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Effects of Various Surface Treatments and Cement Types on Tensile Bond Strengths of Cements
to Three Zirconia Materials

Surface and Cement Effects on Bond Strength to Zirconia

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Abstract

Effects of Various Surface Treatments and Cement Types on Tensile Bond Strengths of Cements to Three Zirconia Materials

Objectives: To evaluate the effects of various surface treatments and cement types on the tensile bond strength (TBS) of cements to three zirconia materials.

Methods: Three zirconia materials (KATANA Zirconia HTML, STML and UTML), 135 specimens each, were divided into three surface treatment groups: no air abrasion (NO), air abrasion with glass beads (GB) and air abrasion with aluminum oxide (AL). For each group, printed resin (Formlabs Grey Resin) was cemented to zirconia specimens using three cement types: RelyX Luting Plus (RXL), RelyX Unicem 2 (RXU) and PANA VIA V5 (PAN). A total of 405 cemented specimens (27 groups, N=15/group) were stored in distilled water at 37 °C for 24 hours and tested for TBS. The Data were analyzed with Kruskal-Wallis and Mann-Whitney U tests ($\alpha=0.05$).

Results: RXL showed the lowest median TBS of all three zirconia materials, which was statistically different from RXU and PAN irrespective of surface treatments ($P<0.05$). RXL exhibited mostly adhesive failures including pre-test failures. For HTML, RXU with AL surface treatment showed the highest median TBS ($P<0.05$). For STML, RXU with AL and PAN with GB or NO surface treatment had significantly higher median TBS than the other groups ($P<0.05$). For UTML, RXU with AL and PAN with AL, GB or NO surface treatment showed significantly higher median TBS ($P<0.05$) than the other groups.

Conclusions: Cement types had significant effects on TBS. RXL performed significantly less than RXU and PAN. Performance of RXU and PAN varied depending on zirconia materials and surface treatments.

Introduction

Polycrystalline ceramics (e.g., alumina or zirconia) were not practical prior to the availability of CAM technology because they were more difficult to process into complex shapes due to their strength¹. With continuous advancements in CAD/CAM technology, zirconia has been widely used as a restorative material in dentistry. It has the highest strength amongst all dental ceramics. According to the article on dentist material selection for single-unit crowns, the top three choices for posterior crowns are all-zirconia (32%), PFM (31%) and lithium disilicate (21%). The top three choices for anterior crowns are lithium disilicate (54%), layered zirconia (17%) and leucite-reinforced glass ceramic (13%)². Zirconia is also known to have a great long-term success rate. Zirconia cantilever resin-bonded fixed dental prostheses (RBFDP) yielded a 10-year survival rate of 98.2% and a success rate of 92.0%³.

Zirconia materials are divided into four generations according to their mechanical and optical properties⁴. The first generation, 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP), was introduced to the dental market more than 25 years ago⁴. The second generation, 3Y-TZP with reduced alumina content, was presented around 2013⁴. The third generation, 5 mol% partially stabilized zirconia (5Y-PSZ), was introduced in 2015 with the cubic content amounts to approximately 50%⁴. In 2017, the fourth generation, 4 mol% partially stabilized zirconia (4Y-PSZ), was presented with the reduced yttria content to improve the mechanical properties⁴.

Despite its favorable properties as restorative material, traditional zirconia's opacity has been a challenge to overcome. Full-contour zirconia crowns have been selected to restore mainly posterior areas. With the developments of more translucent zirconia (4Y-PSZ or 5Y-PSZ) within recent years, its application has broadened to restore highly esthetic anterior regions. Although

its translucency is improved, strength and toughness have shown to become diminished. Physical properties of translucent zirconia have been actively investigated by many researchers. Zirconia restorations are considered cementable with conventional cements due to their high flexural strength, but may need to be bonded if mechanical retention is limited. For strong and durable long-term resin bonds, it has been recommended to pretreat the intaglio surface of zirconia with air-particle abrasion and use adhesive resin luting cement with an application of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer⁵.

However, it was proposed that abrasion of the intaglio surface of cubic-containing zirconia with aluminum oxide may weaken them significantly because of no occurrence of transformation toughening⁶. The surface treatment with glass beads instead of aluminum oxide has been suggested for translucent zirconia, and it does not seem to result in a strength degradation⁷. A recent study found that airborne-particle abrasion with alumina had a weakening effect on 5Y zirconia materials and a slight strengthening effect on a 4Y zirconia compared to airborne-particle abrasion with glass beads⁸. This research evaluates the tensile bond strength of traditional zirconia and translucent zirconia, and the effect of different air-particle abrasion treatments and cement types on the tensile bond strength between zirconia and cement. Null hypotheses were as follows: 1) There would be no significant difference in the effect between the different generations of zirconia on the tensile bond strength; 2) There would be no significant difference in the outcome of surface treatments regarding the tensile bond strength; 3) Tensile bond strength would not be significantly affected by different cement types.

Materials and Methods

Three independent variables were tested in this study: zirconia material type, air-particle abrasion method and cement type. A total of 405 specimens were prepared. The specimens

were randomly divided into 27 groups of 15 specimens each. These were subjected to the following experimental protocols: 1) Three different zirconia material types: KATANA HTML (3Y-TZP), KATANA STML (4Y-PSZ), and KATANA UTML (5Y-PSZ) (Kuraray Noritake Dental Inc., Tokyo, Japan); 2) Three different surface treatments: No air-particle abrasion, air-particle abrasion with aluminum oxide, and air-particle abrasion with glass beads; 3) Three different cement types: RelyX Luting Plus Automix Cement (Resin Modified Glass Ionomer Cement) (3M ESPE, St Paul, MN, USA), RelyX Unicem2 Self-Adhesive Resin Cement (3M ESPE), and PANAVIA V5 (Dual-Cure Resin Cement) (Kuraray Noritake Dental Inc.) with CLEARFIL CERAMIC PRIMER PLUS (Kuraray Noritake Dental Inc.).

The zirconia material selected for this study was KATANA HTML (3Y-TZP) shade A2, KATANA STML (4Y-PSZ) shade A2, and KATANA UTML (5Y-PSZ) shade A2. The blocks were designed using Dassault Systèmes SolidWorks 3D CAD software (Dassault Systèmes SolidWorks Corporation, Waltham, MA, USA) and the file was imported into iCAM V5 smart milling software (imes-icore GmbH, Eiterfeld, Germany). A computer-aided manufacturing machine, CORiTEC 450i (imes-icore GmbH, Eiterfeld, Germany) was used to mill the zirconia blocks out of the zirconia discs.

Specimens were prepared by sectioning the zirconia discs into blocks (8 x 8 x 4 mm). Milled zirconia blocks were then polished with 1200-grit silicon carbide paper and cleansed with isopropyl alcohol and water. The blocks were dried and then sintered in a long cycle (VITA ZYRCOMAT 6000 MS, VITA North America, Yorba Linda, CA). After sintering was completed, specimens were cleansed again with isopropyl alcohol and underwent ultrasonic cleaning with distilled water for 5 minutes (Pro-Sonic 600 Ultrasonic Cleaner, Branson Ultrasonics, Brookfield, CT). Air-particle abrasion was conducted with 50-micron

aluminum oxide particles (Ivoclar Vivadent Blasting Compound, Ivoclar Vivadent North America, Amherst, NY) or 80-micron glass beads (Ivoclar Vivadent Glass Beads), Ivoclar Vivadent North America, Amherst, NY) for 10 seconds with a 30 psi (2 bar) air pressure at 10 mm distance (AccuFlo, COMCO Inc., Burbank, CA). All specimens including those with no surface treatment, underwent ultrasonic cleaning again with distilled water for 2 minutes and air dried thoroughly. A representative sample of each zirconia material with no air particle abrasion (Figures 1a-c, 2a, 3a and 4a), glass beads and aluminum oxide particle abrasion (Figures 2b-c, 3b-c, 4b-c), and a sample of aluminum oxide and glass beads particles (Figures 5a-b) were examined using a scanning electron microscope (JSM-IT500 InTouchScope Scanning Electron Microscope, JEOL USA, Inc., Peabody, MA).

Printed gray resins (Formlabs, Somerville, MA, USA) with 3 mm in diameter was air abraded with 50-micron aluminum oxide for 10 seconds with a 30 psi (2 bar) air pressure at 10 mm distance. After air-particle abrasion, they were cemented to zirconia specimens using the three different cements. The luting and bonding protocols followed the manufacturers' instructions. A representative sample of each zirconia material with and without surface treatments was examined using a scanning electron microscope. To determine the baseline area surface roughness values (S_a in μm^2), the specimens were measured with a noncontact optical profilometer (3D Laser Scanning Confocal Profilometer, Keyence), and the data was analyzed by its proprietary software (Figure 6).

A total of 405 cemented specimens (27 groups, 15 specimens per group) were stored in distilled water at 37 °C for 24 hours and tested for tensile bond strength in a universal testing machine, (Instron Type 5943; Figure 7), at a crosshead speed of 1 mm/min until failure. Tensile bond strength value in megapascals (MPa) was calculated from the peak load of failure

(Newtons) divided by the surface area. A Shapiro-Wilk test determined that the data was not normally distributed ($P < 0.05$). A median and interquartile range (IQR) were determined for each group. All specimens were inspected to determine failure modes utilizing cement remnant index (CRI) scores (Tables 1 and 2). Data were analyzed with Kruskal-Wallis and Mann-Whitney U tests ($\alpha = 0.05$) using IBM SPSS Statistics 27 (IBM, Chicago, IL)

Results

Rely X Luting Plus showed the lowest median tensile bond strength of all three zirconia materials, which was statistically different from Rely X Unicem 2 and Panavia V5 irrespective of surface treatments ($P < 0.05$). Rely X Luting Plus and Rely X Unicem 2 exhibited lower cement remnant index scores whereas majority of Panavia V5 exhibited higher cement remnant index scores as shown in Table 2. For HTML, Rely X Unicem 2 with aluminum oxide air abrasion surface treatments showed the highest median tensile bond strength ($P < 0.05$). For STML, Rely X Unicem 2 with aluminum oxide air abrasion and Panavia V5 with glass beads air abrasion or no surface treatment had significantly higher median tensile bond strength than the other groups ($P < 0.05$). For UTML, Rely X Unicem 2 with air abrasion with aluminum oxide and Panavia V5 with aluminum oxide or glass beads air abrasion or no surface treatment showed significantly higher median tensile bond strength ($P < 0.05$) than the other groups (Table 3). Aluminum oxide air abrasion groups had significantly higher surface roughness ($P < 0.05$) compared to the other groups for all three zirconia materials (Figure 4).

Discussion

An air-particle abrasion with aluminum oxide and utilization of self-cure or dual-cure composite resin cement with the application of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer have been recommended for strong and durable long-term bonds to zirconia⁸.

While full coverage zirconia restorations can be luted with an adequate abutment height, the results from this study indicate the initial tensile bond strength between zirconia and luting cement may not be as strong as self-cure or dual-cure resin cements.

For glass beads air-particle abrasion and no surface treatment groups, the median tensile bond strength of both STML and UTML was significantly higher than HTML when Panavia V5 was applied. For all three zirconia materials with either glass beads air-particle abrasion or no surface treatment, the application of PANAVIA V5 showed a higher median tensile bond strength than other cement groups.

Panavia V5 had a higher mean cement remnant index score of 2.1 compared to Rely X Unicem 2 (mean CRI of 1.1) and Rely X Luting Plus (mean CRI score of 1.2). Specific adhesive monomers that chemically bond to zirconia are necessary for long-term stable bonds⁹. The application of 10-MDP primer as a part of the bonding protocol for Panavia V5 may have contributed to a higher mean CRI score than the other cement groups.

An aluminum oxide air-particle abrasion demonstrated surface decontamination, nano roughness creation, and aluminum oxide particles embedment in zirconia surface for better chemical and micro-mechanical bonds¹⁰. With an aluminum oxide air-particle abrasion and the application of self-cure or dual-cure resin cements suggested to have a higher initial tensile bond strength between zirconia and cement compared to the application of luting cement. However, the long-term tensile bond strength needs to be evaluated to assess the stability.

Conclusion

Cement types had significant effects on the tensile bond strength. Rely X Luting Plus had significantly less tensile bond strength than Rely X Unicem 2 and Panavia V5. Surface roughness with aluminum oxide abrasion showed significantly higher than glass beads air

abrasion or no surface treatment for all three zirconia materials. Performance of Rely X Unicem2 and Panavia V5 varied depending on zirconia materials and surface treatments.

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Mr. James Pizzini, Biomedical Engineer, Medical CAD/CAM Laboratory

Minju Yi, Capt, USAF, DC

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Tables

Cement Remnant Index (CRI)	
Score	Quantity of Cement Remaining on Zirconia
0	No Cement on Zirconia
1	< 50% on Zirconia
2	≥ 50% on Zirconia
3	All Cement on Zirconia

Table 1. Cement Remnant Index (CRI) Score.

Cement Type	Total Number and Percentage of Each CRI Score			
	0	1	2	3
RMGI (RXL)	11 (8.2%)	91 (67.4%)	33 (24.4%)	0 (0%)
Unicem (RXU)	31 (23%)	60 (44.4%)	42 (31.1%)	2 (8.9%)
Panavia (PAN)	0 (0%)	1 (0.7%)	122 (90.4%)	12 (8.9%)

Table 2. Cement Remnant Index (CRI) Score Distribution for Each Cement.

Surface Treatment	Cement Type	Tensile Bond Strength (MPa)		
		Median (IQR)		
		Zirconia Type		
		HTML	STML	UTML
None (NO)	RMGI (RXL)	0.07 (0.21) Aa	0.00 (0.06) Aa	0.02 (0.06) Aa
	Unicem (RXU)	1.25 (1.55) Ab	0.89 (1.18) Ab	0.75 (0.74) Ab
	Panavia (PAN)	2.88 (2.60) Ac	5.94 (4.36) Bc	6.01 (2.27) Bc
Glass Beads (GB)	RMGI (RXL)	0.03 (0.05) Aa	0.00 (0.04) Aa	0.00 (0.01) Aa
	Unicem (RXU)	2.10 (0.94) Bb	1.06 (0.76) Ab	1.64 (0.78) Bb
	Panavia (PAN)	2.17 (2.76) Ab	6.25 (3.35) Bc	7.74 (3.08) Bc
Aluminum Oxide (AO)	RMGI (RXL)	0.16 (0.35) Aa	0.43 (0.49) Aa	0.57 (0.69) Aa
	Unicem (RXU)	8.82 (3.05) Bc	7.72 (2.27) ABc	6.73 (2.29) Ab
	Panavia (PAN)	3.27 (2.20) Ab	2.28 (3.51) Ab	5.74 (3.10) Bb
Based on type of Surface Treatment, groups with the same upper case letter per row or lower case letter per column are not significantly different (P>0.05).				

Table 3. Median and Interquartile Range (IQR) of Tensile Bond Strength (MPa) for Each Group.

Figures

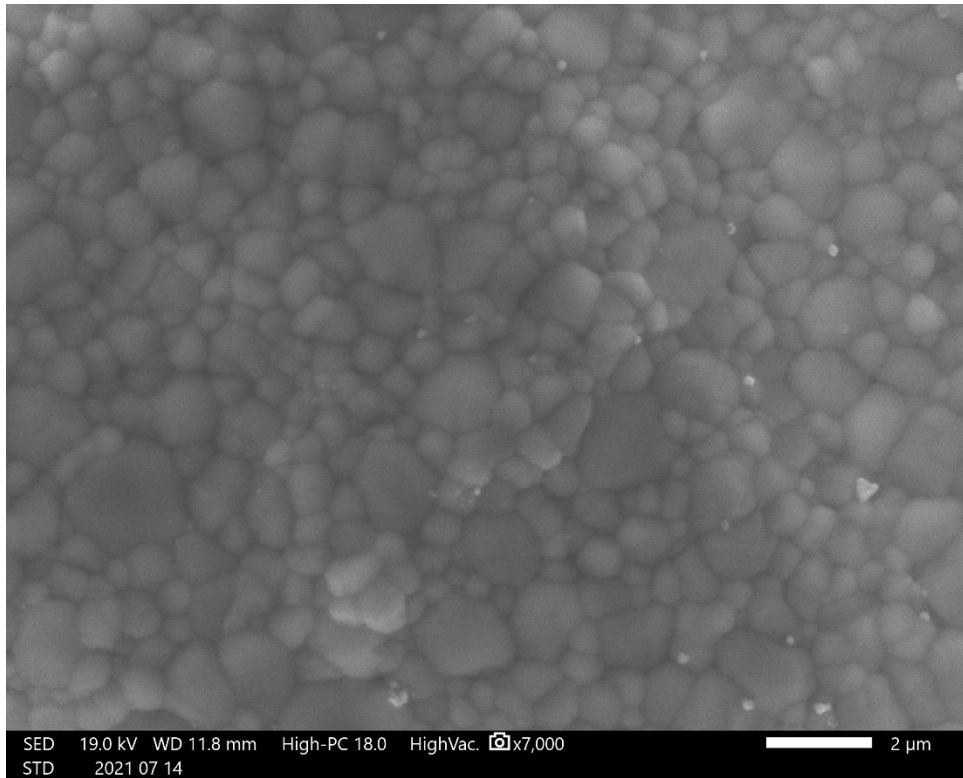


Figure 1a. SEM Image (x7,000 / 2 μm) of HTML with no air-particle abrasion.

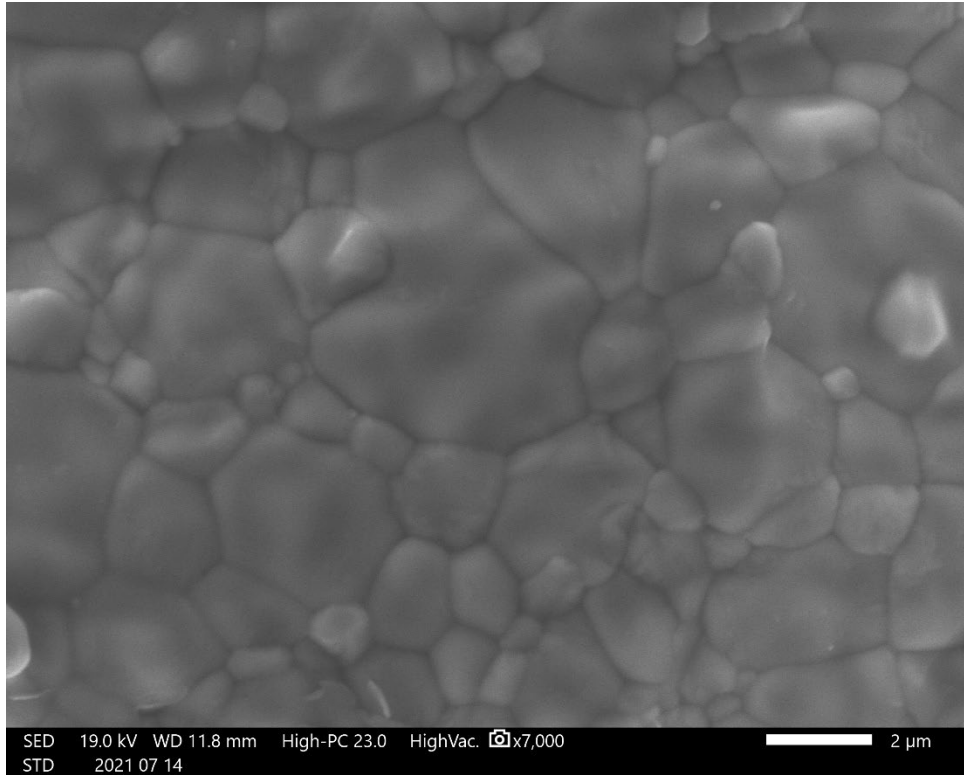


Figure 1b. SEM Image (x7,000 / 2 μm) of STML with no air-particle abrasion.

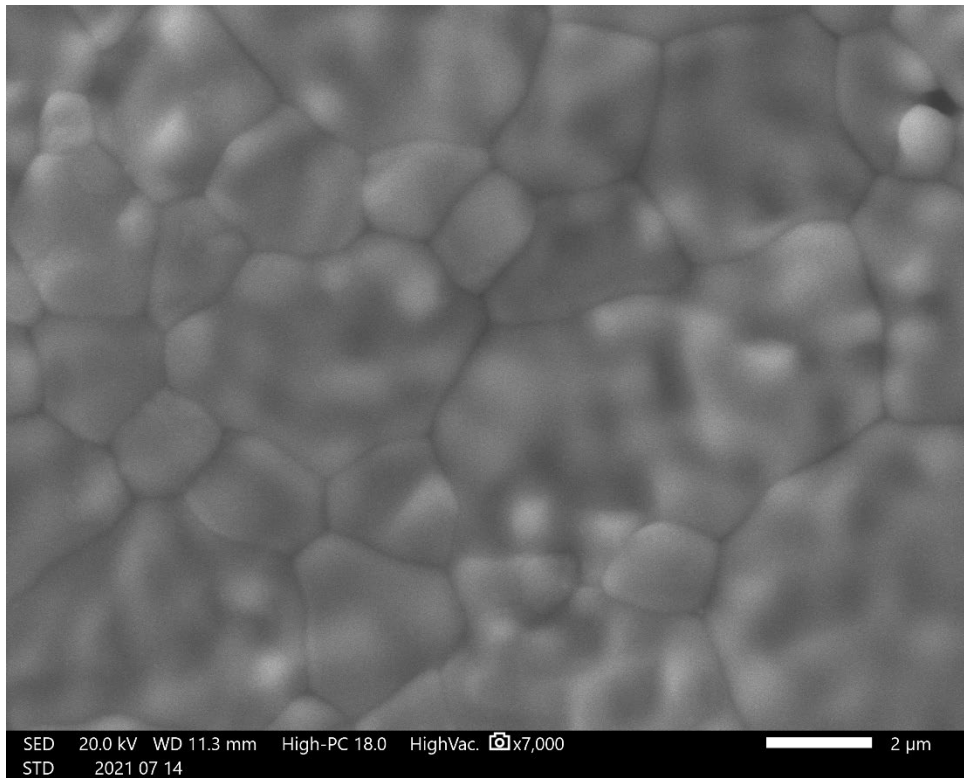


Figure 1c. SEM Image (x7,000 / 2 μm) of UTML with no air-particle abrasion.

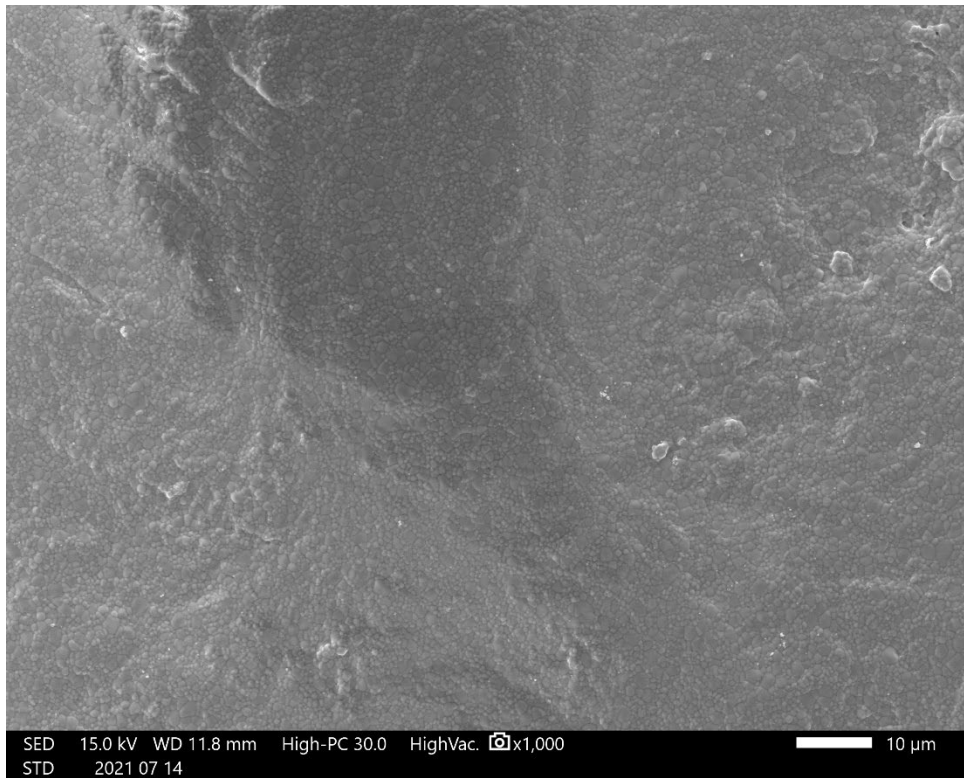


Figure 2a. SEM Image (x1,000 / 10 μ m) of HTML with no air-particle abrasion.

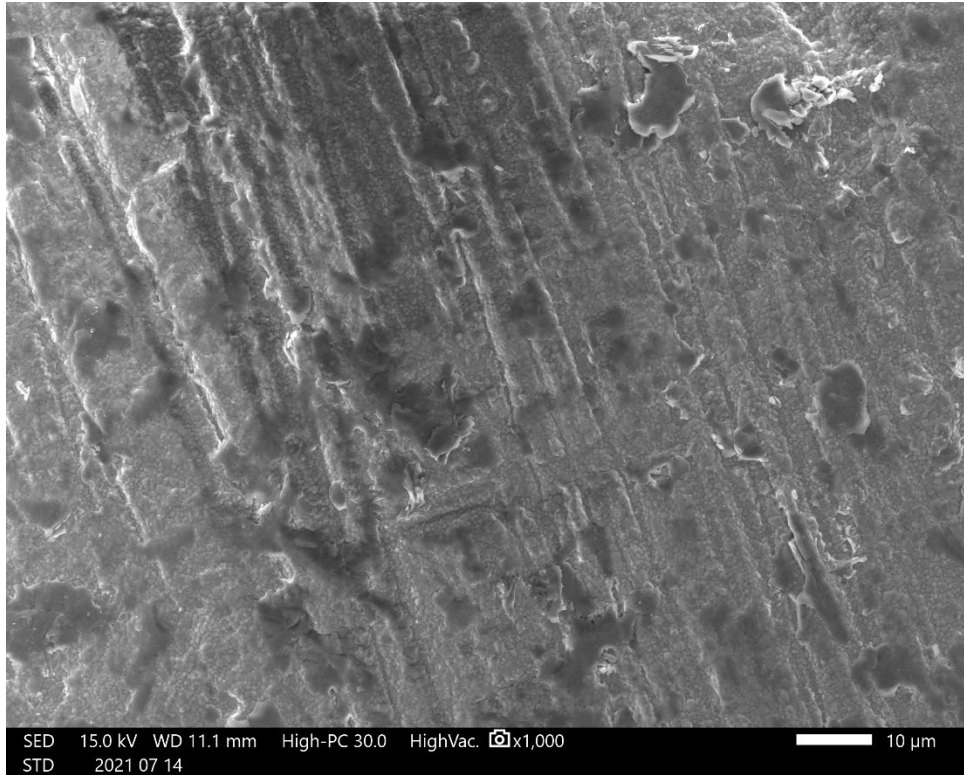


Figure 2b. SEM Image (x1,000 / 10 µm) of HTML with glass beads air-particle abrasion.

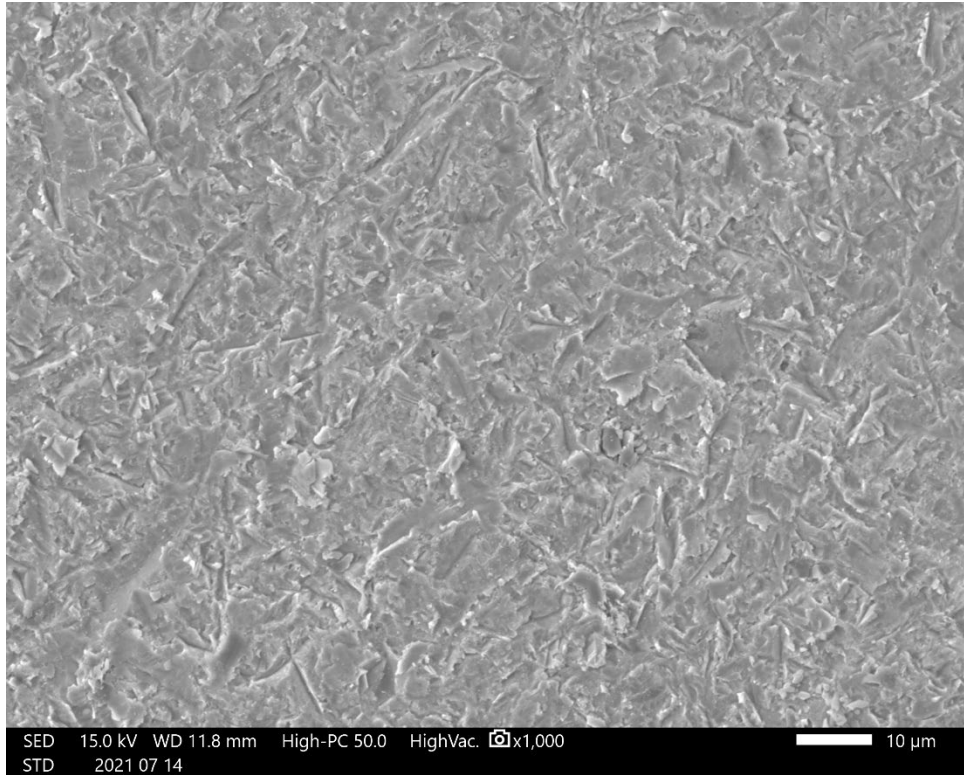


Figure 2c. SEM Image (x1,000 / 10 μm) of HTML with aluminum oxide air-particle abrasion.

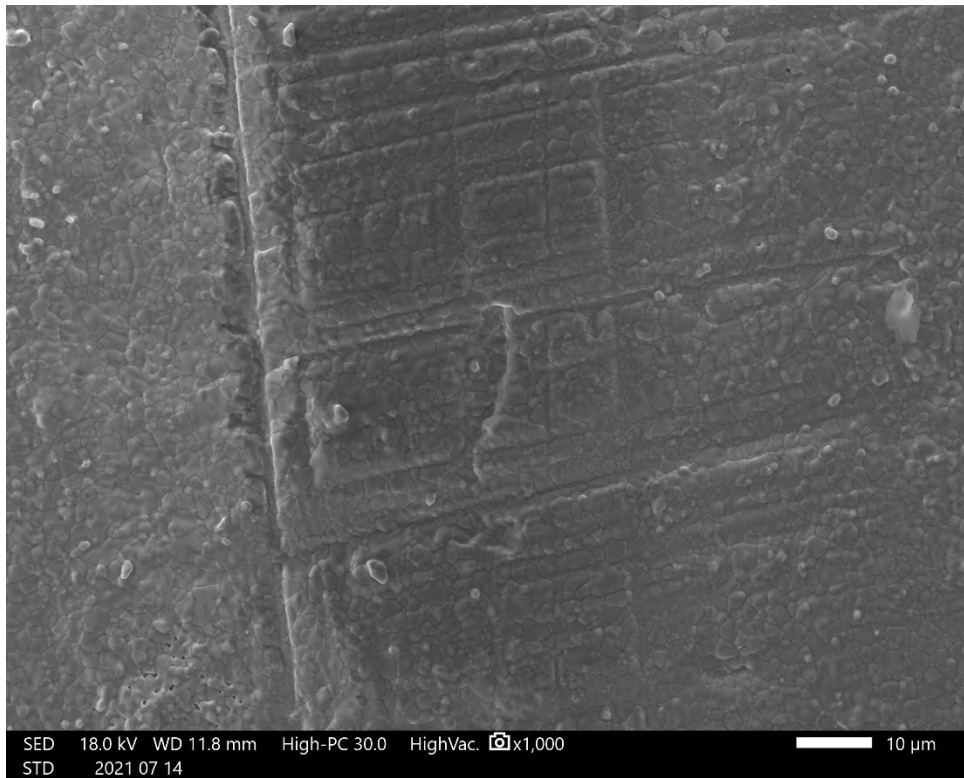


Figure 3a. SEM Image (x1,000 / 10 μ m) of STML with no air-particle abrasion.

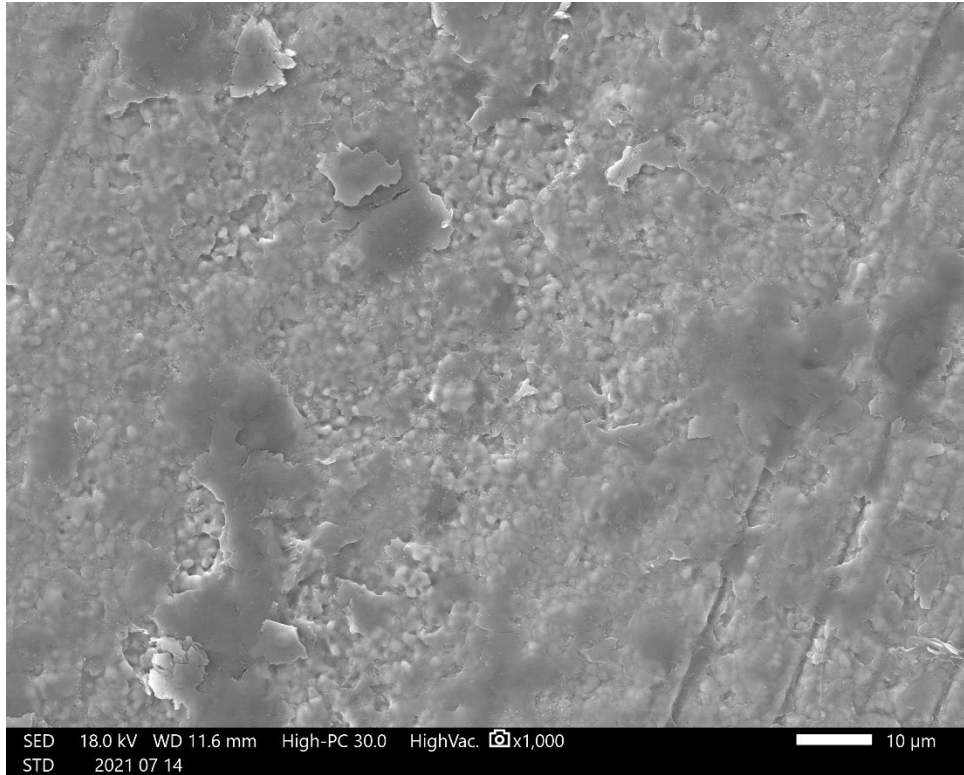


Figure 3b. SEM Image (x1,000 / 10 μ m) of STML with glass beads air-particle abrasion.

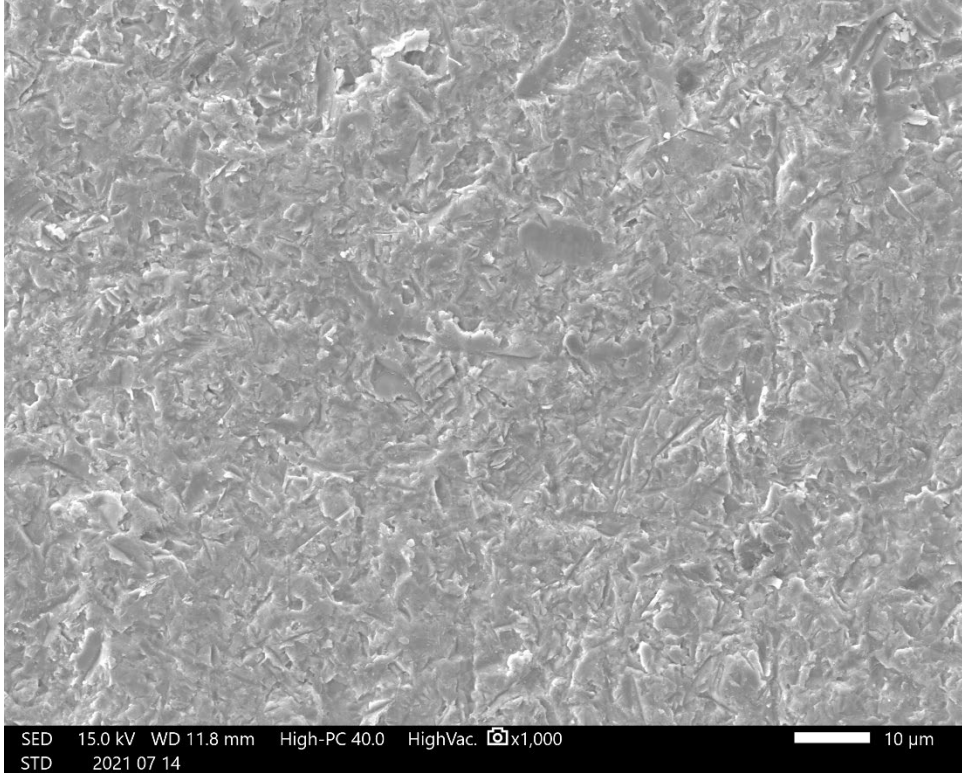


Figure 3c. SEM Image (x1,000 / 10 μm) of STML with aluminum oxide air-particle abrasion.

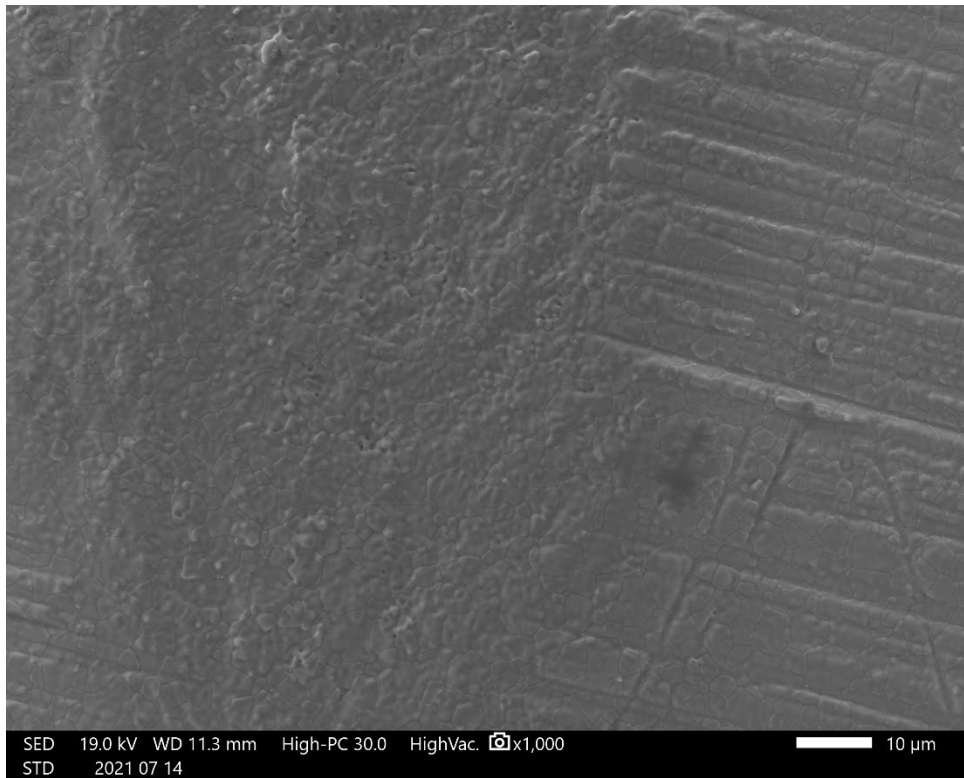


Figure 4a. SEM Image (x1,000 / 10 μ m) of UTML with no air-particle abrasion.

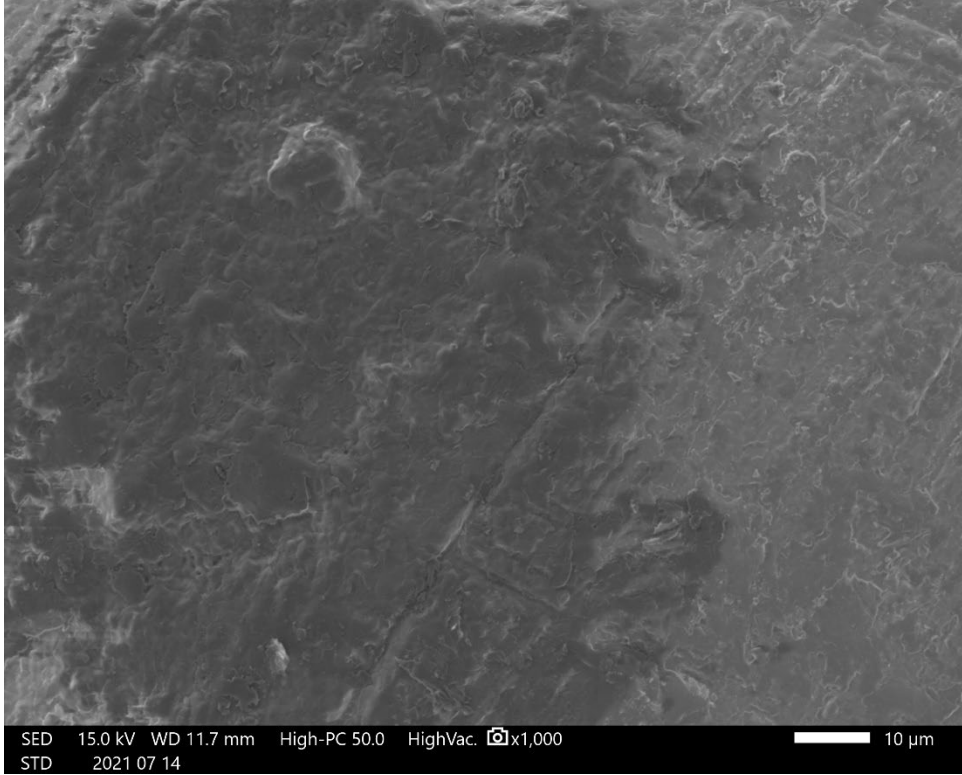


Figure 4b. SEM Image (x1,000 / 10 μ m) of UTML with glass beads air-particle abrasion.

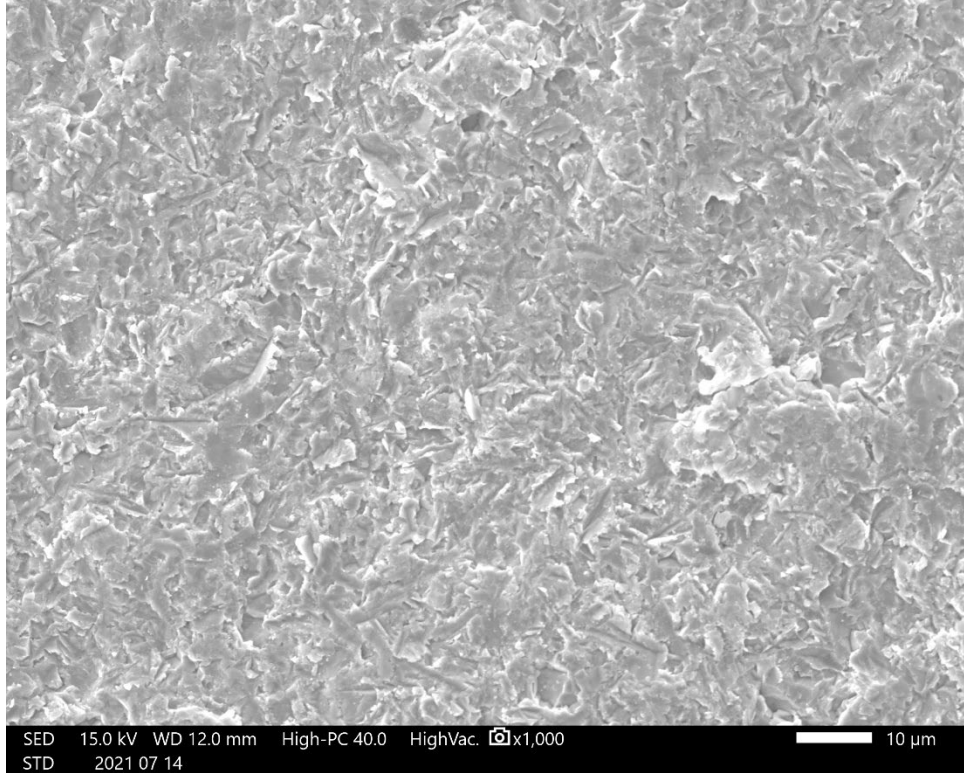


Figure 4c. SEM Image (x1,000 / 10 μm) of UTML with aluminum oxide air-particle abrasion.

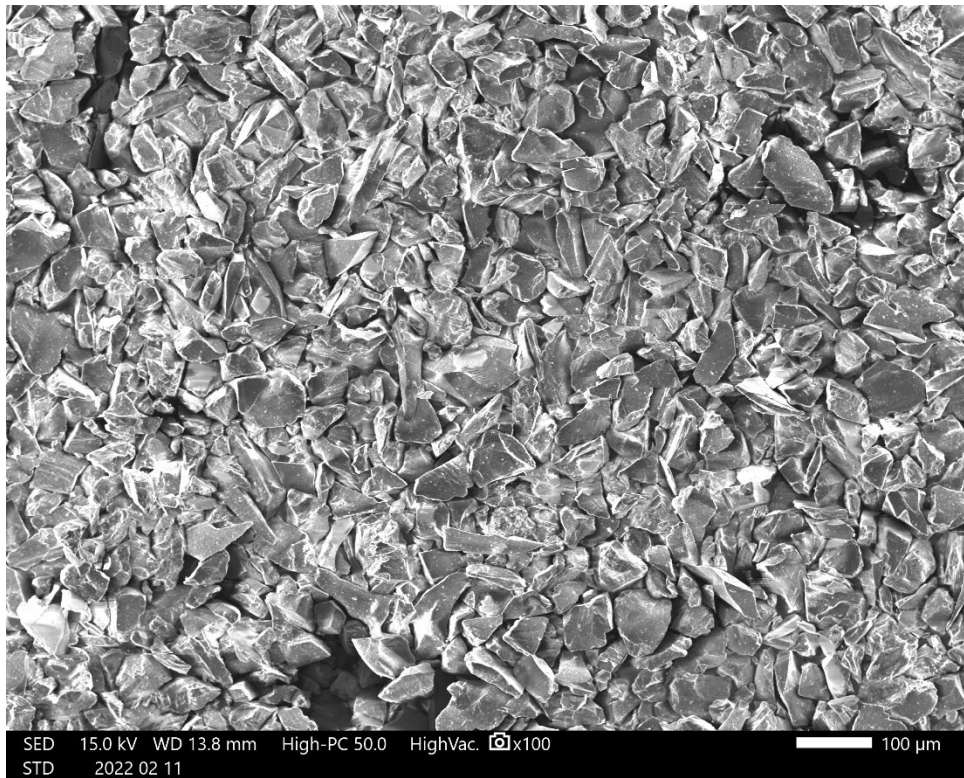


Figure 5a. SEM Image (x100 / 100 μm) of 50-micron aluminum oxide particles.

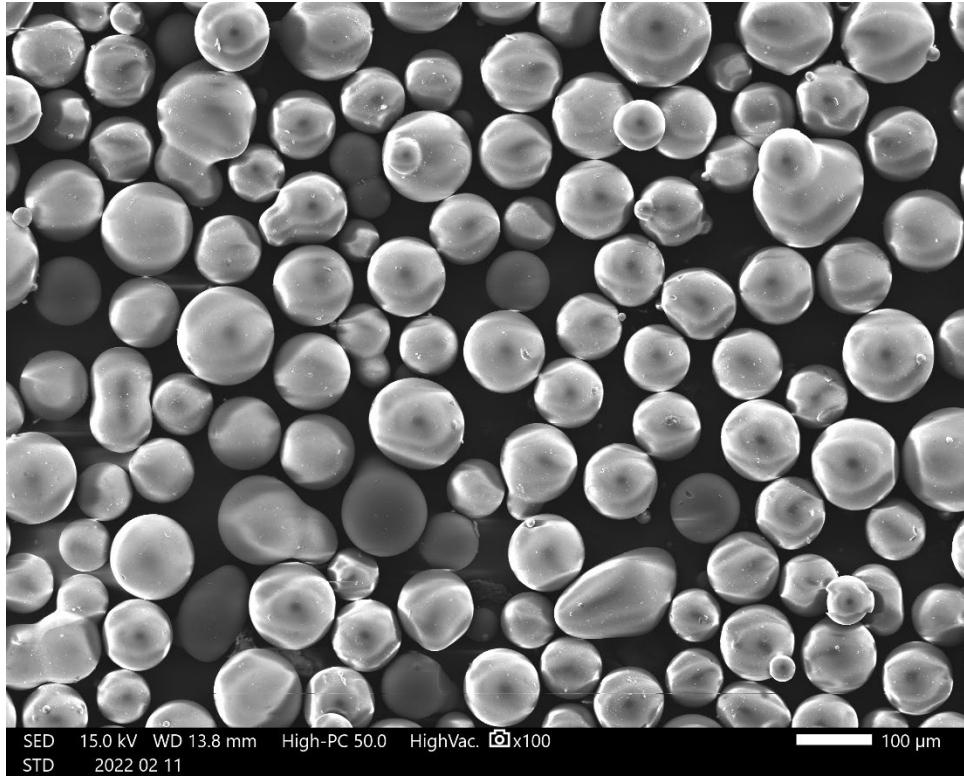


Figure 5b. SEM Image (x100 / 100 μ m) of 80-micron glass beads particles.

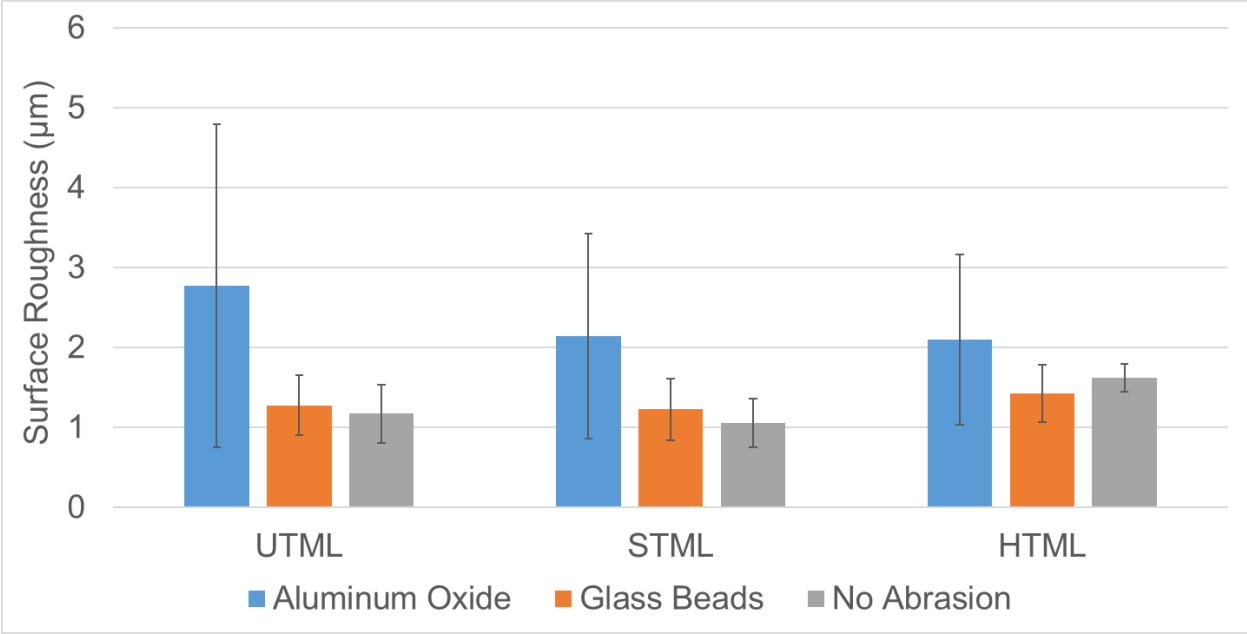


Figure 6. Surface Roughness (μm) of three zirconia materials with three different surface treatments.



Figure 7. The specimen (Staged for Photo).