Keysight Technologies
Tribology of Dental Enamel: Effect of Etching
Nanoindenter G200 Application Brief

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 2µm 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 (Fig. 1b) was much larger compared to the polished enamel (Fig. 1a). 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 5mN to 10mN and 20mN. Although the polished sample exhibited almost elastic deformation at 5mN, the etched sample showed considerable plastic deformation. So, at 0.04, the normalized friction force at 5mN 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.

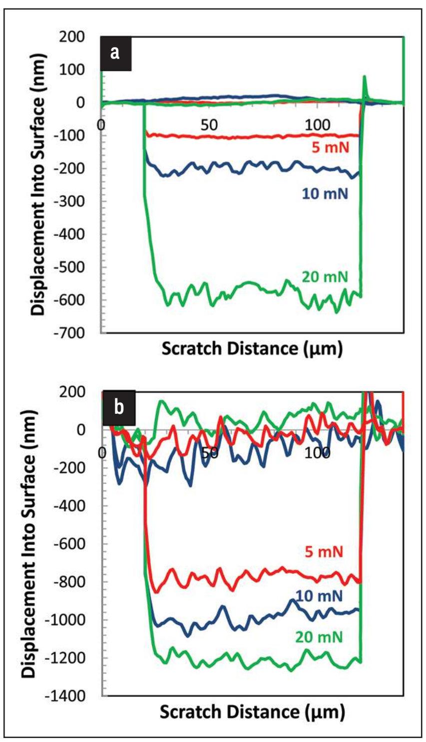

Figure 1. The displacement profiles during constant-load wear tests on (a) polished dental enamel, and (b) enamel after acid etching.

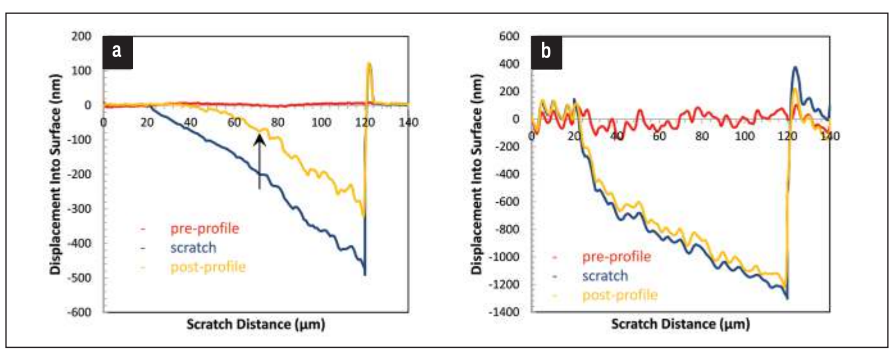

Figure 2. The displacement profiles for ramp-load scratch tests on (a) polished dental enamel, and (b) enamel after acid etching.

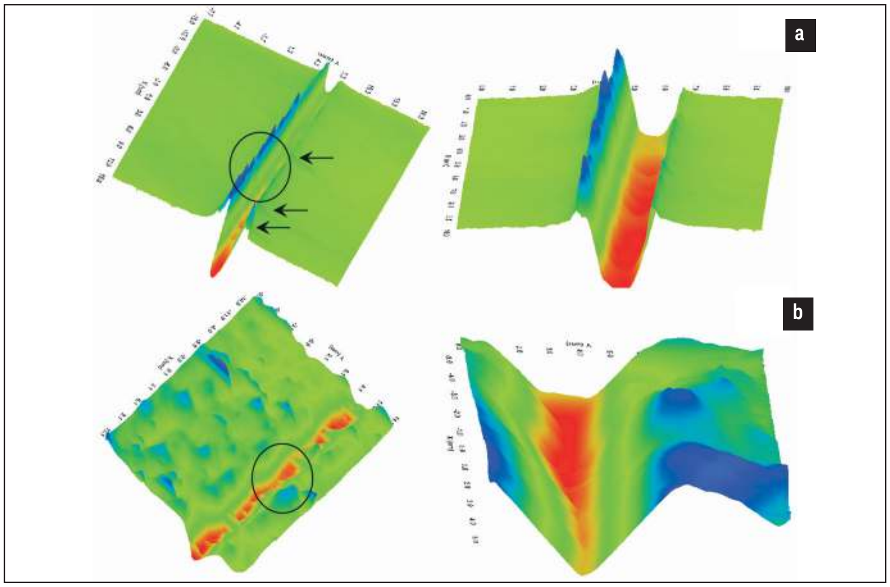

Figure 3. Scan images showing the surface morphologies along and around the nano-scratch on the surface of (a) polished dental enamel, and (b) dental enamel after acid etching. The images on the right are high-resolution scans (10µm x 10µm) performed in the regions marked with the circle in the images on the left.

The nature of the etched surface layer of the enamel and its deformation becomes more evident form 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 20µm, and linearly increases the load up to 20mN at a distance of 120µm. Fig. 2a exhibits that the polished enamel behaves almost elastically up to about 40µm, and then starts to deform plastically. At a scratch distance of about 70µm (arrow in Fig. 2a), 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 2b also shows that the etched enamel deforms quickly down to about 500nm, 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 Fig. 3a (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 (Fig. 3b). The scan also shows the uneven surface of the scratched region, which was also observed in the undulations in the displacement profile in Fig. 2b.

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.

AFM Instrumentation from Keysight

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