4

# Resin bond strength to a zirconia-reinforced ceramic after different surface treatments

Tiago Aurelio Donassollo, DDS, MSC, PhD ▪ Flavio Fernando Demarco, DDS, PhD ▪ Alvaro Della Bona, DDS, MSC, PhD

This study evaluated the microtensile bond strength between a composite resin and a zirconia-reinforced alumina-based glass-infiltrated ceramic after different ceramic surface treatments. Blocks (12 mm x 10 mm x 5 $mm^3$) of a ceramic containing zirconia were fabricated, polished, and divided at random into seven groups, with each group receiving a different surface treatment. The adhesive system and composite resin were applied to the treated ceramic; these composite-ceramic blocks were stored in distilled water (37°C) for seven days. At that point, they were cut along two axes to produce specimens with a cross-sectional area of 1.0 (± 0.1) $mm^2$. The specimens ($n = 20$) were loaded in tension in a universal testing machine with a cross-head speed of 0.5 mm/minute. The microtensile bond strength values were calculated and statistically analyzed using ANOVA and Tukey's test ($\alpha = 0.05$). Among the seven groups, specimens with both a silica coating and a silane coupling agent showed the highest mean microtensile bond strength values. All specimens treated with 9.5% hydrofluoric acid failed during the cutting procedure; among all specimens, the majority of the failures were cohesive. A positive correlation was observed between the fracture type and the microtensile bond strength ($r = 0.63$).

Received: July 3, 2008
Accepted: September 17, 2008

The In-Ceram system (Vident) uses an alumina slip casting technique to build the framework. The framework is fired to form an open-pore microstructure, which is infiltrated with lanthanum oxide-based glass.[1] As a result, the high mechanical properties of this system depend upon glass infiltration causing a complete wetting of the porous microstructure. It has been reported that increasing the zirconia content will improve the flexural strength values of In-Ceram Zirconia (Vident).[2,3]

In-Ceram Zirconia was developed by combining the mechanisms of zirconia with the established technology of partially sintered glass-infiltrated alumina to produce stronger, tougher, metal-free ceramic restorations that could be used successfully as posterior three-unit fixed bridges.[1] However, a number of studies have reported that it is difficult for resin to adhere to this material micromechanically and chemically.[4-7]

The clinical success of the adhesive technique (for both cementation and ceramic repair) relies on adequate bonding resistance, which could be produced by micromechanical retention (acid etching, airborne particle abrasion, silica coating) or chemical bonding (organic silane).[8,9]

One popular method for treating ceramic and metal surfaces involves using intraoral appliances to abrade the surfaces with airborne particles of aluminum oxide ($Al_2O_3$). This treatment produces a retentive surface, increasing both the surface area and the surface energy, thus favoring adhesion.[4,5,7,8,10] Other studies have recommended a silica coating procedure to produce an adequate retentive surface on ceramics with high crystalline content, thus improving the bond to resin materials.[1,5,11-16]

While there are several methods for evaluating the bond strength of adhesive interfaces, the microtensile bond strength test is considered the best for testing bonding properties.[8,17,18] This test attempts to avoid the potential influence of defects at the adhesive interface by using specimens with a reduced surface area. Larger adhesive interfaces increase the possibility of more and larger defects due to locked air bubbles, adhesive peeling from a poorly primed surface, and the formation of irregular adhesive layers.[8,18] By contrast, the microtensile bond strength test has produced a more uniform stress distribution at the adhesive interface, avoiding the cohesive fracture of the substrate and reducing the variability of the results.[8,17,18]

The failure mode must be considered when analyzing the fractured surface of the tested specimens. Observing these fractured surfaces offers information regarding the mode of failure, the distribution of the defects, the size and origin of the critical defect, the type of stress at or near the adhesive interface, some material properties, and the potential effects of the environment on the adhesive interface. All of these factors help in understanding the properties of the adhesive interface.[19]

The well-known bonding mechanism of feldspathic ceramic to resinous materials results from hydrofluoric acid etching, which creates micromechanical retention and allows for the chemical bond of the silane agents.[8,20] Nevertheless, a number of studies have reported that hydrofluoric acid and silanation produce an inadequate bond between high crystalline content ceramics and resinous materials.[1,5-7,11,12,14,16] Other studies have indicated that silica coating is a better surface treatment for these types of ceramics.[1,7,10,12,13,15,16]

This study utilized different ceramic surface treatments to evaluate the microtensile bond strength between a zirconia-reinforced alumina-based glass-infiltrated ceramic (In-Ceram Zirconia) and a composite resin, to test the hypothesis that mean bond strength values to resin are greater when this ceramic is coated with silica rather than treated with airborne particle abrasion, hydrofluoric acid etching, or silanation. The predominant failure mode of the adhesive interface also was determined and correlated to the bond strength values.

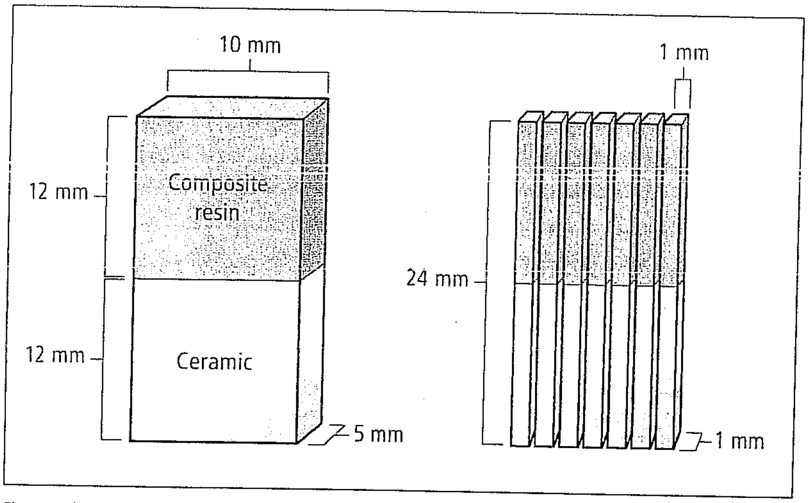


Fig. 1. Fabrication of specimens for the microtensile test. *Left:* The dimensions of a composite resin-ceramic block. *Right:* An illustration of the bar-shaped specimens obtained and their dimensions.[8]

## Materials and methods

Twenty-one blocks (each measuring 12 mm x 10 mm x 5 mm) of In-Ceram Zirconia were fabricated according to the manufacturer's instructions. The surfaces of all specimens were polished using 400–1200 grit silicon carbide (SiC) paper (Norton Abrasives), followed by a 1 µm diamond paste. The ceramic blocks were washed for 10 minutes in a sonic bath of distilled water and divided randomly into seven groups, according to the type of surface treatment. Samples in Group 1 were etched with 9.5% hydrofluoric acid (Ultradent Products, Inc.). The ceramic surface was etched for 60 seconds, washed with an air-water spray for 60 seconds, and air-dried.

For samples in Group 2, silica coating (3M ESPE) was applied using a hand blaster (Chameleon Dental Products). The hand blaster was held perpendicular to the ceramic surface at a distance of 10 mm. The silica-modified aluminum oxide particles (30 µm) were blasted against the surface for 15 seconds using two bars of pressure.

Group 3 samples underwent airborne particle abrasion using aluminum oxide (25 µm) particles in a hand blaster (Handiblaster) and the same parameters described for Group 2.

For samples in Group 4, a silane agent (Espe-Sil, 3M ESPE) was applied to the ceramic surface and allowed to evaporate for five minutes; at that time, samples were air-dried.

In addition to the treatments described above, three other treatment combinations were examined: hydrofluoric acid with a silane agent (Group 5), silica coating with a silane agent (Group 6), and airborne particle abrasion with silica coating (Group 7).

To build a bonded composite block onto the ceramic block, the Single Bond adhesive system (3M ESPE) and a composite resin (Filtek Z250, 3M ESPE) were used on the treated ceramic surfaces, according to the manufacturer's instructions (Fig. 1).

The ceramic-composite blocks were stored in distilled water (37°C) for seven days; at that point, a diamond disk (under cooling) was placed in the precision cutting machine and the blocks were cut along the X and Y axes to produce bar-shaped specimens, each with a length of 24 mm and a cross-sectional area of 1.0 ± 0.1 mm$^2$ (Fig. 1). All specimens were examined for flaws using an optical microscope to determine exclusion criteria. In addition, the bar-shaped specimens obtained from the external walls of the ceramic-composite blocks were eliminated to avoid potential deficiencies of the adhesive material at the interface.[8,17,21]

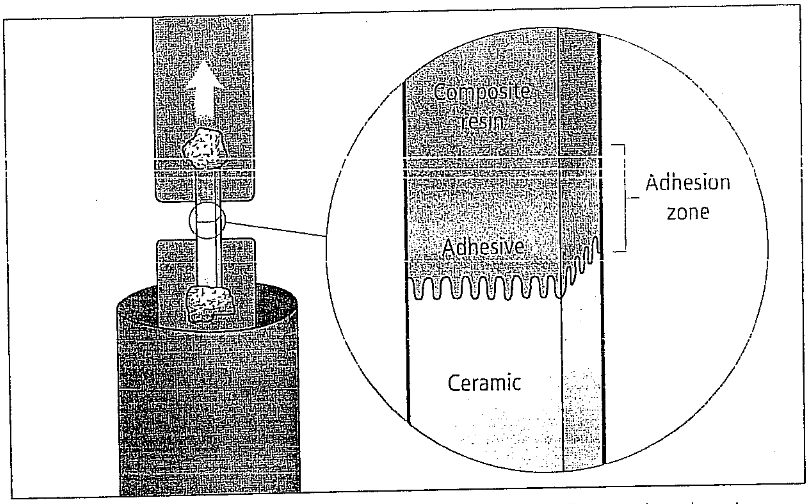


Fig. 2. Schematic illustration of a specimen for the microtensile test. *Left:* A bar-shaped specimen is fixed to the flat grips of the Bencor Multi-T device and loaded to failure under tension. *Right:* The adhesive interfaces and adhesion zone of a specimen.[8]

Twenty specimens from each experimental group were selected randomly for microtensile tests. The one exception was Group 1, where all of the specimens demonstrated adhesive failures during the cutting procedure. Using cyanoacrylate adhesive (Zapit, Dental Ventures of America), the specimens were attached to the flat grips of a Bencor Multi-T testing system (Danville Engineering) and loaded to failure under tension at a cross-head speed of 0.5 mm/minute. A universal testing machine (EMIC DL2000, Emic Ltd.) was used to debond the samples and test the resin/ceramic interface (Fig. 2).[8]

A digital caliper (Digimatic Caliper, Mitutoyo) was used to measure the bonding area of all specimens immediately after testing. The bond strength (μTBS = tensile strength (F)/Area (A)) was calculated (in MPa) by using the values provided by the universal testing machine when the samples broke. The microtensile bond strength data were analyzed statistically using ANOVA and Tukey's test ($\alpha = 0.05$).

The fractured surfaces were examined using light microscopy (magnification 40x) to determine if the failures were adhesive or cohesive. The Pearson correlation test ($\alpha = 0.05$) was used to investigate the association between failure mode and bond strength.

## Results

ANOVA showed that surface treatment has a significant influence on bond strength values ($p < 0.001$). Tukey's test revealed greater mean bond strength values for Groups 2 and 6 ($p < 0.05$). Specimens in Group 7 produced mean bond strength values similar to those for Groups 2 and 3; Groups 3, 4, and 5 produced the lowest bond strengths ($p < 0.05$) (see the table). Group 1 was not included in the table because all specimens failed adhesively during the cutting procedure, which indicates that this treatment is not recommended for this particular ceramic system.

An overall analysis revealed that 61% of all failures were cohesive failure of the adhesive resin, while 37% were adhesive failures at the ceramic/adhesive interface; only 2% were cohesive failures of both the adhesive and composite resin. All failures were within the adhesion zone. There was a positive correlation ($r = 0.63$) between the failure mode and the mean bond strength values. Samples with lower bond strengths had a higher incidence of truly adhesive failures, while the incidence of cohesive failures increased as bond strength values increased (see the table).

## Discussion

The microtensile test was developed to avoid non-uniform stress distribution at the adhesive interface. It has been used to determine the bond strength between dental structures, resin materials, and ceramics.[8,16-18,21-25]

The tensile strength of brittle materials usually decreases as the cross-sectional area increases, because larger specimens can exhibit more defects. The same statement is true for adhesive interface areas; smaller areas tend to produce higher bond strength values, because small surface areas result in fewer failures.[18,26]

When conducting the microtensile bond strength test, it is likely that a small number of smaller defects will be present in each sample, reducing the degree of variation in the results, which follows a greater incidence of failures at the adhesive interface.[8] Therefore, this test has been considered the most adequate to determine the bond strength of different materials, which justifies the selection of this method for the present study.[8,17,21-25,27] Indeed, the results of the present study showed that all failures happened at the adhesive interface.

Table. Mean tensile strength (microtensile bond strength), standard deviation (SD), statistical groupings, and mode of failure percentage for the In-Ceram Zirconia ceramic bonded to composite resin after different ceramic treatments.

| Group number | Microtensile bond strength ± SD (MPa)* | Mode of failure (%) |
|---|---|---|
| 2 | 20.7 ± 5.3[ab] | Cohesive failure of the adhesive (100) |
| 3 | 15.7 ± 5.6[cd] | Cohesive failure of the adhesive (65)<br>Adhesive failure (35) |
| 4 | 10.2 ± 2.9[d] | Adhesive failure (80)<br>Cohesive failure of the adhesive (20) |
| 5 | 12.3 ± 3.5[d] | Adhesive failure (100) |
| 6 | 22.8 ± 4.7[a] | Cohesive failure of the adhesive (100) |
| 7 | 18.4 ± 3.5[bc] | Cohesive failure of the adhesive (85)<br>Cohesive failure of the adhesive and composite resin (10)<br>Adhesive failure (5) |

*Different superscript letters indicate statistically significant differences ($p < 0.05$).

With small adhesive areas, the probability that the failures could be adhesive in origin increases. However, some problems may occur during the microtensile bond strength test. The preparation of small specimens, their fixation during the test, and keeping them parallel during loading must be considered to avoid inappropriate results or losing an excessive number of specimens.[18,22]

The present study showed an increase in bond strength values between the zirconia-reinforced infiltrated ceramic and composite resin when the ceramic surface was treated with a silica coating procedure (CoJet), as observed for the groups treated with silica coating alone (20.7 MPa) and silica coating with silane coupling agents (22.8 MPa). A 2005 study that used a different ceramic with a high crystalline content showed that two silica coating techniques produced greater microtensile bond strength values with resin than the ceramic treated using airborne particle abrasion with aluminum oxide particles.[23]

Several articles have compared the resistance to fracture between composite resins and ceramics using Bis-GMA-based and phosphate monomer-based resin cements. These studies suggested that luting high crystalline content ceramics with phosphate monomer-based resin cements promotes a high and stable bonding resistance between these materials because of the chemical bonding between the monomer and the aluminum oxide on the ceramic surface.[7,23,28,29] However, no luting cement was used in the present study, which simulates a resin composite repair of a zirconia-reinforced ceramic restoration.

The silane agent provides the link between the inorganic component of the ceramic (silica) and the organic matrix of the composite resin. This agent also increases the surface energy, improving the wettability of the adhesive system.[4,20] Based on these results, the silane coupling agents should increase the bond strength of ceramic to resin, provided that the surface of the ceramic has an adequate amount of silica.[1,4,8] In the present study, a synergetic action was observed between the chemical bonding agent (silane) and the micromechanical retention methods used in Groups 1, 2, and 3. By contrast, Group 4 samples produced lower bond strength values (10.23 MPa).[5,7,8,10,12,13,16]

Although hydrofluoric acid and silane treatments demonstrated adequate bond strengths between the resinous materials and feldspathic ceramics, neither treatment appears to be effective for high crystalline ceramic systems.[16,23]

An adequate micromechanical retentive ceramic surface is essential for providing a strong and long-lasting bond with resinous materials. The adhesive agents penetrate into the porosities created and the posterior polymerization produces the bond.[7,8,11] However, as Della Bona *et al* reported in 2002, different ceramic compositions and microstructures can result in different retentive patterns after surface treatments.[30] The hydrofluoric acid attacks the ceramic matrix (usually the phase formed by silica), forming a hexafluorosilicate layer; this layer is removed by water, leaving a porous surface that is ideal for micromechanical retention of the luting cement.[31]

According to a 2007 article by Della Bona *et al*, hydrofluoric acid is unable to produce significant structural changes on the surface of the zirconia-reinforced alumina-based glass-infiltrated ceramic, due to the acid's low silica content.[1] As a result, in the present study, the samples in Group 1 did not produce a significant bond to resin; all specimens in the group failed during the cutting procedure. The samples in Group 5 showed the lowest mean bond strength value (12.3 MPa), which was not statistically different from the mean bond strength of the samples in Group 4 (10.2 MPa). These results are similar to those found in previous studies.[6,7,32]

Previous studies reported that high crystalline ceramic systems treated with airborne particle abrasion did not produce much greater mean bond strengths to resin when compared with other ceramic surface treatments. It has been suggested that this outcome was the result of similar hardness values between the airborne particles and the crystalline ceramic phase, both of which usually are alumina-based.[11,32] In the present study, Group 3 samples showed an intermediate mean bond strength value of 15.7 MPa, compared to 18.4 MPa for Group 7 samples.

The analysis of the fractured surfaces revealed that the majority of the failures (61%) were cohesive failures of the adhesive system, while 37% were truly adhesive failures. These findings are similar to those produced using a microtensile test, in which the majority of the failures occurred within the adhesion zone.[8,22,25,27]

This study confirmed the experimental hypothesis that silica coating zirconia-reinforced alumina-based glass-infiltrated ceramic produces greater mean bond strength values to resin compared with other ceramic surface treatments.

## Conclusion

Within the limitations of the study, the surface treatment significantly influenced the bond strength between resin and a zirconia-reinforced alumina-based glass-infiltrated ceramic, which relates clinically to resin luting and repairing this type of restoration. These findings indicate that the silica coating procedure plays a major role on the bond strength between this type of ceramic and resin. A silane coupling agent improves the bonding effect of surface treatments that produce micromechanical retentive surfaces. Although a positive correlation was found between bond strength and failure mode, this synergistic effect of micromechanical retentive treatments and chemical bonding via coupling agents did not appear to affect the mode of failure, which was controlled mostly by the micromechanical retentive treatment of the zirconia-reinforced ceramic.

## Author information

Dr. Donassollo is a dentist in the Brazilian Army, Pelotas, RS, Brazil. Dr. Demarco is an associate professor Department of Operative Dentistry, School of Dentistry, Federal University of Pelotas. Dr. Della Bona is an associate professor, Department of Restorative Dentistry, School of Dentistry, University of Passo Fundo, Brazil.

## References

1. Della Bona A, Donassollo TA, Demarco FF, Barrett AA, Mecholsky JJ Jr. Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. Dent Mat 2007;23(6):769-775.
2. Chong KH, Chai J, Takahashi Y, Wozniak W. Flexural strength of In-Ceram alumina and In-Ceram zirconia core materials. Int J Prosthodont 2002; 15(2):183-188.
3. Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater 2004;20(5):449-456.
4. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. Dent Mater 2004;20(4):338-344.
5. Derand P, Derand T. Bond strength of luting cement to zirconium oxide ceramics. Int J Prosthodont 2000;13(2):131-135.
6. Madani M, Chu FC, McDonald AV, Smales RJ. Effects of surface treatments on shear bond strengths between a resin cement and an alumina core. J Prosthet Dent 2000;83(6):644-647.
7. Ozcan M, Alkumru HN, Gemalmaz D. The effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. Int J Prosthodont 2001;14(4):335-339.
8. Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. J Adhes Dent 2000;2(4): 305-313.
9. Lu YC, Tseng H, Shih YH, Lee SY. Effects of surface treatments on bond strength of glass infiltrated ceramic. J Oral Rehabil 2001;28(9): 805-813.
10. Frankenberger R, Kramer N, Sindel J. Repair strength of etched vs silica-coated metal-ceramic and all-ceramic restorations. Oper Dent 2000; 25(3):209-215.
11. Awliya W, Oden A, Yaman P, Dennison JB, Razzoog ME. Shear bond strength of resin cement to densely sintered high-purity alumina with various surface conditions. Acta Odontol Scand 1998;56(1):9-13.
12. Isidor F, Stokholm R, Ravnholt G. Tensile bond strength of resin luting cement to glass infiltrated porous aluminium oxide cores (In-Ceram). Eur J Prosthodont Restor Dent 1995;3(5):199-202.
13. Jedynakiewicz NM, Martin N. The effect of surface coating on the bond strength of machinable ceramics. Biomater 2001;22(7):749-752.
14. Kern M, Thompson VP. Sandblasting and silica coating of a glass-infiltrated alumina ceramic: Volume loss, morphology, and changes in the surface composition. J Prosthet Dent 1994;71(5): 453-461.
15. Sun R, Suansuwan N, Kilpatrick N, Swain M. Characterisation of tribochemically assisted bonding of composite resin to porcelain and metal. J Dent 2000;28(6):441-445.
16. Valandro LF, Della Bona A, Antonio Bottino M, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. J Prosthet Dent 2005;93:253-259.
17. Pashley DH, Carvalho RM, Sano H, Nakaijima M, Yoshiyama M, Shono Y, Fernandes CA, Tay F. The microtensile bond test: A review. J Adhes Dent 1999;1(4):299-309.
18. Pashley DH, Sano H, Ciucchi MY, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: A review. Dent Mater 1995;11(2): 117-125.
19. Della Bona A, Anusavice KJ, Hood JA. Effect of ceramic surface treatment on tensile bond strength to resin cement. Int J Prosthod 2002; 15(3):248-253.
20. Phoenix S, Shen C. Characterization of treated porcelain surfaces via dynamic contact angle analysis. Int J Prosthodont 1995;8(2):187-194.
21. Chen KK, Shono Y, Ogawa T, Kozono Y, Terashita M. Fracture aspects of resin-dentin bonding in non-trimming microtensile test. Dent Mater 2001;20(4):315-324.
22. Abdalla AI. Microtensile and tensile bond strength of single-bottle adhesives: A new test method. J Oral Rehabil 2004;31(4):379-384.
23. Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. Int J Prosthodont 2005;18(1):60-65.

24. Cardoso PE, Braga RR, Carrilho MR. Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. Dent Mater 1998;14(6):394-398.
25. Shono Y, Terashita M, Pashley EL, Brewer PD, Pashley DH. Effects of cross-sectional area on resin-enamel tensile bond strength. Dent Mater 1997;13(5):290-296.
26. Ritter JE. Predicting lifetimes of materials and material structures. Dent Mater 1995;11(2):142-146.
27. Della Bona A, Anusavice KJ, Mecholsky JJ Jr. Failure analysis of resin composite bonded to ceramic. Dent Mater 2003;19(8):693-699.
28. Friederich R, Kern M. Resin bond strength to densely sintered alumina ceramic. Int J Prosthodont 2002;15(4):333-338.
29. Kern M, Wegner SM. Bonding to zirconia ceramic: Adhesion methods and their durability. Dent Mater 1998;14(1):64-71.
30. Della Bona A, Anusavice KJ. Microstructure, composition, and etching topography of dental ceramics. Int J Prosthod 2002;15(2):159-167.
31. Thurmond JW, Barkmeier WW, Wilwerding TM. Effect of porcelain surface treatments on bond strengths of composite resin bonded to porcelain. J Prosthet Dent 1994;72(4):355-359.
32. Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: Adhesive methods and their durability. J Prosthet Dent 1995;73(3):240-249.

## Manufacturers

Chameleon Dental Products, Kansas City, KS
913.281.5552, www.miragecdp.com

Danville Engineering, San Ramon, CA
800.827.7940, www.danvilleproducts.com

Dental Ventures of America, Corona, CA
800.228.6696, www.dentalventures.com

Emic Ltd., Sao Jose dos Pinhais, PR, Brazil
41.3283.1143, www.universaltestingmachines.net/index.php?lang=17

Mitutoyo, Aurora, IL
888.648.8869, www.mitutoyo.com

Norton Abrasives, Worcester, MA
508.795.5000, www.nortonabrasives.com

Ultradent Products, Inc., South Jordan, UT
888.230.1420, www.ultradent.com

Vident, Brea, CA
888.249.1640, www.vident.com

3M ESPE, St. Paul, MN
888.364.3577, www.3mespe.com