

A Comparison of Dental Restorative Materials and Mineralized Dental Tissues for Surface Nanomechanical Properties

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Abstract: Restorations are commonly used to replace the lost or damaged tooth tissues. The aim of this research was to evaluate the surface mechanical properties (hardness and elastic moduli) of mineralized dental tissues. In addition, hardness and elastic moduli of various restorative dental materials have been reported. Freshly extracted maxillary premolars and seven restorative materials were included in the study. All samples were characterized for nanomechanical properties. A minimum of five nanoindentations were performed using Hysitron [TI 725 Ubi] testing instrument. Data was analyzed using the SPSS software (version 20) and t-test was applied measuring the statistical significance. The tooth enamel hardness range was 2.23 to 7.18 GPa being the hardest at cusp tip (absolute hardness of 6.44 ± 0.74). The bulk of dentin exhibited hardness of 0.71 to 0.92 GPa. Porcelain was the hardest material (9.49 ± 0.52 GPa) followed by Co-Cr and Ni-Cr alloys. Poly methyl methacrylate has the lowest hardness (0.18 ± 0.02 GPa) improved hardness for GIC (0.34 ± 0.05 GPa) resin composites (0.54 ± 0.07 GPa) and amalgam (2.55 ± 0.30 GPa). The dental tissues and materials have a wide range of hardness and elastic modulus. The choice of Biomaterials point of view, a single material cannot be used for all mineralized tissues. Clinically, each case (restoration) should be considered on individual bases to evaluate the material of choice.

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1. Introduction

Teeth are made of mineralized hard tissues that perform many vital functions in the oral cavity such as chewing of food, speech and cosmetics. The tooth is comprised of hard tissues (enamel, dentin and cementum) with an inner core of soft and delicate pulp tissues (Nanci, 2012). The mineralized hard tissue of tooth (enamel and dentin) differ from each other in terms of structure and composition however perform as a single functional unit to survive against a variety of forces of mastication (up to ~ 710 N) (Jones, 2001). Enamel is the hardest tissue of the body covering the coronal dentin (Fairpo, 1997; Craig, 2002; Summit et al., 2000; Zafar and Ahmed, 2013). It is a highly mineralized tissue containing 96 wt. % inorganic contents, ~ 3 wt. % organic contents and ~ 1 wt.% water (Craig, 2002; Avery, 1994). The inorganic composition is largely crystalline calcium phosphate in the form of hydroxyapatite [HA] and minute amounts of other minerals such as carbonate, magnesium, strontium, lead and fluoride (Nanci, 2008). The high inorganic contents are responsible for characteristic physical and mechanical properties such as an ability to withstand the heavy forces of mastication, translucency (Craig, 2002; Nanci, 2008; Muhammad et al., 2012a; Muhammad et al., 2012b) and radiopacity for intraoral and extraoral radiographs (Zafar and Javed, 2013).

Enamel is hard but brittle material hence always supported by more elastic tissue, dentin.

Dentin is a distinctive mineralized tissue that is considered vital even there are no blood vessels. Dentin is well innervated and react to the externals stimuli such as tactile, thermal or chemical changes (Summit et al., 2000). Dentin is comparatively less mineralized; inorganics (70 wt. % mainly hydroxyapatite) and 20 wt. % organic contents and 10 wt. % water (Craig, 2002; Summit et al., 2000; Nanci, 2008; Ten Cate, 1994; Linde and Goldberg, 1993; Muhammad et al., 2012c). Dentin forms the bulk of the tooth and has specific properties such as elasticity, pale color, slightly harder than bone (Nanci, 2008; Ten Cate, 1994; Rizvi et al., 2014). The dentino-enamel junction (DEJ) forms the interface between enamel and dentin and plays a vital role in transfer of stresses from enamel to dentin. A widespread terminal branching of dentinal tubules results in the formation of numerous canaliculi and a highly interconnected network system (Ten Cate, 1994; United States. Public Health Service. National Institutes of Health. National Institute of Dental Research., 1958). Cementum is a bone like tissue that covers the root dentin and provides innervation and attachment to the fibers of the periodontal ligament (Jones, 2001; Fairpo, 1997; Nanci, 2008; Gazal et al., 2014).

Teeth or dental tissues may be lost due to highly prevalent diseases (for example caries, periodontal pathologies) or trauma requiring the intervention of restorations using biomaterials. It is

crucial to understand the functional and mechanical properties (such as hardness, elastic modulus) of dental tissues and the dental materials available to replace the lost tissues. Ideally, a very close match of mechanical properties of lost dental tissues and potential dental material is necessary for functional compatibility and longevity of the restoration. The anisotropic nature of the dental tissues has added further complexity resulting in a remarkable variation in mechanical properties from one anatomical site to the other. The aim of this research was to evaluate the surface mechanical properties (hardness and elastic moduli) of mineralized dental tissues at various anatomical positions using nanoindentation. In addition, hardness and elastic moduli of restorative dental materials has been reported.

2. Material and Methods

2.1. Sample preparation

The study included natural teeth and seven restorative materials (Table 1). Human teeth (five maxillary premolars) extracted for orthodontic or periodontal reasons were included in this study. Teeth with carious lesion, wear or any kind of pathology were not included. Hydrogen peroxide solution (1 %) was used for disinfection of teeth (5 °C for a day); followed by storage in plain water at 5 °C until needed for testing.

In order to characterise the dentin and DEJ, teeth were cut transversely into flat disc shape sections using a 2.3 mm diamond disc [standard grit cutting (ISO806104)]. The nomenclature of various

dental hard tissues has been show schematically in figure 1.

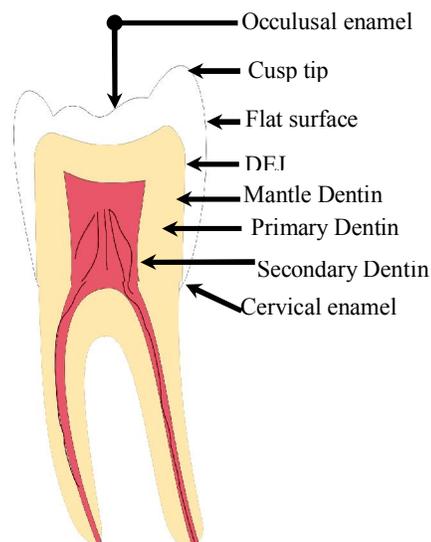


Figure 1: A schematic presentation of tooth section showing mineralised dental tissues

The flat surfaces were finished using motorised silicon carbide discs of different grit sizes (200, 600 and 1200) in a set sequence. Diamond paste of decreasing grits (1.0 and 0.5 micron) was used for fine polishing. Details of restorative materials are given in table 1.

Table 1. Description of dental materials used

Materials	Description	Manufacturer
Human teeth	Maxillary premolars	Freshly extracted
PMMA	BMS 016; Polymethylmethacrylate acrylic resin, Cadmium free	BMS dental Italy
GIC	ChemFil Rock; Capsulated glass ionomers cement (A ₂)	DENTSPLY UK
Composite	Heliomolar; Universal fluoride releasing resin composite (A ₂)	Ivaclor vivadent USA
Amalgam	Megalloy® EZ; High copper spherical capsulated amalgam alloy [alloy to mercury mass ratio=1.3:1]	DENTSPLY UK
Ni-Cr	Wiron® 99; Nickel chrome base metal alloy [Ni (65%); Cr (22.5%); Mo (9.5%)]	BEGO medical Germany
Co-Cr	Wironit® extrahart; Cobalt chrome base metal alloy [Co (63%); Cr (30%); Mo (5%)]	BEGO medical Germany
Porcelain	VITA VM®9; High fusing feldspar ceramic	VIDENT company USA

All materials were manipulated using the manufacturer's instructions and flat disc shapes samples (2 x 6mm) were prepared (n=5). All samples were inspected carefully; any samples with visible crack or voids were discarded. All samples were washed under running distilled water to remove any debris and stored in deionised distilled water at 5 °C until indentation.

1.1. Nanoindentation testing

Nanoindentation was performed using a 3-sided pyramidal Berkovich (142.3 degree diamond probe) fitted in Hysitron [TI 725 Ubi] equipment. In order to prevent any vibration and possible errors, the system was placed on an anti-vibrational table. The prepared samples were installed to the sample holder using epoxy resin. The sample holder was firmly

screwed to the sample table to prevent any movement during the indentation. The indentation was performed using a load of 1N. An optical camera (10X) was used to focus on the exact location of the indentation, after which the machine performs the

indent. A minimum of five indentations with satisfactory loading unloading curves were performed. The representative indentation curves have been shown in figure 2.

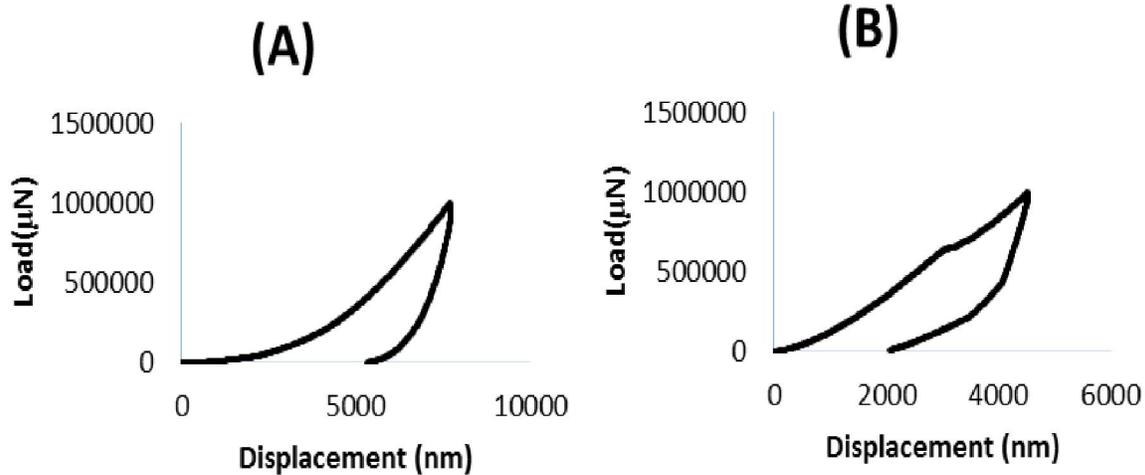


Figure 2: Representative loading and unloading curves produced during nanoindentation testing. (A) Reliable data curves as smooth loading and unloading (B) An example of defective curved suggesting indentation of either an artefact or crack

For data interpretations, force (F) applied and resultant displacement (d) was calculated. The system calculates absolute hardness and reduced modulus (E_r). The reduced modulus can be related with the elastic modulus of the specimen using following equation.

$$\frac{1}{E_r} = \frac{1 - \nu_{sample}^2}{E_{sample}} + \frac{1 - \nu_{indenter}^2}{E_{indenter}}$$

For a standard diamond indenter probe,

$E_{indenter}$ is 1140 GPa and $\nu_{indenter}$ is 0.07.

ν_{sample} has been approximated as 0.3 for the dental hard tissues (Sakaguchi and Powers, 2012) and can be used to calculate the E_{sample} .

The hardness has a nominal definition given by

$$H = \frac{P_{max}}{A}$$

Where P_{max} is the maximum indentation stress and A is subsequent predictable contact area at that load. The medium of indentation was air and longer exposures to air were avoided to prevent the tooth from drying. For an ideal indentation, loading and unloading curve are continuous and smooth (Figure 2 A). However if the indenter falls into a crack or artefact, there will be sharp bends, breaks or step formations in the curve (Figure 2 B). Such faulty

curves may lead to false results and were excluded. Data was analyzed using the SPSS software (version 20) and t-test was applied measuring the statistical significance of values.

3. Results

A range of clinical dental restorative materials and tooth tissues were tested for compatibility in terms of hardness and modulus of elasticity. The absolute hardness values obtained using nanoindentation technique has been presented in figure 3. The hardness of dental hard tissues (enamel and dentine) range between from 0.63 to 7.18 GPa depending on the anatomical areas. The anatomical distribution of various enamel and dentin has been shown schematically in figure 1. The tooth enamel hardness range was 2.23 to 7.18 GPa being the hardest at cusp tip (absolute hardness of 6.44 ± 0.74). The hardness of occlusal surface enamel and cervical enamel was 4.80 ± 0.59 GPa and 4.52 ± 0.50 GPa respectively (Figure 3). The hardness of enamel was observed to reduce from surface towards dentin; near dentin enamel hardness was 2.56 ± 0.33 GPa. Similar trend was observed in dentinal tissues, as hardness was reduced as moving towards pulp, DEJ hardness was 1.24 ± 0.15 GPa and

mantle (first formed) dentin was 1.18 ± 0.19 GPa. The bulk of dentin exhibited hardness of 0.71 to 0.92 GPa.

The dental materials investigated in this study showed a wide range of hardness and modulus of elasticity. Porcelain being a ceramic material was the hardest of all materials (9.49 ± 0.52 GPa) followed by Co-Cr and Ni-Cr alloys having hardness of 7.84 ± 0.51 GPa and 4.56 ± 0.40 respectively. Poly methyl methacrylate (PMMA) has the lowest hardness (0.18 ± 0.02 GPa) and improved hardness for GIC (0.34 ± 0.05 GPa) resin composites (0.54 ± 0.07 GPa) and silver amalgam (2.55 ± 0.30 GPa).

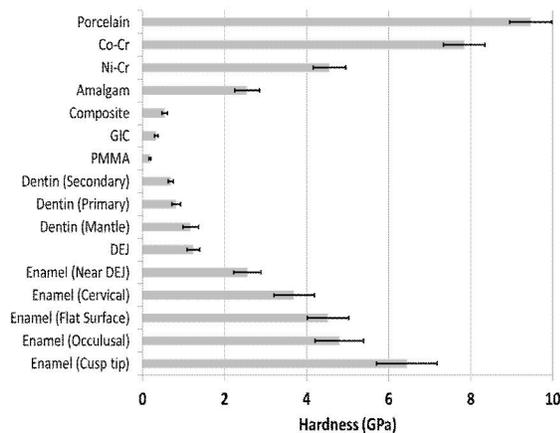


Figure 3: A comparison of nanohardness of dental tissues and a range of dental restorative materials

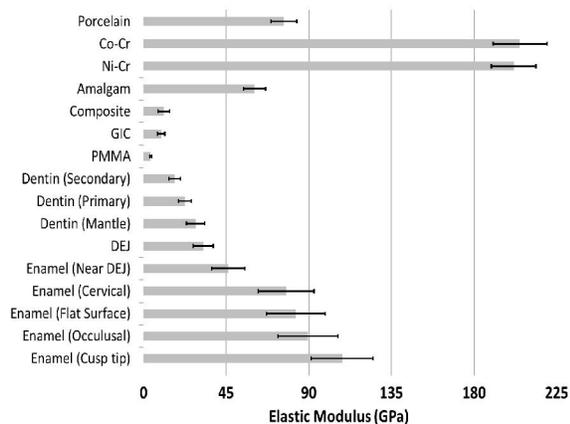


Figure 4: A comparison of elastic moduli of dental tissues and a range of dental restorative

The modulus of elasticity for dental tissues and restorative materials has been shown in figure 4. There was a wide variation in the elastic moduli as well, base metal alloys (both Ni-Cr and Co-Cr) being on the top (190 ± 219 GPa) that was significantly stiffer than any dental tissues as well as restorative

materials ($p=0.05$). The elastic modulus of porcelain (76.45 ± 6.99 GPa) and amalgam (60.44 ± 5.98 GPa) was lower than alloys but significantly ($p=0.05$) higher resin composites (11.16 ± 3.08 GPa), GIC (9.57 ± 2.00 GPa) and PMMA (3.93 ± 0.57 GPa). The elastic modulus of enamel ranged from 72 to 125 GPa depending on the anatomical locations and was significantly higher ($p=0.05$) than dentin tissues (14 to 38 GPa).

4. Discussions

The mean values of hardness and elastic moduli were significantly different among various dental materials as well as enamel and dentin tissues. Nature has adjusted the hardness and elastic moduli depending on the functional requirements of dental tissues. For example, enamel is hardest at cusp tips and occlusal surface, making it wear resistant and suitable to bear the forces of mastication. This achieved by high mineral contents and specialized microstructure such the arrangement of enamel rods. (Mahoney et al., 2000; Malek et al., 2001). Furthermore, the anisotropic nature of enamel composites and fluctuation in mineral contents results in change in hardness at the nanoscale. Enamel and dentin are two different tissues however work together as a single functional unit, for example enamel is hard but brittle and is well supported by underlying more elastic dentin (Nanci, 2012). The interface between these two tissues (scalloped DEJ) also plays a vital role by transmitting heavy forces of mastication the dentin without mechanical failure.

What is the significance of evaluating the hardness and elastic modulus? Ideally, the restorative materials must match hardness and elastic modulus closely to the tissues that is intended to be replaced. Similarly, the interface between formed between the restorative materials and natural tooth is very important. Tooth tissues being natural composites and anisotropic add further complexity to achieve a successful interface. For example, hardness and elastic modulus of dentin varies from one micron to the other, however it may not be possible to mimic in restorative materials. It remains more consistent and reproducible for synthetic materials compared to tooth tissues. A very close match of elastic modulus of dental tissues and restorative materials is required. Otherwise, areas of stress concentration will be produced at the interface of both materials leading to early failure of the restoration. The well-known "sandwich technique" for dental restoration (Knibbs, 1992; Welbury and Murray, 1990) used two different materials (GIC and resin composites) to replace dentin and enamel respectively.

Considering the above discussion, it is clear that understanding hardness and elastic modulus of

restorative materials is very important. This information may help the clinician to choose the restorative materials with the best possible match with dental tissues being replaced. Glass ionomers have the benefit of fluoride release (Zafar, 2013) however the elastic modulus and hardness of GIC and PMMA is significantly lower than dentin and enamel at any location. Resin composites have shown properties comparable to dentinal tissues however remain significantly lower than enamel. Silver amalgam has been used successfully for many years (Phillips, 1957; Hickel et al., 1998) having excellent mechanical properties, longevity and hardness comparable to enamel, however its color remain the major issue for anterior teeth.

The indirect restorative materials (casting metals and porcelains) are harder and stiffer than any direct restorative material (GIC, composite, amalgam). The porcelain and Co-Cr are significantly harder and stiffer than enamel whereas Ni-Cr has comparable properties to enamel. Although these materials have excellent mechanical properties, however there are certain issues. There is a gross mismatch of hardness and elastic modulus compared to enamel and dentin; however interface with the tooth tissue is usually through the luting cements and adhesives. Hardness is simply defined as the resistance to penetrate (indent) in a material. Hardness can influence on the processing such as cutting, finishing and polishing as well as functioning such as mastication. For example, the indirect restorative materials are too hard to finish in the oral cavity. In the laboratory, these materials need more time and efforts for cutting and finishing. Considering clinical functioning, the harder materials will wear the softer materials. For example, porcelain hardness is almost double than enamel and is likely to cause wear of dental tissues of restoration of opposing teeth.

5. Conclusions

Nanoindentation can be used to measure the absolute hardness and elastic modulus of dental hard tissues at micron level. The dental tissues as well as materials have a wide range of hardness and elastic modulus. The choice of Biomaterials point of view, there is no artificial material that can be used for all mineralized tissues. Clinically, each case (restoration) should be considered on individual bases to evaluate what types of tissues have been lost and what kind of material will be ideal for the restoration. This approach can help clinicians to determine the materials of choice [that's having similar properties to the tissues to be replaced] hence preventing restoration failure due to mismatch of mechanical properties.

Conflict of Interest

The authors declared no conflict of interest for conducting this research.

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References

1. Nanci A. Ten Cate's oral histology : development, structure, and function. St. Louis, Mosby ; London: Mosby. 2012
2. Jones FH. Teeth and bones: applications of surface science to dental materials and related biomaterials. Surface Science Reports. 2001; 42(3-5): 75-205.
3. Fairpo JEH. Heinemann dental dictionary. Oxford: Butterworth-Heinemann. 1997
4. Craig JR. Restorative dental materials. St. Louis, Mo. ; London: Mosby. 2002
5. Summit J, Robins W, Hilton T, Schwartz R. Fundamentals of operative dentistry : a contemporary approach. Chicago, Ill. ; London: Quintessence Publishing. 2000
6. Zafar MS, Ahmed N. Nano-Mechanical Evaluation of Dental Hard Tissues Using Indentation Technique. World Applied Sciences Journal. 2013; 28(10): 1393-9.
7. Avery JK. Oral development and histology. New York: Thieme Medical. 1994
8. Nanci A. Ten Cate's oral histology : development, structure, and function. St. Louis, Mosby. ; London: Mosby. 2008
9. Muhammad R, Ahmed N, Roy A, Silberschmidt VV. Turning of advanced alloys with vibrating cutting tool. Solid State Phenomena. 2012; 188: 277-84.
10. Muhammad R, Ahmed N, Shariff YM, Silberschmidt VV. Finite-element analysis of forces in drilling of Ti-alloys at elevated temperature. Solid State Phenomena. 2012; 188: 250-5.
11. Zafar MS, Javed E. Extraoral Radiography: An Alternative to Intraoral Radiography for Endodontic (Root Canal System) Length Determination. European Scientific Journal. 2013; 9(15): 51.
12. Ten Cate AR. Oral histology: development, structure, and function. St. Louis ; London: Mosby. 1994

13. Linde A, Goldberg M. Dentinogenesis. *Critical Reviews in Oral Biology & Medicine*. 1993; 4(5): 679-728.
14. Muhammad R, Ahmed N, Roy A, Silberschmidt VV. Numerical Modelling of Vibration-Assisted Turning of Ti-15333. *Procedia CIRP*. 2012; 1(0): 347-52.
15. Rizvi A, Zafar MS, Farid WM, Gazal G. Assessment of Antimicrobial Efficacy of MTAD, Sodium Hypochlorite, EDTA and Chlorhexidine for Endodontic Applications: An In vitro Study. *Middle-East Journal of Scientific Research*. 2014; 21(2): 353-7.
16. United States. Public Health Service. National Institutes of Health. National Institute of Dental Research. An Electron Microscopic Study of the Early Stages of Dentinogenesis: pp. iv. 55. Washington. 1958
17. Gazal G, Fareed WM, Zafar MS, Al-Samadani KH. Pain and anxiety management for pediatric dental procedures using various combinations of sedative drugs: A review. *Saudi Pharmaceutical Journal*. Accepted 2014.
18. Sakaguchi RL, Powers JM. *Craig's restorative dental materials*. Philadelphia, PA: Elsevier/Mosby. 2012
19. Mahoney E, Holt A, Swain M, Kilpatrick N. The hardness and modulus of elasticity of primary molar teeth: an ultra-micro-indentation study. *J.Dent*. 2000; 28(8): 589-94.
20. Malek S, Darendeliler MA, Swain MV. Physical properties of root cementum: Part I. A new method for 3-dimensional evaluation. *American Journal of Orthodontics and Dentofacial Orthopedics*. 2001; 120(2): 198-208.
21. Knibbs P. The clinical performance of a glass polyalkenoate (glass ionomer) cement used in a 'sandwich' technique with a composite resin to restore Class II cavities. *Br.Dent.J.* 1992; 172(3): 103-7.
22. Welbury RR, Murray JJ. A clinical trial of the glass-ionomer cement-composite resin "sandwich" technique in Class II cavities in permanent premolar and molar teeth. *Quintessence Int*. 1990; 21(6)
23. Zafar MS. Effects of Surface Pre-Reacted Glass Particles on Fluoride Release of Dental Restorative Materials. *World Applied Sciences Journal*. 2013; 28(4): 457-62.
24. Phillips RW. Research on dental amalgam and its application in practice. *Journal of the American Dental Association* (1939). 1957; 54(3): 309-18.
25. Hickel R, Dasch W, Janda R, Tyas M, Anusavice K. New direct restorative materials. *Int.Dent.J.* 1998; 48(1): 3-16.

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