

## Application Note #1544

# Dental Restorative Composites: Effect of Polymerization and Time on Mechanical Properties

Restorative dentistry aims to bring damaged dentition back to acceptable functional and aesthetic levels (see Figure 1). Composite materials for dental fillings are replacing the use of amalgam alloys due to their aesthetic aspects, biocompatibility, and non-toxicity, as well as their lack of pollution by mercury waste. Unfortunately, the lifetime of composite fillings is limited, and filling fractures, secondary caries, or filling loss can occur. Due to mechanisms, such as occlusal loading and composite shrinkage related to the process of polymerization in UV light, stresses can be induced in filling and adhesive layers that cause failures of the tooth-restoration interface and that undermine marginal integrity. UV light is applied from the top of the filling, initiating the polymerization process at the surface, continuing to a certain depth underneath the surface.

The problem of restoration failures could be resolved through minimization of interfacial stress by optimizing the cavity shape and dental restoration procedure. Therefore, establishing a relationship between mechanical properties and polymerization depth could help optimize layered restorations. For this application note, aging of the composites was investigated by measuring the evolution in elastic modulus as a function of time after polymerization. Three different dental filling composites with different chemical composition and microstructure of filler particles were evaluated. Establishing the relationship between aging and mechanical properties, which are related to internal stresses and chemical processes, enables optimization of the cavity shape, prolonging the lifetime and performance of the restoration. This application note discusses how nanomechanical studies can be utilized to determine depth of polymerization by measuring the mechanical properties within the cross-section of a polymerized sample, and to understand how mechanical properties evolve with time.



FIGURE 1

Restorative dentistry examples, showing (a) ceramic inlays; (b) amalgam restoration; (c) composite fillings.

## Experimental Procedure

A Hysitron TriboIndenter nanomechanical test instrument was used to perform nanoindentation experiments on the dental composites. Cylindrical holes were drilled into an epoxy resin and filled with the composite, followed by UV curing applied from the top (halogen lamp,  $\lambda=465$  nm). Filled holes were cross-sectioned and polished.

Nanoindentation measurements were performed through the cross section, at increasing distances from the irradiated surface (see Figure 2). Load-controlled quasi-static nanoindentation tests were performed on the samples using a diamond Berkovich probe. The tests followed a trapezoidal loading function and consisted of 5 seconds loading, 10-second constant force hold segment, and 5 s unloading. An indentation matrix of 3x7, with a distance of 15 microns between indents was placed at intervals of 0.5 millimeters to a final depth of 3.5 millimeters under the irradiated surface of the cross-section.

To observe the effect of aging after polymerization on elastic modulus, nanoindentation tests were conducted at four intervals: 1 hour, 1 day, 1 week, and 1 month after specimen exposure to UV light. To simulate real-world conditions, specimens were continuously stored in the dark, immersed in a physiological solution at 37°C.

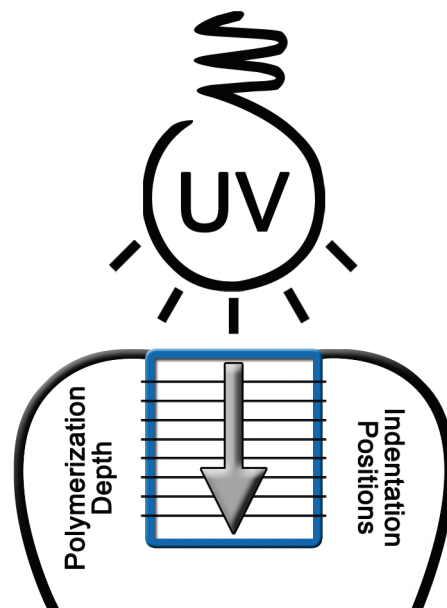


FIGURE 2

Schematic showing the procedure used in testing the dental restorative composites.

## Results and Discussion

The results from both depth profile and aging studies for all three samples of dental composites are shown in Figure 3a and Figure 3b, respectively. Each tested material showed a significant increase in modulus after the first week: Sample 1 from  $11.9 \pm 0.2$  gigapascals to  $14.2 \pm 0.9$  gigapascals; Sample 2 from  $13.1 \pm 0.8$  gigapascals to  $17.6 \pm 0.8$  gigapascals; Sample 3 from  $5.8 \pm 0.2$  gigapascals to  $7.8 \pm 0.3$  gigapascals. A decrease of modulus occurred for Samples 1 and 2 after 1 month: Sample 1 to  $10.8 \pm 1.7$  gigapascals; Sample 2 to  $12.5 \pm 0.3$  gigapascals. The modulus of Sample 3 remained almost unchanged, with a statistically non-significant increase. The results clearly show that the elastic modulus of dental composites vary significantly with time.

Figure 3b demonstrates a dependence of elastic modulus on the depth of UV light absorption. As can be seen, all three samples reached 50-60% of the maximum value at a depth of 3.5 millimeters.

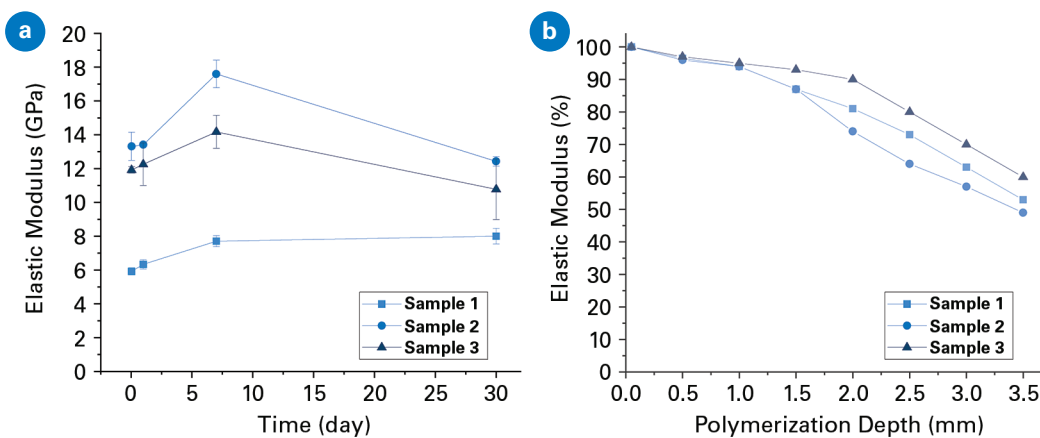


FIGURE 3

(a) Elastic moduli of the resin composite tested at different time periods after the polymerization; (b) Percent of maximum elastic modulus as a function of polymerization depth achieved by UV curing.

## Conclusions

Results indicate that aging time has a considerable effect on the development of the elastic modulus of the restoration composites. These changes can be explained by the setting reaction between the polymer matrix and filling particles after polymerization. The modulus of elasticity depends on curing light intensity, or in other words, on the depth of light absorbance. Minimal decrease of modulus is observed for the depth up to 1.5 millimeters, while a more significant decrease is seen for the depth range between 1.5 and 3.5 millimeters. The polymerization depth that is guaranteed by the producer can be verified by the obtained results. Moreover, layered restoration can be optimized to increase the stiffness of the whole filling, as well as the shape of the cavity.

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## References

1. Eichlerova, R., Lukes, J., Konvickova, S., Bradna, P. *Chem.* **104**, s307 (2010).
2. Eichlerova, R., Lukes, Sepitka, J., Konvickova, S. *Chem.* **105**, s794 (2011).
3. Eichlerova, R., Lukes, Sepitka, J. *Computer Methods in Biomechanics* **14**, 261 (2011).

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