Evaluation of Various Properties of "Bulk Fill Material"
Final Report.

Submitted to:
Xiangxu (Sean) Chen, PhD
Director of R&D
Danville Materials LLC
4020 E. Leaverton Court
Anaheim, CA 92807
Tel: 714-399-0334 ext. 211
Cell: 909-758-1891
Fax: 714-399-0338
Email: schen@daneng.com

Submitted by:
Jack L. Ferracane, Ph.D.
Chair, Department of Restorative Dentistry
Division Director, Biomaterials and Biomechanics
Oregon Health & Science University
2730 SW Moody Ave.
Portland, Oregon, 97201 USA
ph: 503-494-4327
fax: 503-494-8260
email: ferracan@ohsu.edu

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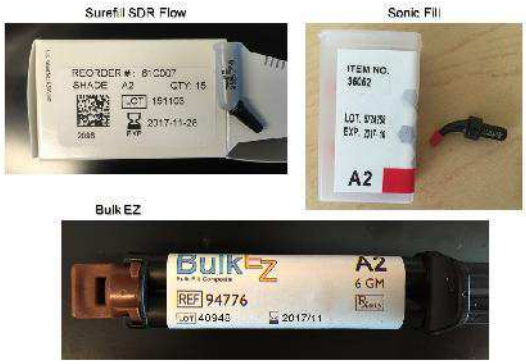
Objective

This protocol was designed to provide basic information on the physical properties of a new material developed by Danville Engineering, further known in this proposal as "Bulk Fill Material."

Materials

The material, BulkEZ (Danville), was compared against Surefill SDR (Dentsply Sirona) and SonicFill 2 (Kerr).

BulkEZ (A2): Lot 40948; Exp. 2017-11
Surefill SDR (A2): Lot 151103; Exp. 2017-11-28
Sonic Fill 2 (A2): Lot 5724258; Exp. 2017-10



Methods:

The properties to be tested included the following:
1. Polymerization contraction stress (Bioman)
2. Flexure strength/modulus
3. Fracture toughness
4. Depth of cure – tooth model

Note: As the new material is a dual cure material, the manufacturer asked that the material be allowed to self-cure for 200 seconds prior to light-curing. This method was followed in all of the study protocols.
Note: Originally volumetric shrinkage was to be assessed, but this protocol was not followed due to equipment issues and was removed from the final budget invoice.

1. Polymerization Contraction Stress:

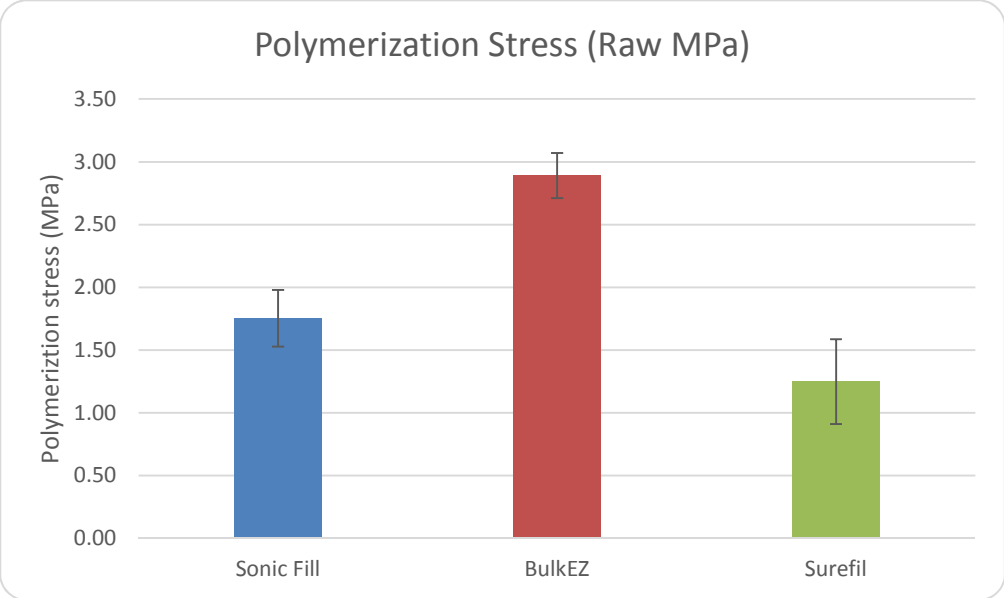
The Bioman stress measurement device has been described previously in detail (Watts et al., 2003). The system is based on a cantilever load-cell (500 kg) fitted with a rigid integral clamp. The compliant end of the cantilever holds a circular steel rod (5 mm diameter × 22 mm long) vertically and perpendicular to the load-cell axis. The counter-face consists of a removable rigid glass plate that is held rigidly relative to the base-plate in a special clamp during measurement.

Test Method: The lower end of the steel rod is abraded on silicon carbide paper and treated with Z-Prime (Bisco), and in contrast to the original methodology described by Watts, the surface of the glass-plate opposing the steel-rod is only treated with silane (3M/ESPE Dental Products, St Paul, MN 55144, USA)) (instead of sandblasting + silanation). The composite is then introduced between the plate and vertical rod to form an uncured specimen-disk of 5 mm diameter and 0.8 mm thickness (which represents a bonded to non-bonded surface area, i.e. C-factor, of ~3). The composite specimen is irradiated through its thickness dimension from below by the LCU (Demi, Kerr) for 20 s (actual irradiance reaching the specimen = 670 mW/cm^2), as measured with the MARC resin calibrator (BlueLight Analytics). *Note: The light application will be delayed until 200 seconds for the Danville product to allow time for the self-cure reaction*. The registered load (in Newton, N) is divided by the disk area to obtain the stress values (MPa). Subsequently, as in

previous studies, (Watts and Satterthwaite, 2008), the raw stress data is treated by a "correction factor" of 4 in order to relate the present data to a low compliance system, such as a human tooth cusp.

Measurements are performed during 10 min after the photoactivation procedure at ~22°C (n = 5). After each evaluation, the Bioman clamps are removed and the set resin sample/glass-plate/metal piston is removed and carefully observed to verify if signs of any debonding is present. It is important to mention that detachments are rare and that these samples will be excluded from the study. The results for the different composites are compared using ANOVA and Tukey's multiple comparison test (α=0.05).

Results: Statistical analysis showed that the contraction stress for BulkEZ was highest, and significantly higher than SonicFill 2, which was also significantly higher than Surefill SDR. This is consistent with previous findings in our lab that Surefill SDR has the lowest contraction stress of the bulkfill materials.



2. Flexural Strength/Modulus:

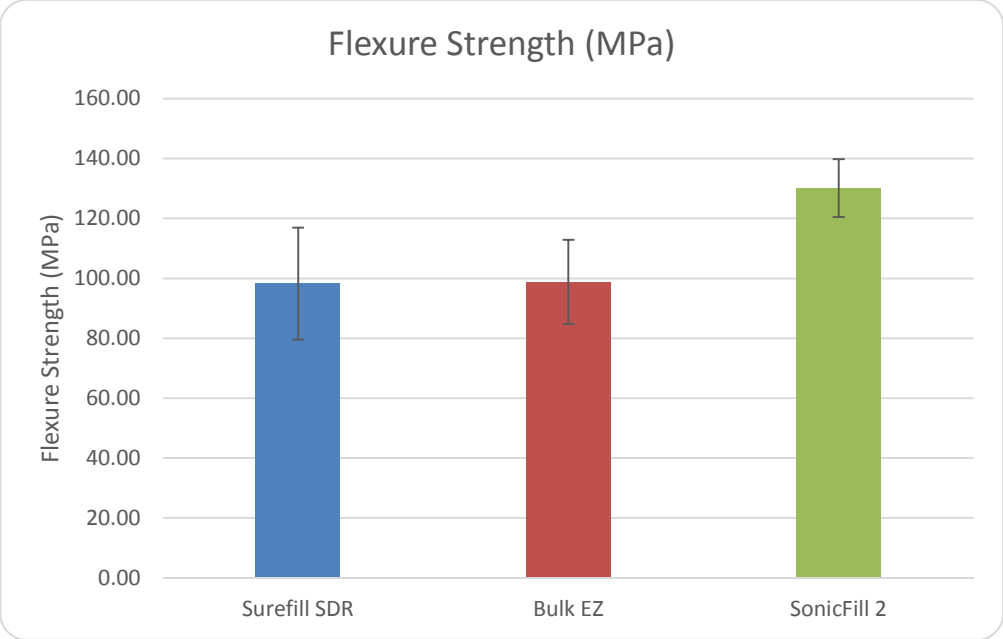
Flexure properties are determined in three point bending, following ISO4049.

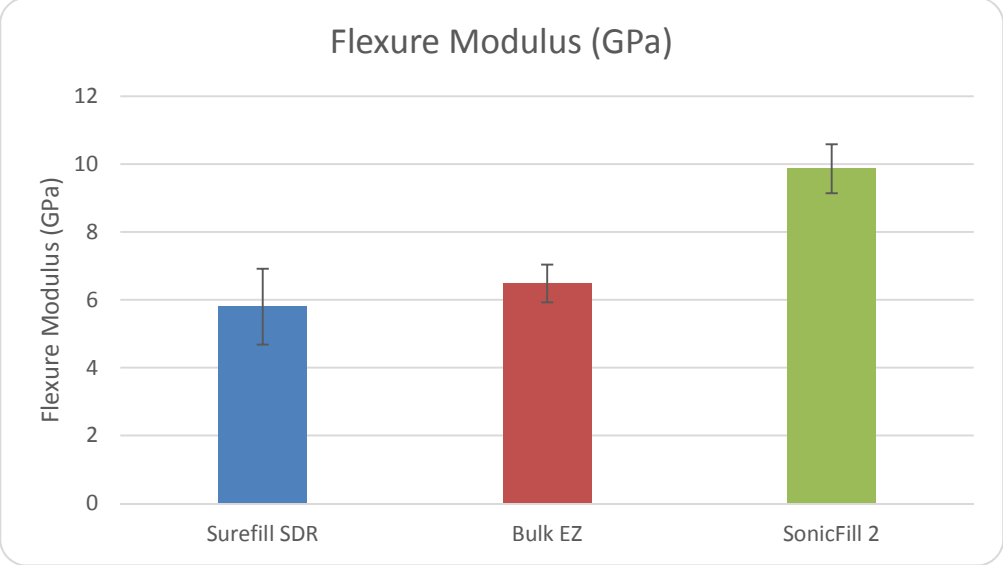
Test method: Specimens will be made in square glass rods with dimensions of 2 mm x 2 mm x 25 mm. The composites will be light-cured from the top and the bottom with a Demi light (560 mW/cm^2; Kerr), 9 mm tip, with stepped exposures of 20 seconds to cover the entire specimen. *Note: The light application will be delayed until 200 seconds for the Danville product to allow time for the self-cure reaction*. The specimens will be stored in water at 37°C for 24 hours and then tested in 3-point bending (20 mm span) on a universal testing machine at a cross-head speed of 0.25 mm/min. The flexure strength will be determined using the maximum load. Any evidence of plastic deformation will be noted. The flexure modulus will be determined from the initial slope of the force-deflection curve. The protocols will follow ISO 4049 for dental

restoratives. Ten specimens will be tested. Data will be presented as mean (with standard deviation). The results for the different composites are compared using ANOVA and Tukey's multiple comparison test (α=0.05).

Results: For both the flexure strength and modulus, the SonicFill2 material was the highest, and there was no difference between Surefill SDR and BulkEZ.

Note: there was a non-normal distribution of the data, due to at least one odd value for Surefill and BulkEZ, thus a ANOVA on ranks was conducted, followed by the Dunn's test to show the statistical differences.



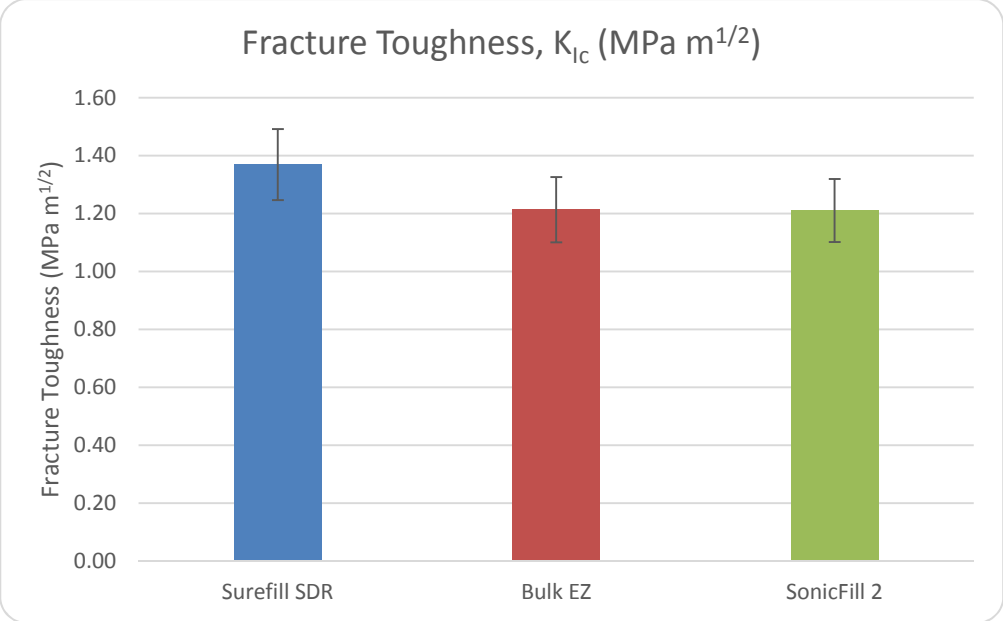


4. Fracture Toughness:

Fracture toughness will be testing on single-edge notched beams tested in three-point bending, following ASTM E-399.

Test method: Specimens will be made in stainless steel molds with dimensions 2.5 mm x 5 mm x 25 mm with a razor blade notch at the mid-span producing an a/w = 0.5. Specimens will be light-cured from the top and bottom as described for the flexure strength test, with stepped exposures to cover the entire specimen. *Note: The light application will be delayed until 200 seconds for the Danville product to allow time for the self-cure reaction*. The specimens will be stored in water at 37°C for 24 hours and then tested in 3-point bending (20 mm span) on a universal testing machine at a cross-head speed of 0.254 mm/min. The fracture toughness will be determined using the maximum load (unless there is evidence of plastic deformation, which is very unlikely). The protocol will follow ASTM E399 and has been used for years in our laboratory (Ferracane et al., 1987). Ten specimens of each composite will be tested. Data will be presented as mean (with standard deviation). The results for the different composites will be compared using ANOVA and Tukey's multiple comparison test ($p<0.05$).

Results: The fracture toughness of SDR Flow was the highest, being significantly greater than BulkEZ and Sonicfill 2, and there was no difference between the latter two.



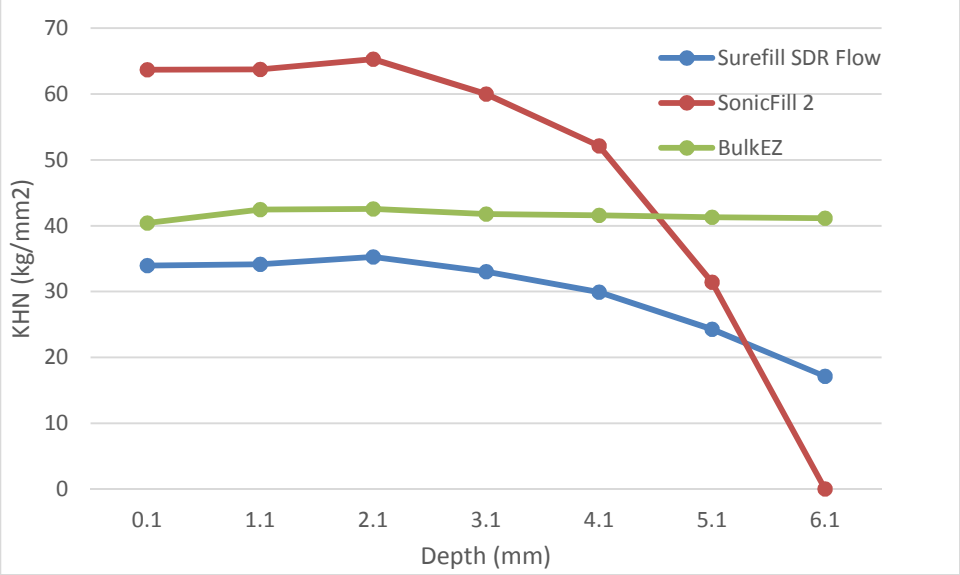
5. Depth of Cure – Tooth Model

A standardized, non-retentive class 2 cavity approximately 5 mm in depth x 7 mm in buccal-lingual dimension x 2 mm in medial-distal dimension will be prepared in a molar tooth. The preparation will be lightly lubricated with Vaseline, a circumferential metal Toffelmire matrix applied, and the restorations using the resin composite will be accomplished (n=10). Each restoration will be cured from the occlusal with a Demi curing light for 20 seconds. *Note: The*

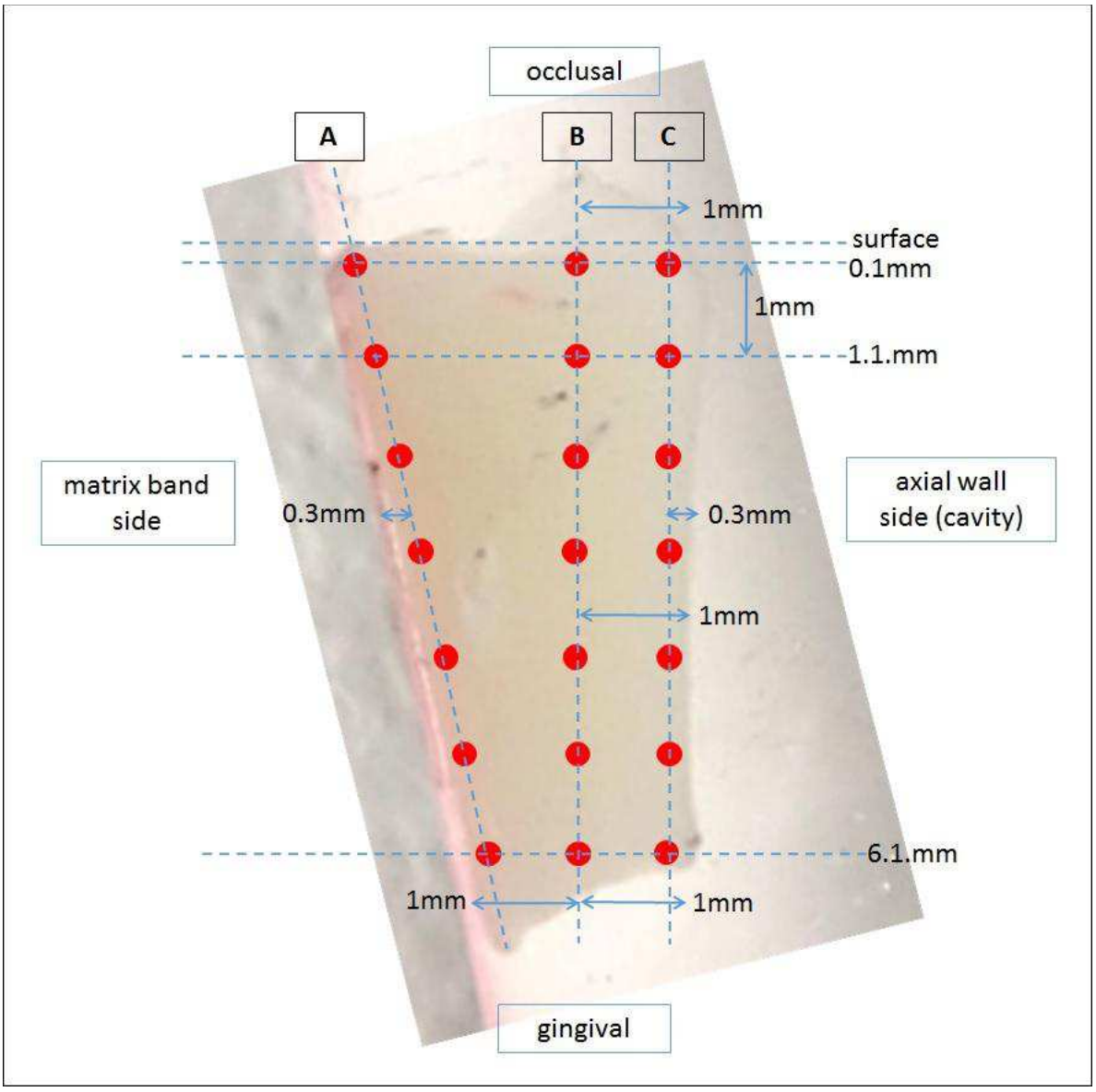
light application will be delayed until 200 seconds for the Danville product to allow time for the self-cure reaction.

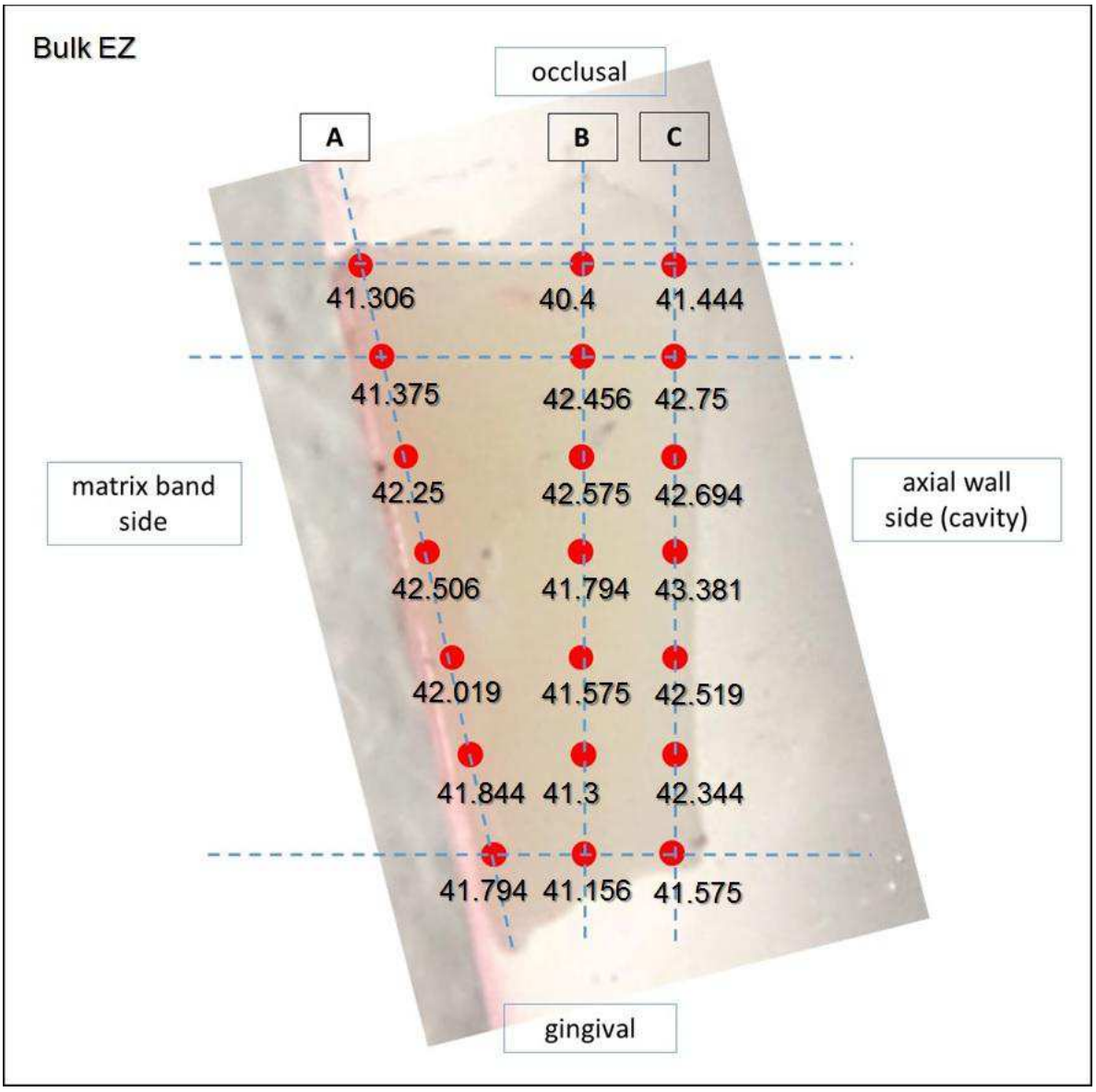
After curing, the composite restoration will be removed from the tooth preparation, embedded in epoxy, and sectioned through the middle in a mesial-distal orientation. The Knoop hardness will be determined in 1 mm increments starting 0.1 mm below the occlusal surface. The last Knoop hardness reading will be made 0.1 mm above (occlusal) to the gingival floor of the restoration. Three Knoop hardness measurements will be made at each depth and averaged for the value at each depth of each specimen. The results for the different composites will be compared using ANOVA and Tukey's multiple comparison test ($p<0.05$).

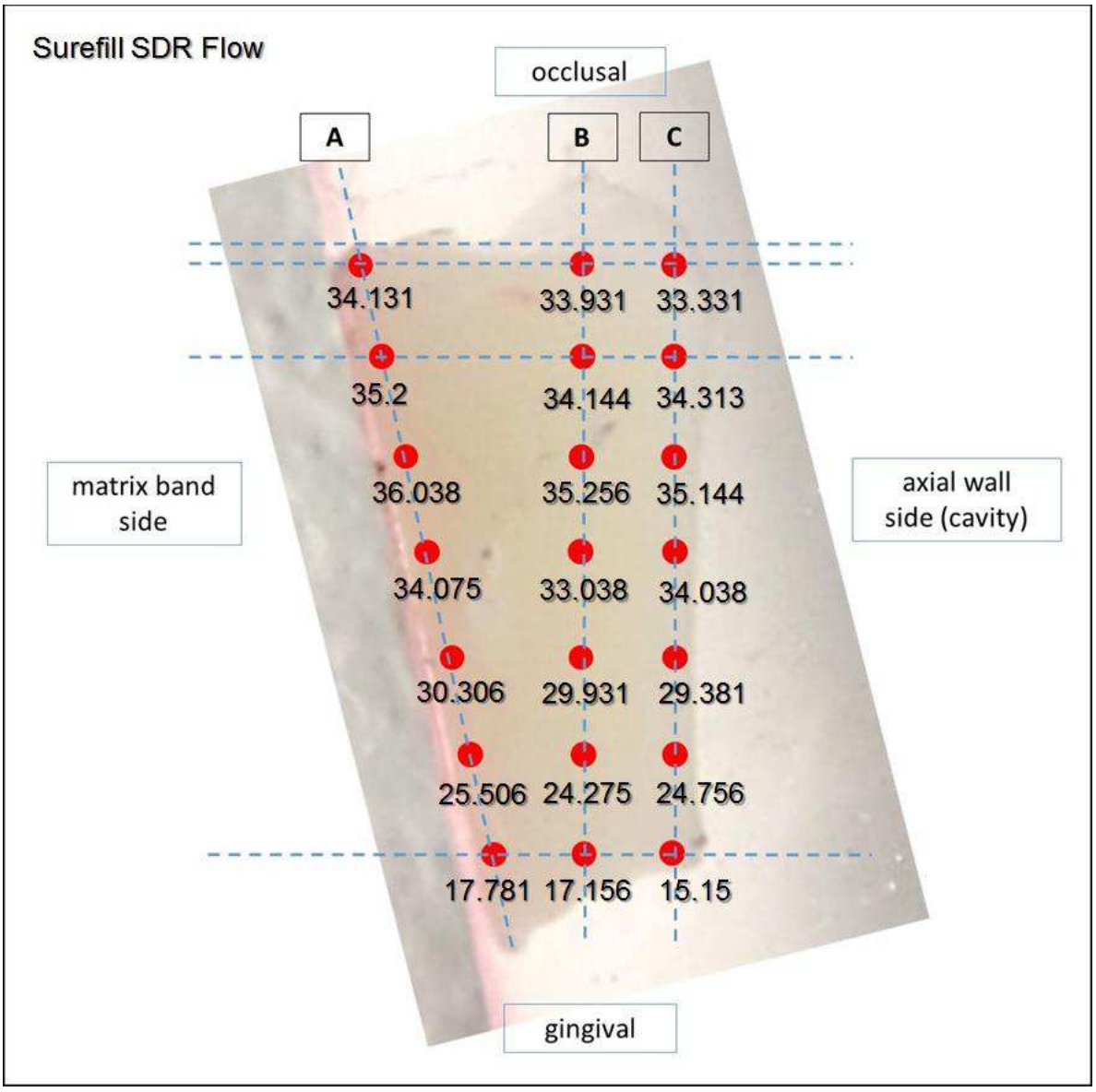
Results: The data shows that the hardness of SonicFill 2 was greater than that of BulkEZ, which was slightly greater than that of Surefill SDR Flow. The depth of cure (defined as the depth at which the hardness is still at least 80% of that at 0.1 mm) for BulkEZ was greater than 6.1 mm, as expected for a material that self-cures. The depth of cure for Surefill SDR and SonicFill 2 were both greater than 4 mm, but less than 5 mm. Sonicfill2 was too soft to measure at 6.1 mm, but Surefill SDR had cured enough to give hardness values at the maximum depth. Note though that the hardness of SonicFill 2 was still greater than that of Surefill SDR at 5.1 mm, though it had reached its defined depth of cure.

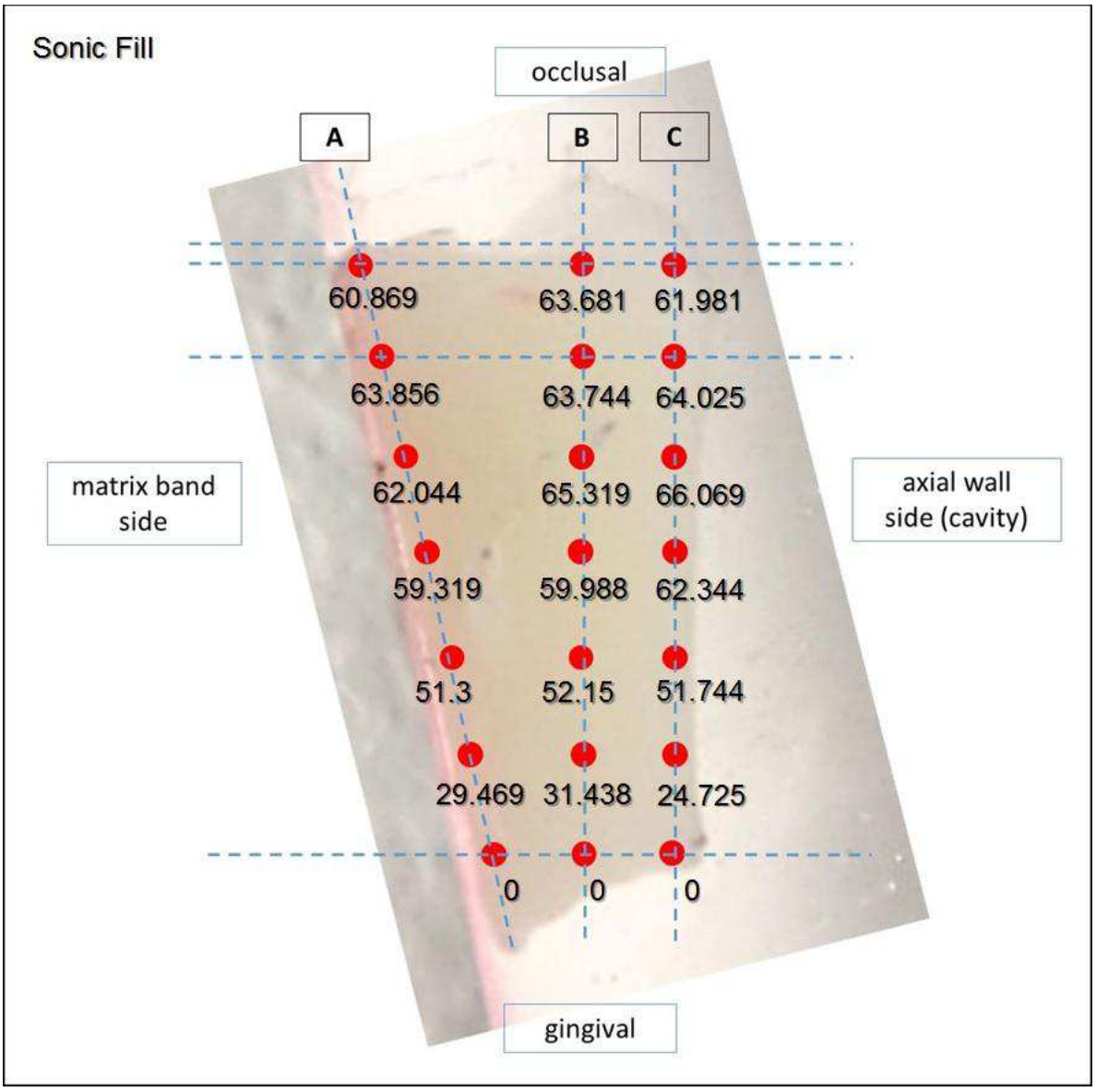


A pictoral summary of the data is shown below for the three composites. The first image shows where the hardness readings were taken on each specimen. The next three images shows the values for the three different composites in these regions.


occlusal
A
B
C
1mm
surface
0.1mm
1mm
1.1.mm
matrix band side
0.3mm
0.3mm
axial wall side (cavity)
1mm
6.1.mm
1mm
1mm
gingival


Bulk EZ
occlusal
A
B
C
41.306
40.4
41.444
41.375
42.456
42.75
42.25
42.575
42.694
42.506
41.794
43.381
42.019
41.575
42.519
41.844
41.3
42.344
41.794
41.156
41.575
matrix band side
axial wall side (cavity)
gingival


Surefill SDR Flow
occlusal
A
B
C
34.131
33.931
33.331
35.2
34.144
34.313
matrix band side
36.038
35.256
35.144
axial wall side (cavity)
34.075
33.038
34.038
30.306
29.931
29.381
25.506
24.275
24.756
17.781
17.156
15.15
gingival


Sonic Fill
occlusal
A
B
C
60.869
63.681
61.981
63.856
63.744
64.025
62.044
65.319
66.069
59.319
59.988
62.344
51.3
52.15
51.744
29.469
31.438
24.725
0
0
0
matrix band side
axial wall side (cavity)
gingival