Physical properties of a new sonically placed composite resin restorative material

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A new nanohybrid composite activated by sonic energy has been recently introduced as a single-step, bulk-fill restorative material. The purpose of this study was to compare the physical properties of this new composite to various other composite restorative materials marketed for posterior or bulk-fill placement. The following physical properties were examined: depth of cure, volumetric shrinkage, flexural strength, flexural modulus, fracture toughness, and percent porosity. A mean and standard deviation were determined per group. One-way ANOVA and Tukey's post hoc tests were performed per property ($\alpha = 0.05$). Percent porosity was evaluated with a Kruskal-Wallis/Mann-Whitney test ($\alpha = 0.005$). Significant differences were found between groups ($P < 0.001$) per test type. Compared to the other composite restorative materials, the new nanohybrid composite showed low shrinkage and percent porosity, moderate fracture toughness and flexural modulus, and high flexural strength. However, it also demonstrated a relatively reduced depth of cure compared to the other composites.

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Composite resin was first introduced in the 1960's as an alternative to acrylic resins for esthetic dental restorations.[1] Initially these materials performed poorly, but increased popularity and demand for esthetic restorations have driven continued improvement in strength, wear resistance, handling, and esthetics.[2] For many years, composite resin restorations have been considered an acceptable treatment choice for anterior applications. Recent advances in composite resin mechanical properties and improved adhesive systems have broadened the application of these materials to include the restoration of posterior teeth.[3] However, it is still generally accepted that posterior composite resin restorations have limitations and that there is no one ideal material available.[4]

A volumetric shrinkage occurs when a composite resin material is cured.[1] The shrinkage is the result of conversion of monomer molecules into a more dense polymer network, which leads to bulk contraction.[5] In vivo studies have demonstrated the percentage of marginal gaps in a composite resin restoration may vary between 14% and 54% depending on the materials and technique.[6] The resulting marginal gap may provide a site for recurrent caries; this is cited as the most common cause of failure for composite resins.[7] In spite of significant advances in composite resin composition, there has not been an equivalent decrease in microleakage and gap formation.[8]

Another concern regarding composite resin is the depth of cure during placement. When composite resin is applied as a single bulk layer, there may be a low degree of polymerization at the depth of deeper cavity preparations due to attenuation of the light.[1] An insufficient degree of curing affects the composite resin's chemical properties and may lead to the elution of possible irritant, allergic, or toxic components from the material.[9] Uncured composite resin at the base of a restoration may also cause microleakage with resulting pulpal sensitivity, staining, and recurrent caries.[10] Additionally, incomplete curing is associated with a reduction in the mechanical properties of the material.[11]

Historically, composite resin restorations have been advocated for use in areas of minimal stress.[10] However, increased demand has led to a greater use of these restorations on posterior teeth, where considerable mechanical challenges occur during function.[12] To withstand these stresses, the modification of filler particle size and morphology has resulted in improved mechanical properties.[13] Heavily filled composite resins have improved mechanical strength, fracture properties, and wear resistance.[4] However, as the maximum filler volume is about 70%, overloading can result in poor handling characteristics and technical difficulties, such as decreased wettability.[14] Filler content not only directly determines the mechanical properties of composite resin but also allows for a reduction in monomer content; improves handling properties; and influences wear resistance, translucency, opalescence, radiopacity, intrinsic surface roughness, and polishability.[15]

Another clinical aspect of concern regarding composite resins is their handling characteristics. The ability of a composite material to flow may play a major role in the ultimate success of a restoration.[16] However, in many Class II cavity preparations, it is difficult to obtain proper contour and adequate proximal contacts because the composite resin is not packable.[17] The desire for composite resins with certain flow characteristics has been addressed by the introduction of packable and flowable composite resins. Packable composite resins were first introduced as an alternative to amalgam.[10] They are characterized by a high filler load and a filler distribution that gives them a different consistency when compared with traditional composite resins. Flowable composite resins contain lower filler concentrations and are characterized by a lower elastic modulus and viscosity.[18] For the average clinician, the ideal composite resin material would be viscous enough to facilitate placement but flowable enough for adequate marginal adaptation.[19]

SonicFill (Kerr Corporation) is a new composite resin material that the manufacturer claims to address many of the problems listed above. SonicFill is a single-step, bulk-fill composite resin system that, according to the manufacturer, has

Table 1. Composite resin components.

Composite	Type	Manufacturer	Resin	Filler	Weight %	Volume %	Filler size
SonicFill	Nanohybrid	Kerr Corporation	3-trimethoxysilylpropyl methacrylate, ethoxylated bisphenol-A-dimethacrylate (Bis-EMA), bisphenol-A-bis-(2-hydroxy-3-methacryloxypropyl) ether, triethyleneglycoldimethacrylate (TEGDMA)	Silicon dioxide, barium glass	83	Unreported	Unreported
QuiXX	Hybrid	DENTSPLY Caulk	Urethane dimethacrylate (UDMA), TEGDMA	Silanated strontium aluminum sodium fluoride phosphate silicate glass	86	66	Unreported
Tetric EvoCeram Bulk Fill	Nanohybrid	Ivoclar Vivadent, Inc.	UDMA, bisphenol A glycidylmethacrylate (Bis-GMA)	Barium glass, ytterbium trifluoride, mixed oxide prepolymer	82-84	64	550 nm mean particle size; range: 40 nm to 3000 nm
Filtek Z250	Microhybrid	3M ESPE	TEGDMA, UDMA, Bis-EMA	Zirconia/silica particles	82	60	0.01-3.5 µm Average: 0.6 µm
Filtek LS	Silorane	3M ESPE	Silorane	Quartz, ytterbium trifluoride	76	55	0.04-1.7 µm

"…ultraefficient curing characteristics that ensure an optimal, full 5 mm depth of cure in 20 seconds."[20] Sonic activation purportedly lowers the viscosity of the material to allow for easy adaptation to cavity walls. The manufacturer also claims that, after placement, the composite resin returns to a "non-slumping state" that allows for easy contouring.[20]

To fully understand SonicFill's place in a clinician's daily practice, one must first understand the different types of composite resins available on the market. Most dental composite resin materials are composed of a polymeric matrix (typically dimethacrylate), reinforcing fillers (typically radiopaque glass), a silane coupling agent to bind the filler to the matrix, and chemicals that promote or modulate the polymerization reaction. Because of the major influence of fillers on the physical properties of dental composite resins, their classification is based on the type and particle size of fillers.[4] Currently, the most traditional methacrylate composite resins for restorative purposes are the hybrid and microfill types.[21] Microfill composite resins are formulated with fillers having an average particle size ranging from 0.01 to 0.05 µm and prepolymerized particles approximately 50 µm in size. These composite resins were designed to overcome the problems of poor esthetic properties. However, the mechanical properties of microfills are typically too low for applications in areas of high functional stress.[4] Microhybrids offer intermediate esthetic properties but excellent mechanical properties by the incorporation of fillers with different average particle sizes, 15-20 µm and 0.01-0.05 µm.[21] A recent development with methacrylate-based composites has been nanocomposites, which contain nanoscale particles and nanohybrids, which contain a mixture of nanoscale particles and larger particles.[4] The manufacturers of these nanocomposites claim that they combine the mechanical strength of hybrids and the superior polishabiliy of microfills, in addition to high wear resistance and reduced polymerization shrinkage.[22] In general, it is difficult to discern dramatic differences between nanohybrids and the more traditional microhybrids because many manufacturers have simply modified their microhybrid composition to include more nanoparticles or even prepolymerized resin fillers.[21] The physical properties of the flexural strength and modulus of nanohybrids and microhybrids tend to be similar.[4] Filtek Z250 (3M ESPE) is a traditional microhybrid composite resin that has demonstrated excellent mechanical properties in multiple laboratory studies and is often used as a standard to compare various new restorative materials.[22]

In addition to the traditional composite resin restorative materials, a unique composite resin, Filtek LS (3M ESPE), has recently been marketed for posterior restorations. Instead of the conventional methacrylate-derived monomer, Filtek LS utilizes a ring-opening silorane monomer. It demonstrates mechanical properties similar to those of methacrylate composite resins but has the distinct advantage of reduced polymerization shrinkage. The expansion of the ring before polymerization has been shown to decrease the polymerization shrinkage to <1.5%.[22]

Historically, the maximum incremental thickness with composite resin placement has been 2 mm. However, restoring deeper preparations with 2 mm increments is time consuming and relatively technique sensitive. Manufacturers have introduced new "bulk-filled" restorative composites, which reportedly can be cured in increments of ≥4 mm. Examples include SonicFill, Tetric EvoCeram Bulk Fill (Ivoclar Vivadent, Inc.), and QuiXX (DENTSPLY Caulk). The compositions of the new bulk-fill composites appear to be similar to those of the nanohybrid and

microhybrid restorative composites currently available. However, a greater depth of cure may be obtained by improving the translucency or by the incorporation of additional photoinitiators.[23] Very little information has been published on the physical properties of this new class of materials.

The purpose of this study was to compare the physical properties of the new sonically placed composite and other composite resin restorative materials marketed for posterior placement or bulk fill. The null hypothesis tested was that there would be no significant difference in physical properties among the various composite resin restorative materials.

Materials and methods

The resin composites used in this study were SonicFill (shade A2), QuiXX (universal shade), Tetric EvoCeram Bulk Fill (shade IVA), FiltekZ250 (shade A2), and Filtek LS (shade A2) (Table 1). The following properties were evaluated: depth of cure, volumetric polymerization shrinkage, flexural strength, flexural modulus, fracture toughness, and internal porosity.

Depth of cure

To determine depth of cure, the composite resins were tested using the scraping technique (ISO 4049).[24] Five specimens per group were created. A 4 mm diameter by 14 mm long stainless steel split mold (Sabri Dental Enterprises, Inc.) was placed on a plastic strip-covered glass slide on a standard white background. The composite resin was injected into the mold and a plastic strip was placed. The composite resin was condensed with a glass slide to displace excess resin. The glass slide was removed and the specimens were immediately polymerized with a curing light (Bluephase G2, Ivoclar Vivadent, Inc.) for 20 seconds. Each specimen was polymerized at a distance of 0 mm utilizing a clamp to hold the curing light. The light emission from the Bluephase G2 was analyzed with a spectrophotometer (Blue Light Analytics, Inc.) and a laser power meter (FieldMax II, Coherent, Inc.). The curing light was connected to a power cord to provide continuous, consistent operation. The emitted light was analyzed during a 20-second curing cycle and the following data were collected: mean irradiance, 1132 mW/cm^2; total energy density, 22.8 J/cm^2; energy density in the 360-420 nm spectrum, 4.2 J/cm^2; and energy density in the 420-540 nm spectrum, 18.6 J/cm^2. The uncured resin was then scraped with a plastic instrument starting from the deepest point on the underside of the mold until polymerized resin was reached. The composite resin was removed from the mold and the length of the remaining polymerized material was measured with an electronic digital caliper (GA182, Grobet USA) and divided by 2, according to the ISO standard.[24]

Volumetric polymerization shrinkage

To determine polymerization shrinkage, the AcuVol method by Bisco, Inc. was used.[25] Ten specimens per group were created. The composite resins were placed on a pedestal in a video imaging device (AcuVol, Bisco, Inc.). The specimens were imaged from the side at a distance of 10 cm. The video camera digitized and analyzed the images with the provided image processing software. The specimens were light-cured for 40 seconds using the curing light unit as before. Polymerization shrinkage was recorded continuously for 5 minutes after the light initiation.

Flexural strength and flexural modulus

To determine flexural strength and flexural modulus, a 3-point bending test was used. Ten specimens per group were created. A 2 x 2 x 25 mm stainless steel mold (Sabri Dental Enterprises, Inc.) was placed on a plastic strip-covered glass slide. The specimens were created by injecting the restorative material into the mold until completely filled. The top surface of the mold was covered with a second plastic strip and glass slide as before. One side of the specimen was exposed to a light polymerization unit in 5 separate overlapping increments of 20 seconds each. Next, the mold was turned, and the opposite side of the specimen was exposed to the light in a similar manner. The specimens were then removed from the mold and stored in distilled water at an intraoral temperature of 37°C for 24 hours. Each specimen was placed on a 3-point bending test device which was constructed with a 20 mm span length between the supporting rods. The central load was applied with a head diameter of 2 mm, and a crosshead speed of 0.25 mm/min using a universal testing machine (MTS Systems Corporation). The flexural strength was calculated using the equation:

$$\sigma_{FS} = \frac{3Fl}{2bd^2}$$

Where F is the loading force at the fracture point, l is the length of the support span (20 mm), b is the width, and d is the depth. Measurements were made using the electronic digital caliper. Flexural modulus was determined from the slope of the linear region of the load-deflection curve using analytical software (TestWorks 4, MTS Systems Corporation).

Fracture toughness

Fracture toughness was determined by a single-edge notched beam method. Ten specimens per group were created. To prepare each specimen, a knife-edged split 2 x 2 x 25 mm stainless steel mold (Sabri Dental Enterprises, Inc.) was placed on a plastic strip-covered glass slide as before. The specimens were made by inserting the restorative material into the mold until completely filled. Then the top surface of the mold was covered with a second plastic strip and glass slide as before. One side of the specimen was then exposed to a light polymerization unit for 20 seconds each in 5 separate overlapping increments. Next, the mold was turned over, and the opposite side of the specimen was exposed to the light in a similar manner. The specimens were stored as before, and after 24 hours, the notched specimens were fractured in the universal testing machine similar to flexural strength testing, but at a crosshead speed of 1.0 mm/min, with the notch on the tensile side. The load-deflection (F = load vs u = deflection) curves were recorded; the height, h, and width, w, of the specimens were measured with the inside jaws of an electronic digital caliper as before and the notch depth, a, with a measuring stereomicroscope (Nikon SMZ-1B, Nikon USA) at 10X magnification. Fracture toughness (K_{IC}) was calculated from measurements with

Table 2. Physical properties of the restorative materials.

Restorative material	Physical property mean (standard deviation)					
	Depth of cure (mm)	Volumetric polymerization shrinkage (%)	Flexural strength (MPa)	Flexural modulus (GPa)	Fracture toughness (MPa $m^{1/2}$)	Percent porosity
SonicFill (A2 shade)	3.67 (0.02)[b]	1.88 (0.15)[b]	136.81 (16.29)[b]	10.32 (0.38)[b]	0.56 (0.03)[ab]	0.02 (0.04)[a]
QuiXX (universal shade)	6.31 (0.02)[e]	2.00 (0.08)[bc]	111.86 (16.84)[a]	13.34 (0.84)[c]	0.61 (0.05)[b]	1.42 (1.17)[c]
Tetric EvoCeram Bulk Fill (IVA shade)	4.08 (0.03)[d]	2.31 (0.11)[d]	101.41 (5.86)[a]	8.55 (0.55)[a]	0.52 (0.05)[a]	0.40 (0.76)[b]
Filtek LS (A2 shade)	2.06 (0.02)[a]	1.21 (0.08)[a]	113.89 (18.57)[a]	9.17 (0.39)[a]	0.52 (0.05)[a]	0.44 (0.57)[b]
Filtek Z250 (A2 shade)	3.79 (0.02)[c]	2.13 (0.08)[c]	139.41 (16.35)[b]	10.86 (0.46)[b]	0.62 (0.08)[b]	0.13 (0.09)[b]

Groups with the same lowercase letter per column are not significantly different.

the single-edge notched-bend specimens using the equation:

$$K_{IC} = \frac{3(a/w)^{1/2}[1.99 - a/w(1-a/w)(2.15-3.93a/w+2.7(a/w)^2]FS}{2(1+2a/w)(1-a/w)^{3/2}hw^{3/2}}$$

Where S is the span distance (20 mm) between supports.

Internal porosity

A novel microtomographic technique was used to evaluate internal porosity. Ten specimens per group were created. To prepare each specimen, a 2 mm long and 8 mm diameter plastic mold (Sabri Dental Enterprises, Inc.) was placed on a plastic strip-covered glass slide. The restorative materials were injected into the mold until completely filled. Then, the top surface of the mold was covered with a second plastic strip and glass slide as before. Both ends of the specimen were exposed to a visible light polymerization unit as before for 20 seconds. After storage for 24 hours as before, they were placed in a microtomography unit (No. 1172, Bruker MicroCT) and scans of the samples were made. Recorded images were then reconstructed (NRecon, version 1.4.4, Bruker MicroCT) into 3-dimensional images, which were analyzed using proprietary software (CT Analyzer, version 1.6.0.0, Bruker MicroCT) for percent porosity.

A mean and standard deviation were determined per group. Data were analyzed with a 1-way ANOVA and Tukey's post hoc tests per test type ($\alpha = 0.05$). Due to the non-normal distribution of the data, percent porosity was evaluated with the nonparametric Kruskal-Wallis and Mann-Whitney tests. A Bonferroni correction was applied because multiple comparison tests were completed simultaneously ($\alpha = 0.005$).

Results

Significant differences were found between groups per test type ($P < 0.05$) (Table 2). For the depth of cure measurements, all the groups were significantly different from each other. QuiXX had the greatest depth of cure (6.31 ± 0.02 mm) and Filtek LS had the lowest (2.06 ± 0.02mm). Tetric EvoCeram Bulk Fill, Filtek Z250, and SonicFill performed more moderately.

Filtek LS had the lowest polymerization shrinkage (1.21% ± 0.08%). SonicFill and QuiXX had low shrinkage and were not significantly different from each other. QuiXX was not significantly different from Filtek Z250.Tetric EvoCeram Bulk Fill had the greatest shrinkage (2.31% ± 0.11%).

Filtek Z250 had the greatest flexural strength (139.41 ± 16.35 MPa), but it was not significantly different from SonicFill. TetricEvoCeram Bulk Fill had the lowest flexural strength (101.41 ± 5.86 MPa), but it was not significantly different from QuiXX and Filtek LS.

QuiXX had the greatest flexural modulus (13.34 ± 0.84 GPa). Filtek Z250 and SonicFill had more moderate flexural moduli and were not significantly different from each other. Tetric EvoCeram Bulk Fill had the lowest flexural modulus (8.55 ± 0.55 GPa), but it was not significantly different from Filtek LS.

Filtek Z250 had the greatest fracture toughness (0.62 ± 0.08 MPa $m^{1/2}$), but it was not significantly different from QuiXX or SonicFill. Tetric EvoCeram Bulk Fill and Filtek LS had the lowest fracture toughness (0.52 ± 0.05 MPa $m^{1/2}$), but they were not significantly different from SonicFill.

SonicFill had the lowest percent porosity (0.02% ± 0.04%). QuiXX had the greatest porosity (1.42% ± 1.17%). Filtek LS, Tetric EvoCeram Bulk Fill, and Filtek Z250 had more modest porosity formation and were not significantly different from each other.

Discussion

The null hypothesis was rejected in this study. Statistically significant differences in physical properties were found between composite resins per test type. Very little published research is available evaluating the depth of cure of the new bulk-fill composite resin restorative materials. In this study, using the ISO 4049 standard, SonicFill's average depth of cure was 3.67 mm. Recent studies by Garcia et al and Benetti et al found similar depths of cure of 3.46 mm and 3.43 mm, respectively, using the same ISO 4049 standard.[26,27] Other studies have concluded that the ISO 4049 method is very liberal, and may overestimate the depth of cure compared to other techniques, such as hardness or degree of conversion.[27,28] The depth of cure for SonicFill (3.67mm) was slightly less than Filtek Z250's average depth of cure of 3.79 mm, which is recommended by the manufacturer for placement in incremental layers of only 2.5 mm.[29] The composite resin which had the highest depth of cure was QuiXX at 6.31mm, which exceeded the manufacturer's claim of 4.2 mm.[30] The greater depth of cure may be due to the

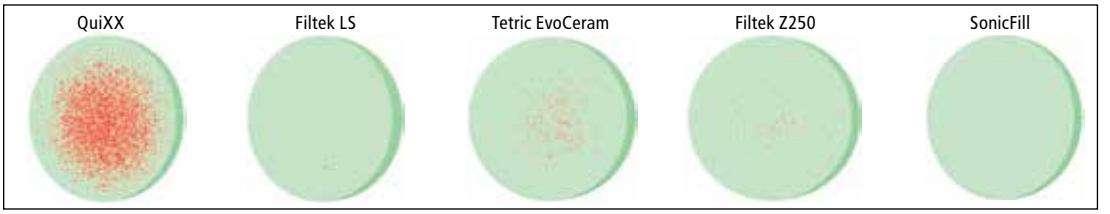

Figure. Representative specimens from each group with porosity formation.

translucent appearance of QuiXX when completely polymerized. Tetric EvoCeram Bulk Fill also met the manufacturer's claim of a 4 mm bulk fill.[31] The manufacturer states that in addition to the traditional camphorquinone/amine photoinitiator system, it contains Ivocerin, an "initiator booster" which reportedly contributes to the increased depth of cure.[23] The manufacturer of SonicFill recommends that it be cured with 10 seconds of additional light curing on the buccal and lingual surfaces after the initial 20 second light cure from the occlusal.[20] Additional light curing from the proximal would likely increase the polymerization of the other composite resins tested in this study. However, laboratory studies have shown that enamel and dentin significantly attenuate the light from a curing unit.[32] Limited research has been completed on the effects of tri-sited light curing on the depth of cure of bulk-fill composites.

Studies evaluating the efficacy of incremental versus bulk filling have been somewhat equivocal, with higher shrinkage stress and cuspal deflection in some studies but reduced cuspal deflection in others.[33] Incremental layering may allow flow during curing with additional free surface area. However, incremental curing allows more maximum polymerization and potentially more shrinkage stress. Little clinical evidence exists to support one particular composite resin application method over another.[4]

Polymerization shrinkage has been steadily reduced through improvements in chemistry and composition.[8] A new composite resin, Filtek LS, is promoted as a low-shrinking composite resin based on a ring-opening polymerization mechanism.[34] As expected, Filtek LS had the lowest shrinkage of all of the composite resins tested (1.21%). SonicFill had the second lowest polymerization shrinkage of (1.88%), although it was not significantly different from QuiXX (2.00%). However, all the composite resins tested exhibited relatively volumetric low shrinkage. An average volumetric shrinkage of 2%-3% occurs when restorative composite resins are polymerized, with the ring-opening silorane-based composite, Filtek LS, reportedly approaching 1%.[1,22]

For restorations exposed to greater mechanical loads, the ideal minimum flexural strength is 90-100 MPa.[35] In addition, a relatively high modulus is expected from posterior composite resin restorations to withstand the occlusal forces and preserve the adhesive interface.[13] All composite resins tested showed adequate flexural strength and flexural moduli, although there were statistically significant differences among groups.

Another important mechanical property for dental composite resin materials is fracture toughness, which indicates the relative resistance to crack propagation from the surface or inherent flaws in the materials.[36] Resin composites with higher fracture toughness will be better able to withstand high stress levels and thus have improved clinical outcomes.[37] Filtek Z250 and QuiXX had the highest relative fracture toughness values, while SonicFill, Filtek LS, and Tetric EvoCeram Bulk Fill had slightly lower fracture toughness values. Despite the statistical differences, the results of this study show that all the restorative materials tested have adequate fracture toughness for use in posterior restorations.[4]

Voids within a composite resin restoration may cause marginal leakage and discoloration, increased wear (due to stress concentration around the voids), decreased flexural strength, and incomplete adhesion between the resin composite and tooth structure.[38] These voids may be incorporated into the composite material due to the manufacturing process or from handling techniques during clinical placement. With the new sonically placed composite resin, it was unknown if sonic energy would have an influence on the number and size of porosities. The results of this study showed less porosity with SonicFill, at least within the body of the specimen, as compared to the other composite resins tested (Figure). QuiXX had the largest number of porosities. Significant variability in porosity was found in the composite resins tested. The variability may be due to differences in handling characteristics of the different composite resin and the subsequent inclusion of larger voids during the fabrication of the specimens.

Overall, SonicFill has satisfactory mechanical properties for use as a direct posterior composite resin restorative material. The potential convenience of sonic placement and the advantage of the reduction in viscosity would likely be operator-dependent preferences. However, a disadvantage of SonicFill is that the depth of cure was determined to be significantly less than the other bulk-fill composites tested in this study. Furthermore, although the composite resin refill compules are similar in price to other comparable materials, the restorative dentist would incur an additional expense per each SonicFill handpiece and coupler. More research is necessary to evaluate the clinical performance of this new sonically placed composite resin material and the new class of bulk-fill restorative materials.

Conclusion

Compared to the other composite resin restorative materials, SonicFill showed low shrinkage and percent porosity, moderate fracture toughness and flexural modulus,

and high flexural strength. However, it had a relatively reduced depth of cure compared to the other composites.

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Disclosure

The views expressed in this study are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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Blue Light Analytics, Inc., Halifax, Canada
902.407.4242, www.curingresin.com

Bruker MicroCT, Kontich, Belgium
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Coherent, Inc., Santa Clara, CA
800.227.8840, www.coherent.com

DENTSPLY Caulk, Milford, DE
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Grobet USA, Carlstadt, NJ
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Ivoclar Vivadent, Inc., Amherst, NY
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Kerr Corporation, Orange, CA
800.537.7123, www.kerrdental.com

MTS Systems Corporation, Eden Prairie, MN
899.328.2255, www.mts.com

Nikon USA, Melville, NY
631.547.4200, www.nikonusa.com

Sabri Dental Enterprises, Inc., Downers Grove, IL
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3M ESPE, St. Paul, MN
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