Effect of bracket bonding on nanomechanical properties of enamel

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Introduction: In this study, we investigated the nanohardness and elastic modulus of enamel after debonding metal brackets. Methods: The surfaces of 3 maxillary premolars were subdivided into 3 regions. Two regions were exposed to a conventional etching system (Transbond XT, 3M Unitek) and a self-etching system (Transbond Plus primer, 3M Unitek); the third region was not etched. Metal brackets were bonded with Transbond XT composite to the 2 etched regions. After storage for 24 hours in distilled water, the brackets and residual adhesive were removed, and the teeth were sectioned transversely. Seven nanoindentations (2 mN load) were placed 1 to 25 µm from the surface in each region. Mean nanohardness and elastic modulus were compared with analysis of variance (ANOVA) and the Scheffé test. Results: Locations 1 and 5 µm from the enamel surface had significantly (P < 0.05) lower nanohardness and elastic modulus values for the conventional system compared with the self-etching system and the unetched region. All other locations for the conventional system and all locations for the self-etching system and unetched area had no significant differences. The nanohardness was much higher than the Vickers hardness for enamel. Conclusions: The minimal effect of the self-etching system on the nanomechanical properties of enamel arises from much lower chemical attack. The much greater effects of the conventional system require further study. (Am J Orthod Dentofacial Orthop 2010;138:735-40)

After acid etching was introduced in the mid-1950s, the direct bonding of orthodontic appliances to enamel with epoxy resin was introduced by Newman in the mid-1960s and is now widely accepted by most orthodontists. Bonding practices based on a self-etching primer, which reduces clinical bonding steps by combining etching and priming into 1 step, are now being used in clinical orthodontics. In addition to saving time and reducing procedural errors, their lower etching ability from higher pH compared with phosphoric acid, might minimize the potential for iatrogenic damage to enamel. The mechanical properties of the etched enamel surface should be affected by the degree of demineralization and penetration of the resin tags by bracket bonding. Knowledge of the change in mechanical properties of enamel after debonding brackets is important in understanding iatrogenic damage, such as enamel fracture. Recently, the nanoindentation test has become a common technique for analyzing mechanical properties such as hardness and elastic modulus for small areas and thin regions of materials.

The purpose of this study was to use the nanoindentation test to examine the effects of conventional etching and self-etching on the nanomechanical properties (hardness and elastic modulus) of the enamel surface region. It was hypothesized that self-etching would cause less degradation of these properties than conventional etching. This is the first study that has investigated the nanomechanical properties of enamel after bracket bonding with conventional etching and self-etching systems.

MATERIAL AND METHODS

Three noncarious maxillary premolars were used in this study. The teeth had been extracted for orthodontic
reasons and with the patients’ informed consent. Selection criteria included no visible decalcification or cracking of the enamel surface under a stereoscopic microscope (SMZ 1500, Nikon, Tokyo, Japan) at magnification of 10 times. The extracted teeth were stored in 0.5% chloramine solution at approximately 4°C. The buccal surfaces of the teeth were cleaned by using nonfluoridated pumice. The teeth were subsequently polished by using a rubber cup and thoroughly washed and dried with a moisture-free air source.

The buccal surfaces of the 3 premolars were divided into mesial and distal regions with masking tape (approximately 1 mm width) (Fig 1, a). The mesial region was etched with conventional 35% phosphoric acid gel (Transbond XT Etching Gel, 3M Unitek, Monrovia, Calif) for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Transbond XT primer (3M Unitek) was then applied to the etched surface. Transbond Plus self-etching primer (3M Unitek) was next applied to the distal region by rubbing on the enamel surfaces for approximately 3 seconds; afterward, an air jet was lightly applied to the enamel. After the masking tape was removed, metal brackets (Victory Series, 3M Unitek) were bonded with Transbond XT composite resin (3M Unitek) (Fig 1, c) and light-cured for 20 seconds (10 seconds from each proximal side). After storage for 24 hours in distilled water, the metal brackets were debonded, and the residual adhesive was removed by grinding with a low-speed tungsten carbide bur (CA1172, lot 2710291, Shofu, Kyoto, Japan), followed by polishing with nonfluoridated pumice. All teeth were cut with a slow-speed water-cooled diamond saw (Isomet, Buehler, Lake Bluff, Ill) so that they were divided into occlusal and cervical halves (Fig 1, f); 1 sectioned specimen (transverse planes) was then encapsulated in epoxy resin (Epofix, Struers, Copenhagen, Denmark) for the nanoindentation test. All samples were ground with 600-grit sandpaper and polished by using diamond suspensions (3, 1, and 0.25 μm particle size) to obtain a suitably polished surface.

Nanoindentation testing was carried out at 28°C with a nanoindentation system (ENT-1100a, Elionix, Tokyo, Japan) by using a 2-milliNewton (mN) load. The indentations were placed at 49 locations spaced 4 mm apart (7 × 7 locations; 1-25 μm depth from the surface) for each of the 3 regions (control, etching, and self-etching regions) on each tooth (Fig 1, h). Nanohardness and elastic modulus were calculated by using the equations in ISO standard 14577.18

Statistical analysis

Statistical comparisons were performed with SPSS software for Windows (version 16.0J, SPSS, Chicago, Ill). Mean values of nanohardness and elastic modulus were compared by using 1-way analysis of variance (ANOVA) and the Scheffé multiple range test, with a significance level of 5%.

RESULTS

Mean values and standard deviations for nanohardness and elastic modulus of the polished enamel specimens (transverse planes) are shown in Figures 2 and 3.
respectively. Locations at 1 and 5 μm from the enamel surface for the conventional etch-and-rinse system had significantly lower nanohardness and elastic modulus values than did other locations. All mean values of nanohardness and elastic modulus obtained for the self-etching system were not significantly different.
from those for the control specimen. The nanohardness and elastic modulus values were correlated.

**DISCUSSION**

Iatrogenic damage to enamel, such as fractures and creation of other surface defects during debonding of brackets, is a serious concern to clinicians.19 The enamel fracture during debonding for the early ceramic brackets was the result of a silane coupling agent used to establish chemical bonding between the bracket base and the adhesive resin.20,21 Although recent improvements in bracket engineering, debonding methods, and debonding instruments have been made, enamel damage during debonding is still a matter of concern in clinical orthodontics.22-24 On the other hand, enamel color alteration in clinical orthodontics is sometimes observed after debonding of brackets and is probably caused by retained resin tags in the enamel or enamel damage during debonding.25,26 The mechanical properties of the etched enamel surface should be affected by the degree of demineralization and penetration of the resin tags during bracket bonding. Measurement of changes in near-surface mechanical properties of enamel after debonding of brackets is important in understanding the iatrogenic damage caused by fracture during debonding and discoloration from retained resin tags.

In conventional microindentation methods, such as the Vickers and Knoop hardness tests, a diamond indenter is pressed into the sample by using a known load. After some dwell time in the test sample for the indenter, the area of the residual permanent indentation in the sample is measured, and the hardness is defined as the maximum load divided by the permanent indentation area. The original idea of nanoindentation arose from the realization that an indentation test is an excellent way to measure mechanical properties of small volumes of materials.14-17 The load and the displacement of the nanoindenteter are recorded during the indentation process, and these data are analyzed to obtain the contact area, and thereby the mechanical properties, without having to actually observe the nanoindentations. The term _nanoindentation_ refers to depth-sensing indentation testing in the submicron range, in which small indentations are made while the load and displacement are recorded with high accuracy and precision. These load-displacement data can be interpreted with appropriate mathematical relationships to obtain values of hardness, elastic modulus, and other mechanical properties, with the aid of suitable software.

In this study, the enamel at locations 1 and 5 μm from the surface bonded with the conventional etch-and-rinse system had significantly lower nanohardness and elastic modulus values than for the unetched control and the self-etching system, suggesting that the mechanical properties of the enamel surface decreased after bracket bonding with the conventional etch-and-rinse system. In contrast, the self-etching system did not decrease the nanohardness and elastic modulus values of the enamel surface as a result of bracket bonding, probably because of the lower etching ability of this system.

Since the hardness and elastic modulus are greatly affected by the organic components16,27 and the orientation of the rods in the enamel,27 similar locations were chosen for the nanoindentation test with the 3 experimental conditions (control, conventional etch-and-rinse system, and self-etching system) in this study. Figures 2 and 3 show that the nanohardness and elastic modulus values were correlated. However, a recent study reported the microhardness and fracture toughness of different components of rat incisor enamel and found that the fracture toughness and hardness values were inversely correlated.27 The Table summarizes recent published data for the hardness and mechanical properties of enamel that were obtained by indentation methods.16,27-33

<table>
<thead>
<tr>
<th>Test method</th>
<th>F&lt;sub&gt;max&lt;/sub&gt; or h&lt;sub&gt;max&lt;/sub&gt;</th>
<th>Hardness (GPa)</th>
<th>Elastic modulus (GPa)</th>
<th>Investigators</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nanoindentation</td>
<td>2 mN</td>
<td>7.4-8.3</td>
<td>113-127</td>
<td>This study</td>
</tr>
<tr>
<td>Nanoindentation</td>
<td>~200 nm</td>
<td>6-7</td>
<td>120-130</td>
<td>Braly et al&lt;sup&gt;31&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanoindentation</td>
<td>20 mN</td>
<td>2.9-5.4</td>
<td>61-90</td>
<td>Mahoney et al&lt;sup&gt;30&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanoindentation</td>
<td>10 mN</td>
<td>3.4</td>
<td>91</td>
<td>Willems et al&lt;sup&gt;28&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanoindentation</td>
<td>100-2000 nm</td>
<td>3.6-5.7</td>
<td>70-104</td>
<td>Zhou and Huang&lt;sup&gt;29&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanoindentation</td>
<td>10-250 mN</td>
<td>~4.5</td>
<td>95-115</td>
<td>He and Swain&lt;sup&gt;16&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vickers</td>
<td>0.98 N</td>
<td>2.6-2.7</td>
<td>–</td>
<td>Wongkhanne et al&lt;sup&gt;31&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vickers</td>
<td>2.94 N</td>
<td>2.8</td>
<td>–</td>
<td>Sakar-Deliormanli and Guden&lt;sup&gt;32&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vickers</td>
<td>0.98 N</td>
<td>3.2-3.4</td>
<td>–</td>
<td>Baldassarri et al&lt;sup&gt;27&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vickers</td>
<td>2.50 N</td>
<td>2.8-3.7</td>
<td>–</td>
<td>Xu et al&lt;sup&gt;14&lt;/sup&gt;</td>
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_F<sub>max</sub>_ Test load; _h<sub>max</sub>_ maximum depth of indentation.
hardness obtained with the nanoindenter (3.4-8.3 GPa) were usually greater than those obtained with the Vickers hardness test (Table), which samples a much larger microvolume of enamel. This result is consistent with a recent article on the effect of indentation depth on the mechanical properties of enamel; it reported that both the hardness and the elastic modulus continuously decreased with increasing penetration depth. These changes were attributed to continual variations in the enamel structure that is being probed by the nanoindenter with increasing depth below the surface. Moreover, variations in the enamel structures of premolars in individual patients with varying caries risks have been detected by small-angle x-ray scattering. Such variations are consistent with our observations of differences in hardness and elastic modulus detected by the nanoindenter for the premolars from 3 patients. More fundamental understanding of the relationships between near-surface and bulk properties of etched enamel and the variations in enamel among patients is needed. Such knowledge is essential for interpreting the results of future research in which near-surface values of enamel fracture toughness are compared with bulk values after debonding brackets.

CONCLUSIONS

Under the conditions of this study, the following conclusions were drawn.

1. The hardness and elastic modulus of the enamel surface region should be decreased after bracket bonding with the conventional etch-and-rinse adhesive system.
2. The self-etching adhesive system causes minimal decreases in the hardness and elastic modulus values of the enamel surface as a result of bracket bonding.
3. The values of hardness of enamel obtained with the nanoindenter in this study were much greater than the reported Vickers hardness of enamel.

REFERENCES


