindentation technique. Five freshly extracted human premolars for orthodontic reasons were used. The crown was sectioned parallel to the occlusal plane to expose the dentin. Nanoindentation was first done on each of the enamel and dentin regions to determine their mechanical properties prior to treatment. One batch of the tooth samples were then kept in the Hank's balanced salt solution as control while the other was bleached using 30% hydrogen peroxide for 24 hours. The same numbers of nanoindentations were done near the previously indented regions. Comparison of Young's modulus and hardness obtained before and after treatments was made. Using a paired sample t-test with $\alpha = 0.05$, it was found that there were no significant differences in both the Young's modulus and hardness of dentin and enamel kept in the Hank's balanced salt solution. For example, for intertubular dentin, figure 4 compares the Young's modulus while figure 5 compares the hardness of the baseline values and the values after treated with Hank's balanced salt solution. However, the hardness and Young's modulus of dentin and enamel bleached with 30% hydrogen peroxide were significantly decreased. At the intertubular dentin, the hardness decreased by 29% to 55% and the Young's modulus decreased by 19% to 43%. Figure 6 compares the Young's modulus while figure 7

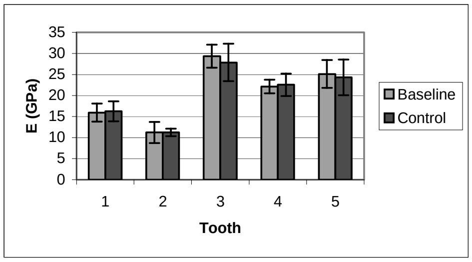

Figure 4: Comparing Young's Modulus of intertubular dentin for both baseline and controlled samples

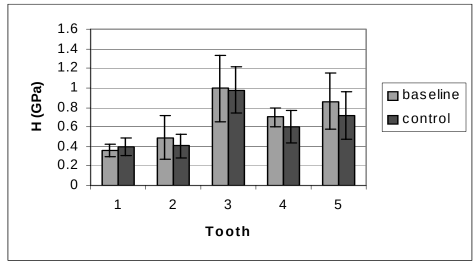

Figure 5: Comparing hardness of intertubular dentin for both baseline and controlled samples

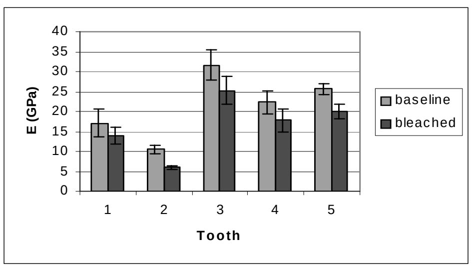

Figure 6: Comparing Young's Modulus of intertubular dentin for both baseline and bleached samples

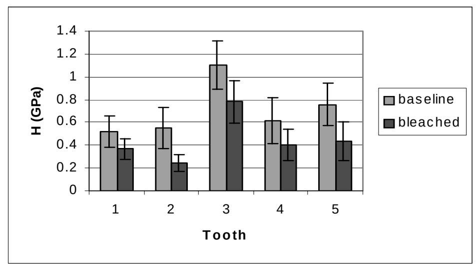

Figure 7: Comparing hardness of intertubular dentin for both baseline and bleached samples

compares the hardness of the baseline values and the values obtained after bleaching. For enamel, the hardness decreased by 13% to 32% while the Young's modulus decreased by 18% to 32%.

The exact mechanism with which hydrogen peroxide affects the dentin and enamel has yet to be fully elucidated. However, the main cause may be due to demineralization of enamel and dentin. Some studies have indicated that hydrogen peroxide caused dissolution of the inorganic material found in tooth. There might also be a reduction in the calcium-phosphorus ratio as well as the organic components of dentin by the process of protein oxidation. All this may result in the decrease in Young's modulus and hardness of dentin and enamel.

Chng et al. [16] also investigated the effect of 30% hydrogen peroxide on the surface changes of intertubular dentine using nanoindentation. It was found that exposure to 30% hydrogen peroxide for 24 hours caused recession of the intertubular dentine surface and significantly decreased the hardness and Young's modulus of intertubular dentine. The effect of hydrogen peroxide on dentine is likely a result of both its strong oxidizing action and its low pH. Peritubular dentine appeared more resistant to the effects of hydrogen peroxide than intertubular dentine and this is likely due to the difference in composition between intertubular and peritubular dentine.

4 CONCLUSIONS

Nanoindentation can provide an excellent way of probing and relating the structures and mechanical properties of teeth at the submicron and nanometer scales. As can be seen from this article, nanoindentation can help to elucidate the mechanisms with which certain diseases or even cleansing and bleaching agents can damage and compromise the structural integrity of teeth. The useful information obtained from these studies can assist researchers in proposing solutions on how best to tackle this problem or even preventing it.

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