Acid etching of dental enamel is a standard practice to ensure good bonding between resins and dental enamel during many clinical procedures on tooth. This acid etching process creates a rough surface (an increase in the surface area) to enhance mechanical bonding and wettability of the resin. However, the enamel surface becomes weaker after etching, and care needs to be taken to minimize any mechanical damage during the clinical procedure. In this study, we characterize the nano/micro-scale friction and wear behavior of the dental enamel – pre- and post-etching – using the Keysight Technologies, Inc. G200 nanoindenter. A nanoindenter is suitable in this context to realistically simulate the forces that these enamel surfaces experience during the clinical procedures.

Two similar dental enamel samples were mounted in epoxy and polished to obtain a smooth flat surface for nanomechanical characterization. One of the samples was then etched with an acid solution for 10 minutes. Nanomechanical scratch tests in the G200 were carried out using a diamond conical indenter with 90° included angle and 2 µm tip radius. Constant-load scratches were made with different normal forces on both samples. Figure 1 shows the displacement profiles before and during the constant-load scratch tests. By comparing the pre-scratch profiles, it is clear that the roughness of the etched sample (Figure 1(b)) was much larger compared to the polished enamel (Figure 1(a)). The deformation during the scratch is also much deeper in case of the etched sample, suggesting the highly porous nature of the etched layer. The lateral force measurement (LFM) option on the G200 nanoindenter measured the friction force during all the tests. As expected, the normalized friction force showed an increasing trend with increased plastic deformation when the normal load was increased from 5 mN to 10 mN and 20 mN. Although the polished sample exhibited almost elastic deformation at 5 mN, the etched sample showed considerable plastic deformation. So, at 0.04, the normalized friction force at 5 mN was estimated to be the friction coefficient for the polished enamel in contact with the diamond tip. In comparison, the value for the etched enamel at similar normal load was 0.28.
The nature of the etched surface layer of the enamel and its deformation becomes more evident from the ramp-load scratch tests. Figure 2 shows the displacement profiles on both surfaces – before, during and after the scratch. The indenter starts loading at the scratch distance of 20 µm, and linearly increases the load up to 20 mN at a distance of 120 µm. Figure 2(a) exhibits that the polished enamel behaves almost elastically up to about 40 µm, and then starts to deform plastically. At a scratch distance of about 70 µm (arrow in Figure 2(a)), the enamel starts to crack resulting in the oscillations observed beyond this point. In contrast, the etched enamel deformed plastically almost from the very beginning. The displacement in the post-profile is also similar to the profile during the scratch, indicating that there is not much elastic recovery of the etched layer and it is extremely prone to abrasion. Figure 2(b) also shows that the etched enamel deforms quickly down to about 500 nm, beyond which the slope of change in displacement decreases. It indicates that the porosity in the etched enamel decreases as a function of increasing displacement. One of the challenges in nano/micro-scratch tests was to visualize the surface features along and around the scratch. These deformation features were not large enough to be observed in an optical microscope, and observation in an SEM is a more time consuming and requires sample transfer. Here, the surface morphology around the scratches in the polished and etched samples was investigated by the high resolution scanning capability in the nanoindenter G200 – especially using the DCM transducer and the nanopositioning stage. Figure 3 clearly exhibits the increase in surface roughness of the dental enamel due to acid etching. The cracks that formed during deformation of the polished enamel can be seen in Figure 3(a) (arrows). The magnified image shows the pileup around the scratch, along with some conformal deformation bands that formed along the scratch. In contrast, the pileup in the etched sample is not as prominent because of the higher surface roughness (Figure 3(b)). The scan also shows the uneven surface of the scratched region, which was also observed in the undulations in the displacement profile in Figure 2(b).

In summary, the nano-tribological behavior of the dental enamel as a function of acid etching is characterized using a nanoindenter. The friction and wear behavior is important for determination of the critical parameters that may cause damage to dental enamel during various clinical procedures.

Nanomeasurement Systems from Keysight Technologies

Keysight Technologies, the premier measurement company, offers high-precision, modular nanomeasurement solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight’s leading-edge R&D laboratories ensure the continued, timely introduction and optimization of innovative, easy-to-use nanomeasure system technologies.

www.keysight.com/find/nano