Young's Moduli of Human Tooth Measured using Micro-Indentation Tests

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Abstract: Micro-indentation test results were used to curve fit the Young's moduli of human tooth enamel and dentine in this work. The applied load and unloaded curve portion effects on the measured Young's moduli were investigated. The variation in measured Young's modulus for different applied loads, from 10 *mN* to 500 *mN*, was studied. The experimental results indicate that the measured Young's moduli are very sensitive to the applied load if the load is greater than 100 *mN*. The measured results also reveal that the measured Young's moduli were dependent upon the unloaded curve data portion to be curve fitted. The large portion of the unloaded curve data could be used, i.e. 90% of unloaded curve data, to curve fit a smaller Young's modulus value.

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1. Introduction

The mechanical properties of materials, i.e. Young's modulus, hardness, are very important for biomechanical engineering research. The knowledge of tooth mechanical properties plays a key role in predicting the mechanical behavior and helps clinicians to understand the tooth stress distribution under different bite loads. However, because of the small size of human teeth, it is difficult to measure tooth material properties using traditional methods. In the last decade, micro-depth sensing indentation tests have been used to obtain the Young's modulus of teeth[1-9]. It is assumed that the variation in the load and unloading curve in the micro-scale indentation test is dominated by the material properties[10, 11]. Linear elastic behavior occurs particularly during the unloading period. The portion of the unloaded part of the load-indentation depth curve data is used to derive the Young's modulus of the tested specimen. The slope at the initial unloading point in load-indentation depth curve is derived to approximate the Young's modulus of the tested specimen[11, 12]. However, different portions of the unloaded curve may affect the derived slope in this method. Finding a proper portion of the unloaded curve to derive a reliable Young's modulus in the micro-indentation test has always been discussed. Besides this problem, the applied load and the loading position may also have significant effects on the measured data. As noted, based on the hardness and density, the tooth structure is considered as two distinct portions, enamel and dentine. In these two parts, the tooth structures are porous and inhomogeneous. This porous structure may introduce significant variation in the measured load and penetration depth data. In other words, a wide variation in the measured Young's modulus values is expected when using this method.

This work examines the maximum applied load and load position effects on the variation in measured Young's moduli in the enamel and dentine portions of human teeth.

2. Load-indentation depth curves

The micro-indentation test technique has been widely used to measure the surface properties for a long time. The penetration deformation and its spring back behaviors during the loading and unloading processes in the indentation test have been used to extract the corresponding material elastic and plastic properties. The material behavior at initial unload is considered as an elastic recovery in essence. The load-indentation depth curve slope at the initial unloading can be derived the Young's modulus of the tested specimen[11]. Referring to the load-indentation depth curve, as shown in Figure 1, a stiffness at the initial unload can be expressed as,

$$S = \left.\frac{dP}{dh}\right|_{h=h_{\max}} \qquad (1)$$

where S, P, and h denote stiffness, load, and indentation depth, respectively.

Based upon Sneddon's analysis[13], the load (P) -indentation depth (h) relationship for a rigid cylinder indenter can be approximated as,

$$P = \left(\frac{4GR}{1-\nu}\right)h \qquad (2)$$

where R, G, and ν are the cylinder radius, shear modulus and Poisson's ratio, respectively. The circular contact area is πR^2, and elastic modulus E can be in terms of the shear modulus and Poisson's ratio as $E=2G(1+\nu)$, differentiating P with respect to h, it leads to

$$S=\frac{dP}{dh}=\frac{2}{\sqrt{\pi}}\frac{E}{(1-\nu^2)}\sqrt{A} \qquad (3)$$

In a real case the indenter cannot be perfectly rigid. The deformation of the indenter therefore contributes to the measured displacement. It is convenient to define a reduced modulus E_r[14],

$$\frac{1}{E_r}=\frac{(1-\nu^2)}{E}+\frac{(1-\nu_i^2)}{E_i} \qquad (4)$$

where E and ν are the tested specimen's elastic modulus and Poisson's ratio, respectively, E_i and ν_i are the indenter's elastic modulus and Poisson's ratio, respectively. Equation (3) can be rewritten as

$$S=\frac{dP}{dh}=\frac{2}{\sqrt{\pi}}\sqrt{A}E_r \qquad (5)$$

Therefore, providing a reasonable estimate of the Poisson's ratio and contact area, the specimen's modulus can be computed from the initial unloading slope.

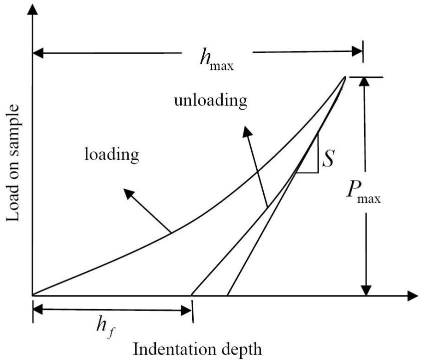


Figure 1. Load-indentation depth curve of the micro-indentation test.

3. Micro-indentation tests

3.1 System of micro-indentation tests

A Nano Indenter XP, produced by the MTS System Corporation, was used in this work. The fundamental characteristics of the system are listed as below,

1. Resolution of indenter displacement: 0.1 nm
2. Range of indentation depth: 25 nm ~ 500 μm
3. Maximum applied load: 0.5 N
4. Resolution of load: less than 500 mN
5. Linear loading controlling system

3.2 Specimen preparation

A human molar tooth aged 60 years old was chosen to make the specimen. The specimen was embedded with a cold-curing epoxy resin and its interior sectional surface was chosen to perform the indentation tests. The specimen's surface, as shown in Figure 2, was hand-ground polished using wet silicon carbide papers of #80, #800, #1200, and #2500 grit sizes progressively. Fine polishing was achieved on a rotary polishing machine using 0.1 μm-size aluminum oxide suspensions. The specimen was stored in dry conditions at room temperature before testing.

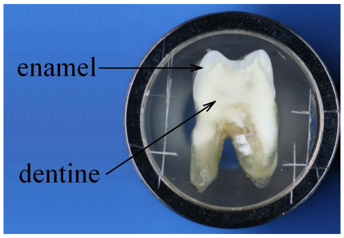


Figure 2. Specimen for micro-indentation tests.

3.3 Experiment setup

The micro-indentation tests in this study were performed using a calibrated Berkovich indenter. To investigate the applied load effect on the variation in molar tooth Young's modulus measured using the micro-indentation tests, six groups of indentation tests were carried out with maximum applied loads of 10, 30, 50, 100, 200, and 500 *mN*, respectively. A total of 12 groups were studied for measuring both the enamel and dentine moduli. In order to observe the variation in measured data, an 8 x 8 indent matrix, as shown in Figure 3, was measured for each test group. The loading, holding and unloading time periods for each indention test were controlled at 15, 30, and 15 seconds, respectively. The allowable drift rate was less than 0.5 nm/s.

Figure 3. Residual indents of one group micro-indentation tests.

To study the possible adopted portion of the unloaded curve effect on the measured Young's modulus, different percentages of unloaded data of the load-indentation depth curve were used to derive the slope. Five groups of test data, i.e. 20%, 30%, 50%, 70%, and 90% of the unloaded data, were chosen to generate the corresponding Young's moduli.

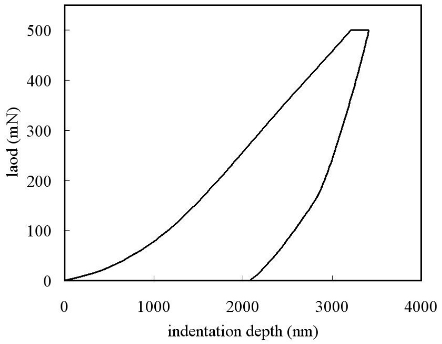


Figure 4. The measured load-indentation depth cycle on enamel part.

4. Results and discussions

Figure 4 shows the variation in load and indentation depth on the enamel portion in a loading cycle. As mentioned in the previous section, due to the porosity of teeth, some measured data are unreasonable. Eliminating these unavailable data, there are more than 50 acceptable results in the 8 x 8 test data collected. These available load-indentation depth curves were used to derive the corresponding Young's modulus values. Figures 5 and 6 show the variation in the obtained moduli with different maximum loads for the enamel and dentine parts of the tested tooth. The modulus values were derived using the top 50% unloaded curve data. Some data were distributed unlike a Gaussian distribution. The key reason for this wide spread distribution may come from the inhomogeneous and porous tooth structures. In the micro-indentation test, the indent size was just a few micro-meters. It is possibly affected by the local porosity, leading to a non-Gaussian distribution data.

The average value and the corresponding standard deviation of the derived Young's modulus are listed in Table 1. Some distribution cases show no obvious central peak at the average modulus, however, the standard deviation is small and most deviation to average modulus ratios listed in Table 1 are less than 10%. Nevertheless, the results in this study present extracting enamel and dentine Young's modulus using micro-indentation tests can still obtain quite narrow-band distributed data.

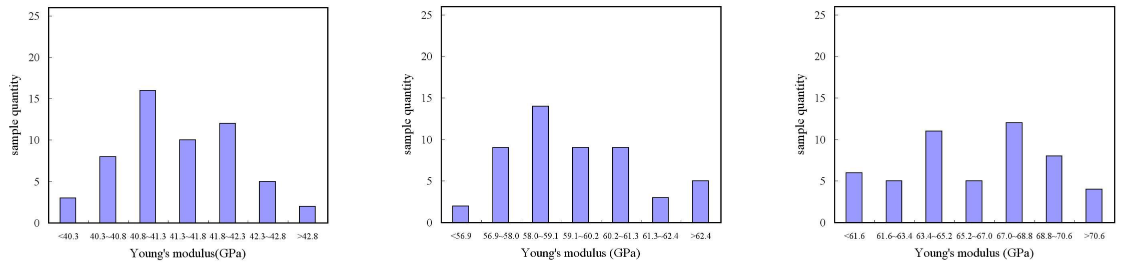
Figure 7 presents the enamel and dentine average moduli which vary with different percentages i.e. 20%, 30%, 50%, 70%, and 90% of the adopted unloaded data. The results reveal that the derived modulus decreases with the increase in adopted unloaded curve data in most cases. The largest modulus value is derived with 20% of the adopted unloaded data. In general, the percentage of adopted unloaded data is not sensitive to the derived results. Similar results were observed for the dentine cases. A greater applied load may derive a smaller Young's modulus value. The ratio of the modulus at 20% to the one at 90% is between 103.8% ~ 106.2% for the dentine cases. Based on the measured results, the unloaded data in the 20% to 50% range from the initial unloaded data is suggested for deriving the Young's moduli values.

The variation in average Young's modulus with applied load is shown in Figure 8. For dentine, the mean value of the derived Young's modulus decreases from 20.7 GPa to 16.2 GPa as the applied load is increased from 30 mN to 500 mN. A difference of 21% is introduced. Similarly, the mean value of the derived Young's modulus for enamel decreases from 75.3 GPa to 41.5 GPa as the applied load is increased from 10 mN to 500 mN. The difference is extended to 45% in this case. The measured results reveal that the derived Young's modulus is very sensitive to the applied load in the micro-indentation test of tooth specimen.

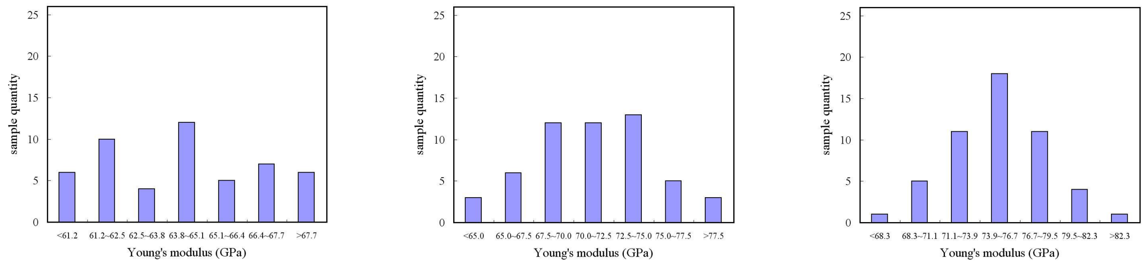
A comparison between the published values for enamel and dentine Young's moduli[2, 3, 5-7, 15] and the results derived in this study is listed in Table 2. Figure 9 shows the variation in published moduli and results derived in this work. All of the results indicate that the derived Young' modulus values relate significantly to the applied load. To eliminate the local porosity effect, a lower applied load 10 *mN* is suggested to measure the tooth Young's modulus using the micro-indentation method. The derived mean values for enamel and dentine are then 75.3 and 19.6 *GPa*, respectively.

Table 1. The average moduli and its standard deviation for the enamel and dentine portions.

max. load (*mN*)	enamel			dentine		
	Young's moulus E (*GPa*)	standard deviation D (*GPa*)	ratio D/E	Young's moulus E (*GPa*)	standard deviation D (*GPa*)	ratio D/E
10	75.3	3.6	4.8%	19.6	2.7	13.8%
30	71.2	3.9	5.4%	20.7	1.4	6.6%
50	64.3	2.7	4.1%	20.7	0.9	4.4%
100	65.4	3.3	5.0%	20.0	0.7	3.6%
200	59.6	1.8	3.0%	17.6	1.6	9.3%
500	41.5	0.8	1.8%	16.2	0.5	3.1%

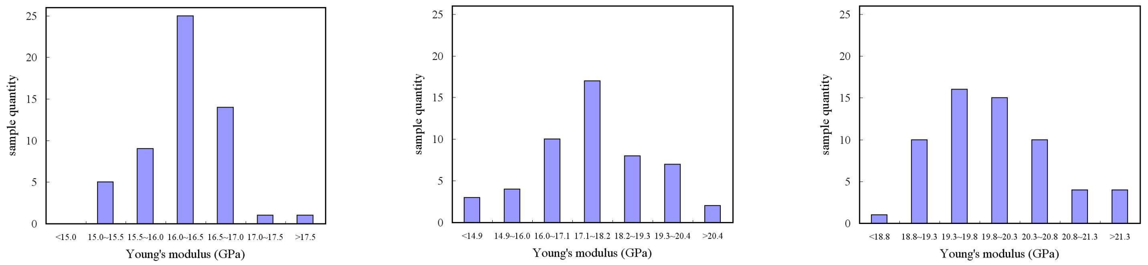


(a) max. load: 500 *mN* (b) max. load: 200 *mN* (c) max. load: 100 *mN*

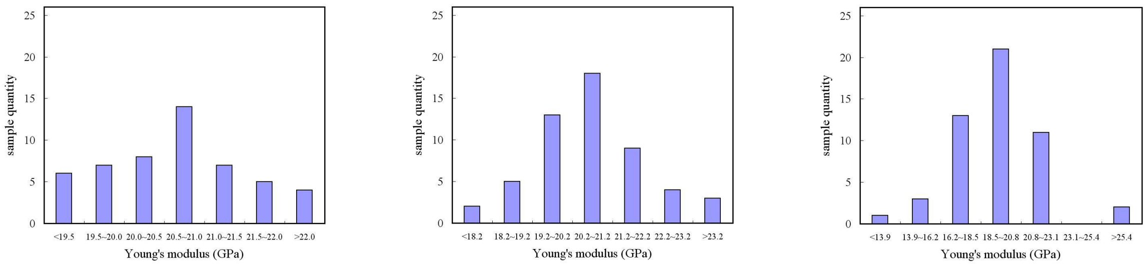


(d) max. load: 50 *mN* (e) max. load: 30 *mN* (f) max. load: 10 *mN*

Figure 5. The variation in Young's moduli in the tooth enamel portion with different loads.

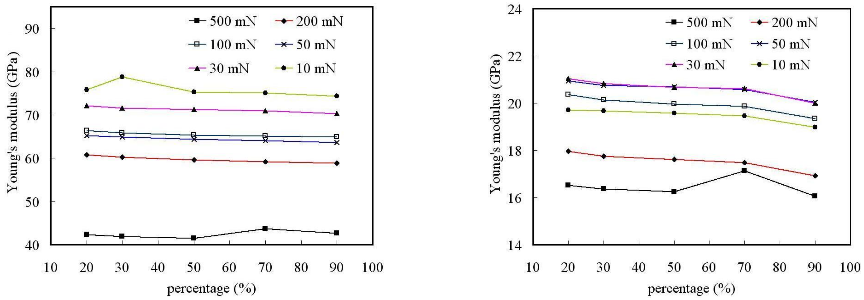


(a) max. load: 500 *mN* (b) max. load: 200 *mN* (c) max. load: 100 *mN*



(d) max. load: 50 *mN* (e) max. load: 30 *mN* (f) max. load: 10 *mN*

Figure 6. The variation in Young's moduli in the tooth dentine portion with different loads.



(a) enamel (b) dentine

Figure 7. The derived Young's modulus values with different percentages adopted using unloaded curve data.

Table 2. Literature values for enamel and dentine moduli compared with the results in this work.

enamel			dentine		
	applied load (*mN*)	Young's modulus (*GPa*)		applied load (*mN*)	Young's modulus (*GPa*)
Mahoney *et al.* [2]	50	74.5 ~ 99.9	Mahoney *et al.* [2]	50	18.2 ~ 24.0
Mahoney *et al.* [2]	150	73.3 ~ 95.2	Mahoney *et al.* [2]	150	16.4 ~ 22.5
Marshall *et al.* [3]	30	62.1 ~ 65.0	Marshall *et al.* [3]	30	18.6 ~ 20.7
Balloch *et al.* [5]	0.0025	60.0 ~ 70.0	Balloch *et al.* [5]	0.0025	17.0 ~ 23.0
He *et al.* [15]	10	115*	Hosoya *et al.* [6]	10	21.0 ~ 27.7
He *et al.* [15]	250	95*	Hosoya *et al.* [7]	3	22.7 ~ 29.2
This work (50 indents)	10	64.9 ~ 84.5	This work (50 indents)	10	13.3 ~ 29.3
This work (50 indents)	500	39.5 ~ 43.3	This work (50 indents)	500	15.1 ~ 18.3

* Literature providing the mean values only.

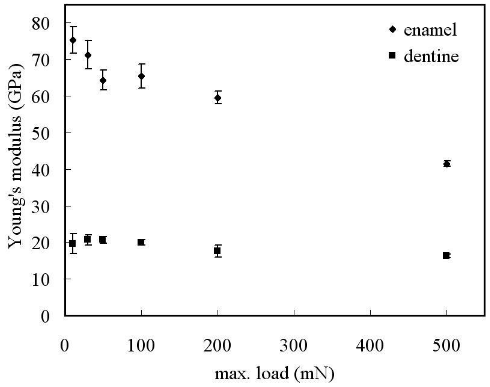
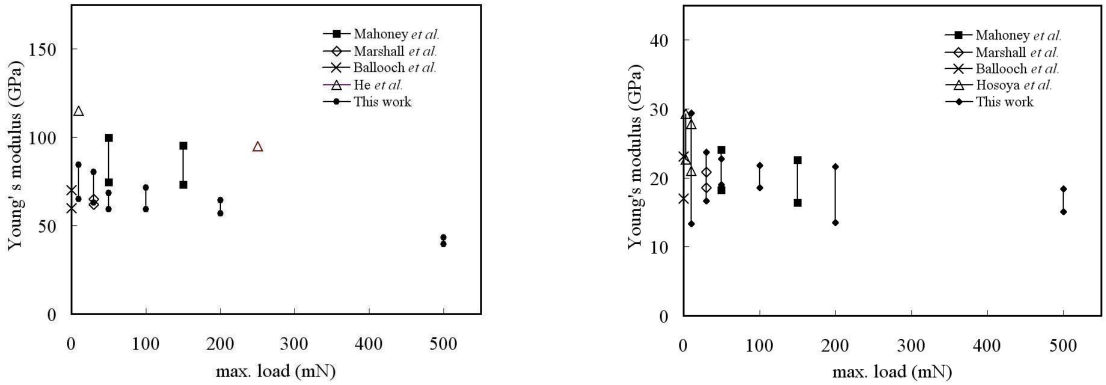


Figure 8. The variation in derived Young's modulus for a tooth with different micro-indentation test applied loads.



(a) enamel (b) dentine

Figure 9. The variation in published Young's modulus with the applied loads.

5. Conclusions

This study presented the adopted unloaded data percentage and applied load effects on the derived Young's modulus values for a studied tooth. The results reveal that the percentage of adopted unloaded data is not sensitive to the derived Young's modulus for the tooth. The unloaded data in the 20% to 50% range from the initial unloaded data is suggested for deriving the Young's moduli values. However, the measured results also indicated that the derived Young's modulus is very sensitive to the applied load in the micro-indentation test. To eliminate the local porosity effect a lower applied load, 10 *mN*, is suggested. Because of the local porosity and inhomogeneous tooth structure, a wide variation in measured Young's moduli was found for enamel and dentine in this study. For safety considerations, a conservative estimate of the Young's modulus value in tooth mechanics analysis is suggested.

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