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The Contribution of Boron on Body-Glaze Interactions

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The practice of glazing ceramics is almost as ancient as ceramics themselves. Glazes are characterized by there strong colors, textures, and glass like appearance. Until recently (2006) it was thought that the effects of the interactions between ceramic bodies and glazes were insignificant, however, recent work by Rein, Goldberg, Finkelnburg, Carty, and Lee has shown otherwise. This work has shown that there are significant changes to both the body and the glaze after firing, and has mapped the relationship between sintering time and glaze penetration depth. This thesis is built off of work done by Rein and Goldberg, which investigates the interactions between the ceramic body and the glaze, specifically the glaze penetration into the body. The hypothesis of this work was that adding boron to the glaze would increase the body-glaze interactions, specifically the glaze penetration depth.

The work done by Rein established that there was a relationship between sintering time and glaze penetration. As sintering time was increased, so too did glaze penetration, following a power-log relationship. Furthermore, Goldberg's work established that glazes composed of similar constituents, but in different ratios, did not affect the penetration depth of the glaze. For this thesis, the affect of boron on the body glaze interactions was investigated. It has been shown that boron acts as a high temperature viscosity modifier, that is, that at elevated temperatures boron will reduce the viscosity of a glass in comparison to a similar glass without boron. It is hypothesized that this reduction in viscosity will then lead to an increase in ionic diffusion of the glaze, leading to a greater penetration depth with respect to sintering time. Also tested by this thesis was the affect of the level of boron within the glaze. Assuming that boron will have an affect on the glaze, it has been hypothesized that increasing the level of boron within the glaze will further increase the body-glaze interactions.

To test this hypothesis, an experiment was created. Two glaze compositions were created with different boron levels. The two levels selected were 0.2 B_2O_3 (boron trioxide, a common boron constituent) and 0.5 B_2O_3. These numbers refer to the amount of boron on a molar ratio basis. The molar ratio basis is a way to create glazes and ceramic bodies with respect to their most basic elemental constituents.

Ceramic glazes are broken up into the ratio of alumina, silica, and fluxes that make up the glaze. However, it is generally not possible to simply pick a recipe and add X amount of silica or Y amount of flux. Common ceramic raw materials, such as clay, are a combination of all of these materials. Therefore, the molar ratio basis allows one to batch glazes or bodies with respect to the elements that the raw materials are made of, rather than assuming that the raw materials will never have any variations. Table I shows the rest of the glaze composition common to both boron containing glazes.

Table I: Elemental constituents of the glazes on a molar ratio basis.

	B_2O_3 Level	
Constituent	**0.2**	**0.5**
KNaO (R_2O)	0.3	0.3
CaO (RO)	0.7	0.7
Al_2O_3	0.4	0.4
SiO_2	3.0	3.0
B_2O_3	0.2	0.5

It is important to note that these glazes contained a large amount of calcium. This calcium is what was used to measure the glaze penetration depth into the body, as the body contained no calcium.

After these glazes were batched, a body also needed to be prepared. A typical porcelain body was used with zircon added. This combination was proved by Rein to produce a body that was capable of marking the original glaze-body interface, due to the fact that zircon is insoluble in both the glaze and the body. This means that the glaze penetration can then be reliably measured using both Backscatter Electron and Wavelength Dispersive Spectroscopy Analysis. Backscatter Electron analysis relates the molecular weight of what it is scanning to a grayscale image. In relation to what is in the sample, the lowest molecular weight will be shown as either a dark gray or even black, and the lighter the spot is, the higher the molecular weight of that area. As zircon's molecular weight is much higher than that of the porcelain body that it is in, it shows up on the backscatter image as bright white spots. Wavelength Dispersive Spectroscopy uses X-rays that are projected at the sample,

which are then diffracted back at a sensor. This sensor is set to "count" the number of times an X-ray of a specific wavelength (corresponding to each individual element) is detected, offering a concentration for each scanning site. These scans are then compiled to form an image that shows relative concentrations of that element. Figure 1 shows examples of both BSE and WDS scans for calcium and zircon, the mapping elements used in this work.

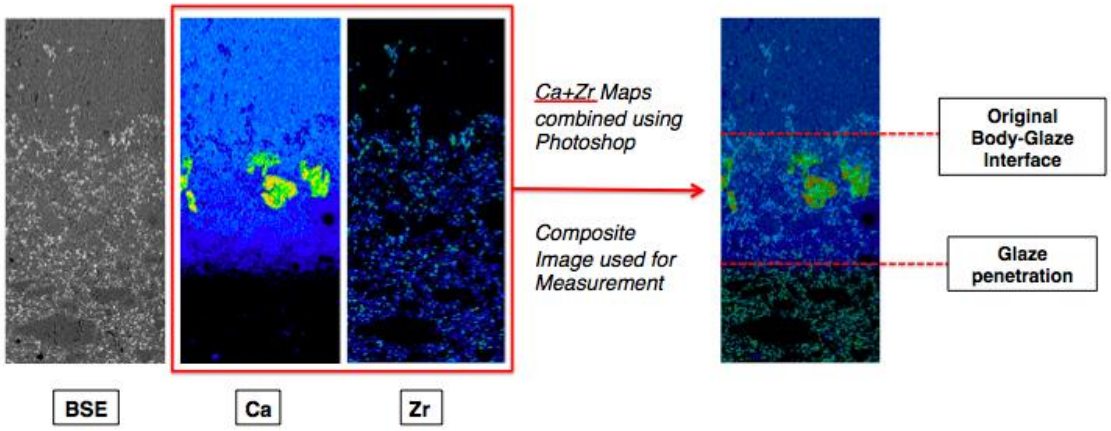

Figure 1: BSE and WDS scans for illustrative purposes, as well as the superimposed image used for glaze penetration measurement.

After preparing both the body and glazes, samples were made and fired. Three different temperatures (1150°C, 1250°C , and 1300°C) and four different dwell times (1.0, 3.2, 10, and 32 hours) were investigated, for a total of 24 samples. Figure 1 also outlines the image preparation used to be able to take measurements. Using the Zircon map, the original body-glaze interface can be determined, and using the calcium map, the end of the calcium, and therefore the glaze, can be determined. By measuring from the original body-glaze interface to the end of the glaze the glaze penetration can be determined. Due to fluctuations in the zircon levels, an average of over twenty measurements was taken. This data was compiled, and conclusions could be made.

Two important conclusions could be deduced from the data gathered in this study. The first, and most important, relates to the original hypothesis. When

compared to the previous data presented by Rein, the addition of boron was in fact found to increase the glaze penetration into the body. Figure 2 shows the comparison of both data sets.

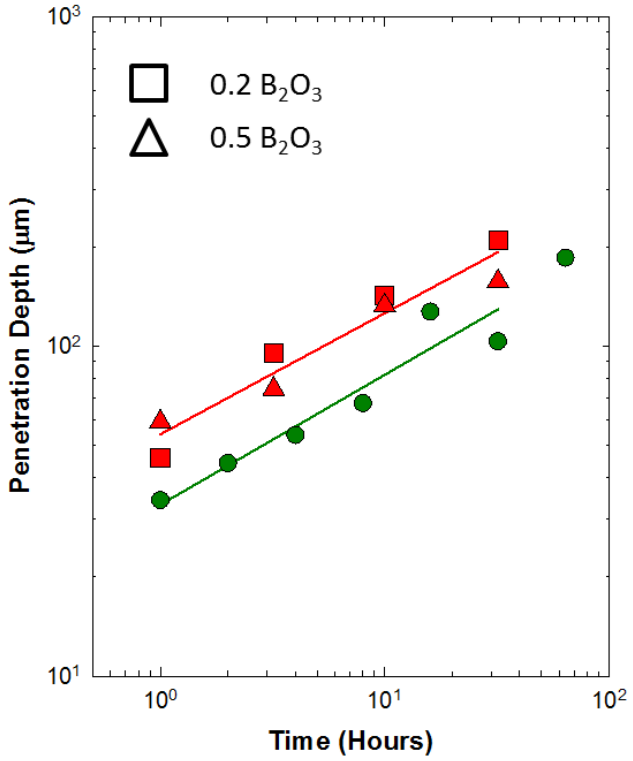

Figure 2: Comparison of data found in this work to the work presented by Rein.

The data itself is scattered, so to better approximate the trend regression lines were fit to the data points. As can be seen, the data representing the work done in this thesis correlates with higher depth penetration, and is also very close to parallel to the line representing Rein's work. This work demonstrates that in needing a given glaze penetration depth, glazes containing boron could be fired for a shorter time.

The second conclusion to be made from the work is that while the affect that boron-containing glazes has is evident, the amount of boron within the glaze seems to make negligible difference. Figure 3 plots the data found with respect to both the 0.2 B_2O_3 levels and 0.5 B_2O_3 levels.

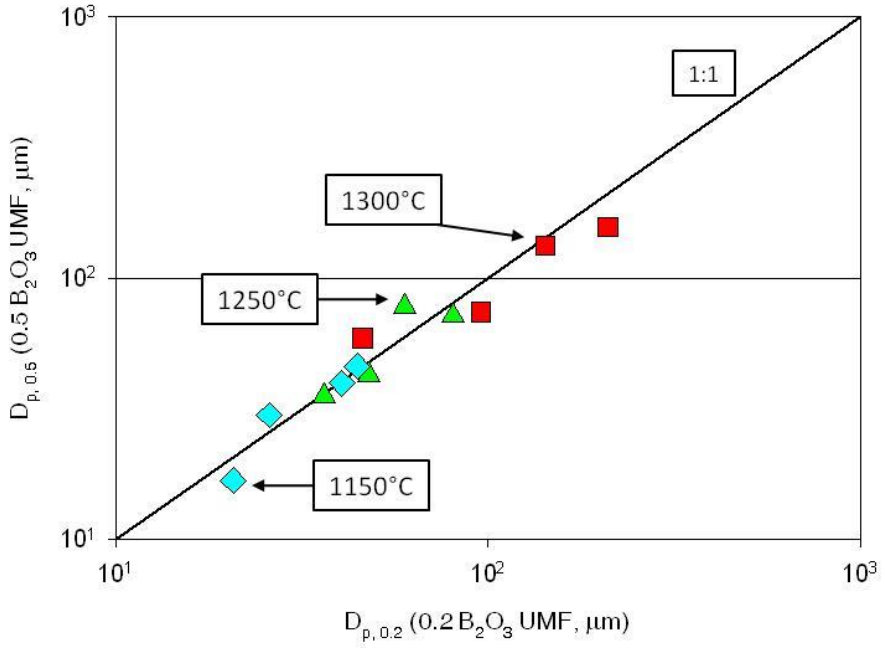

Figure 3: Comparison of data from both 0.2 B_2O_3 levels and 0.5 B_2O_3 levels.

The line extending from the bottom left corner of the plot to the upper right corner of the plot represents a 1:1 correlation between the two axes, in this case 0.2 B_2O_3 is plotted on the X-axis and 0.5 B_2O_3 is plotted on the Y-axis. If the data points were directly on the line, it would mean that the data from both axes were exactly the same. If the data points were shift preferentially towards one side of the line, which was what was hypothesized, that would mean that there was an appreciable difference between the two boron levels. However, what is seen is that the points are scattered on either side of 1:1 line, meaning that there is no significant difference between the two boron levels.

The purpose of the work done in this thesis was to prove or dis-prove the hypothesis that adding boron to a ceramic glaze increases the glaze penetration into to ceramic body. Through this study the hypothesis was proved to be correct, that boron does in fact increase body-glaze interactions, and thus increase the glaze penetration.

References

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by

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A Thesis

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Abstract

The presence of boron in a glaze will increase the body-glaze interactions, specifically the glaze penetration depth. Adding boron to a glaze decreases the viscosity and thus increases ionic diffusion rates. An increase in diffusion rate should result in an increase in body-glaze interaction. To evaluate this hypothesis, two glazes with different boron levels were batched and applied to a typical porcelain body containing zircon as a marker of the original body-glaze interface. These samples were then heat-treated to three temperatures (1150°C, 1250°C, and 1300°C) for four soak times (1.0, 3.2, 10, and 32 hours) for 24 sample conditions. The body-glaze interaction depth was measured using backscatter and wavelength dispersive spectroscopy. The results demonstrated that boron does, in fact, alter body-glaze interactions, and increases glaze penetration for a given sintering time, however, the time dependence remains the same. While the presence of boron does alter the interactions, there were no appreciable differences between the two levels of boron tested.

Acknowledgements

I would like to first thank Dr. Carty, for being both a mentor and my advisor. I would also like to thank Gerry Wynick for his constant help in analyzing and preparing my samples. I would also like to thank all of my friends from the office, whose conversations were always helpful sorting things out and helped to keep the stress off. Finally, I would to thank Tom Rein and Andy Goldberg, whose work is the basis for my own.

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1 Introduction

It has been hypothesized that adding boron to a typical ceramic glaze would increase the body-glaze interactions, specifically increasing the glaze penetration depth into the body. In other words, for a given sintering time a boron-containing glaze would have a greater penetration depth compared to a similar glaze without boron. If this hypothesis is correct, measuring the body-glaze interaction depth to predict firing time would overestimate the actual time at temperature if a boron-containing glaze was used.

It was previously established that the body-glaze interaction depth follows a log-log relationship with time. It was also determined that the glaze composition does not appear to alter this relationship, either with the overall penetration depth or the time dependence (for glazes that do not contain boron). The introduction of boron, however, is expected to change the body-glaze interaction because boron is known to be a high temperature melt viscosity modifier. That is, for a given temperature, glasses containing boron have lower viscosities than similar compositions without boron. This reduction in viscosity would be expected to increase ionic diffusion rates within the glass, and would thus increase the interaction (or corrosion) of the body by the glaze. To evaluate this, two glazes were prepared with different boron levels. Comparatively, the data collected in this thesis also allowed the comparison of not only the penetration depths, but also the consistency of the glaze-body interactions. Also consistent with previous work, the body-glaze interactions have the potential to alter the glaze texture due to the shift in chemistry of the glaze due to the "introduction" of the body chemistry (due to corrosion of the body). Therefore, understanding the level of the body-glaze interaction for boron containing glazes can then be compared to similar body-glaze interactions for standard glazes (i.e., without boron), allowing the potential shift in texture to be predicted.

2 Experimental Procedure

2.1 Glaze Composition:

Glazes compositions were batched on a molar ratio basis (UMF). Two different boron levels were selected as a way to investigate whether or not the level affected the body-glaze interactions, as well as the presence of boron in general. Table I and Table II outline the glaze compositions tested in this thesis. The glazes were batched and then water was added, starting with 1:2 water to dry material on a mass ratio. The viscosity of the glaze was then adjusted by adding more water, not by adding dispersant, to avoid particle segregation. Finally, the glazes were vibratory milled for 20 minutes to facilitate mixing.

Table I. Outline of Glaze Compositions on a Molar Ratio Basis (UMF).

	B_2O_3 Level	
Constituent	**0.2**	**0.5**
KNaO (R_2O)	0.3	0.3
CaO (RO)	0.7	0.7
Al_2O_3	0.4	0.4
SiO_2	3.0	3.0
B_2O_3	0.2	0.5

Table II. Batch Composition for the two glazes.

Component	Name	0.2 UMF	0.5 UMF	Source	Location
Frit	3124	78.47	31.75	Ferro	Cleveland, OH
Nepheline Syenite	A400	3.96	28.07	Unimin	Nephton, Ontario
Clay	EPK	10.98	9.07	Imerys	Edgar, FL
Whiting	$CaCO_3$	1.81	13.78	Huber Carb	Edison, NJ
Flint	Sil-Co-Sil 63	17.34	4.78	U. S. Silica	Berkeley Springs, WV

The constituent 3124 is a commonly used ceramic flux, and provides the boron for the glaze.

2.2 Body Composition:

The ceramic body used for the samples was a porcelain body (Porcelain for the People, Matt and Dave's Clays, Alfred, NY). Rein's work demonstrated zircon to be an efficient marker of the original glaze/body interface.[3] Therefore, 10% on a dry

weight basis was added to the body and mixed thoroughly with a stand mixer. Table III outlines the body composition on a molar ratio basis.

Table III. Outline of Body Composition on a Molar Ratio Basis.

KNaO (R_2O)	0.86
CaO (RO)	0.14
Al_2O_3	3.72
SiO_2	20.62

2.3 Sample Preparation:

The body suspension was first slip casted into 6 cm diameter disks using gypsum molds. These disks were then bisque fired to 1000°C. The glazes were then applied by dipping. To minimize the number of samples to be prepared, as well as allow more samples to each polishing mount, both glazes were applied to a single disk as shown in Figure 1.

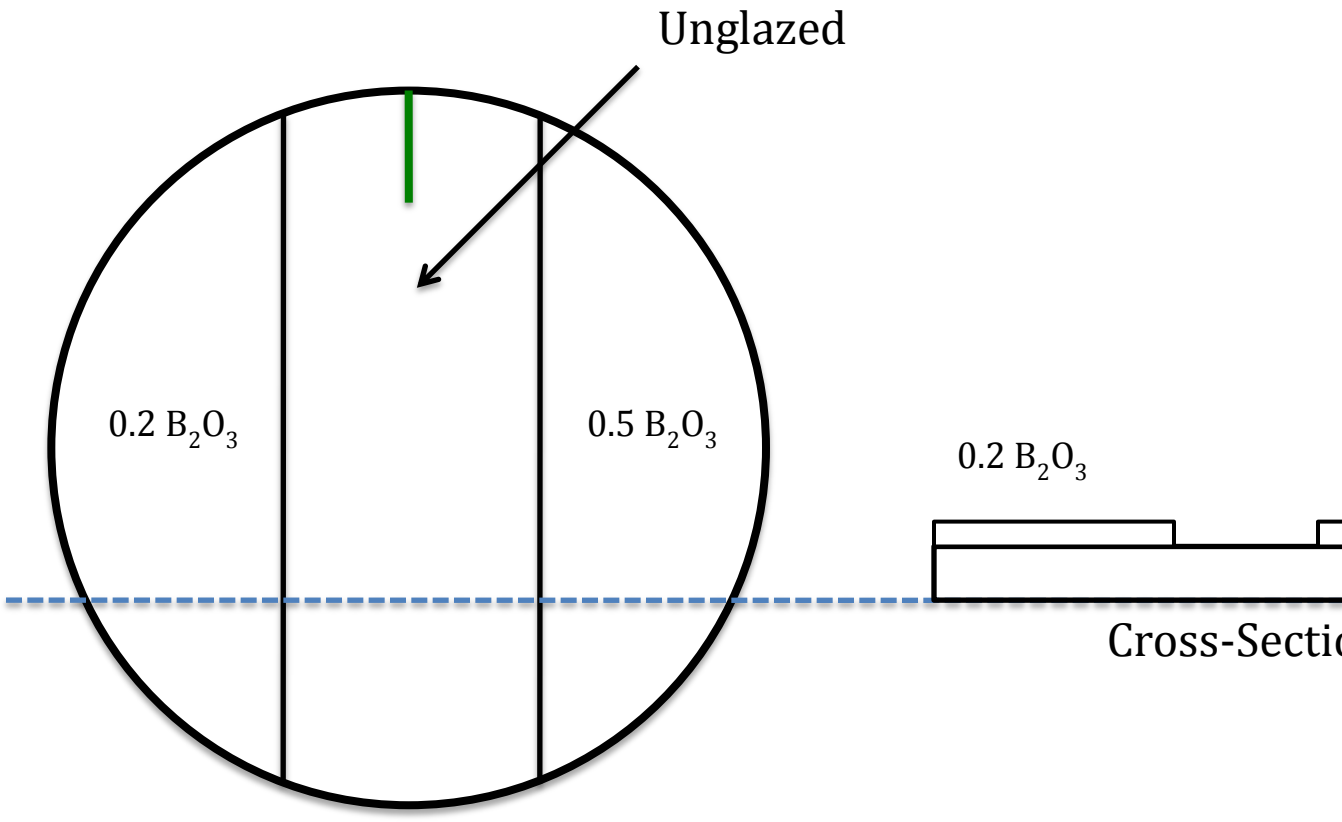

Figure 1. Illustration of Sample Preparation.

After samples were prepared, they were fired as outlined in Table IV, for a total of twenty-four samples. All samples were sintered at a heating rate of 5°C/min.

Table IV. Outline of Firing Schedule.

	1.0 Hours	3.2 Hours	10 Hours	32 Hours
1150°C	X	X	X	X
1250°C	X	X	X	X
1300°C	X	X	X	X

Once the samples were fired, they were sectioned perpendicular to the glaze-body interface using a diamond saw, as illustrated in the above figure. These cut-offs were mounted in epoxy, and then polished down to 1μm using diamond paste. The polished samples were then analyzed using Wavelength Dispersive Spectroscopy (WDS) as well as backscatter electron (BSE) images.

2.4 Measurement of Body-Glaze Interactions:

Measurement of the glaze penetration depth was done using two different techniques, BSE and WDS imaging. A computer program ImageJ (Ver. 1.46, National Institute of Health, Bethesda, MD) was used to measure the digital images. ImageJ allows the setting of a certain number of pixels to a known unit of measurement, and then uses that scale to provide a measurement. Using the size bar returned with all of the BSE and WDS images, an accurate scale could be created.

For the BSE images, minor to no image modification was necessary, as the zircon was added to create a distinct marker of the original body-glaze interface. Occasionally the contrast of the image would be adjusted to aid in measuring. Measurements were taken from the zircon layer to the first presence of porosity or un-dissolved quartz grains. Figure 2 illustrates the process of measuring glaze penetration from a BSE image.

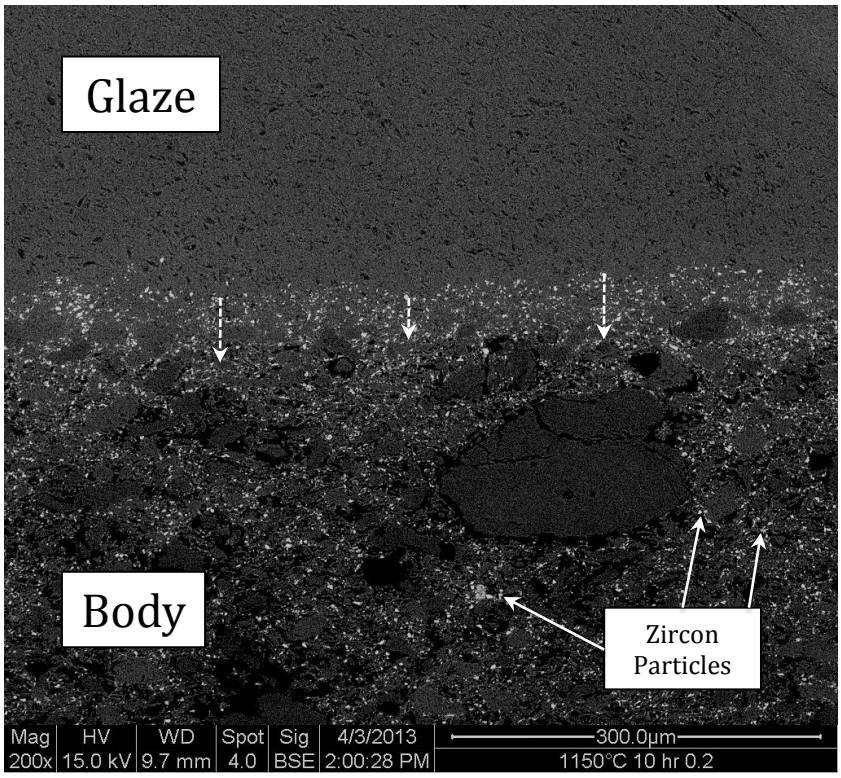

Figure 2. Outline of Measuring BSE Images. Arrows Represent Sample Measurements.

To measure the WDS images, some image modification was necessary. Individual calcium and zirconium scans were returned after analysis, and to properly measure the body-glaze interactions, the images were superimposed using Photoshop. Figure 3 is an example of the results returned from a typical WDS scan (0.2 B_2O_3 fired at 1250°C for 32 hours).

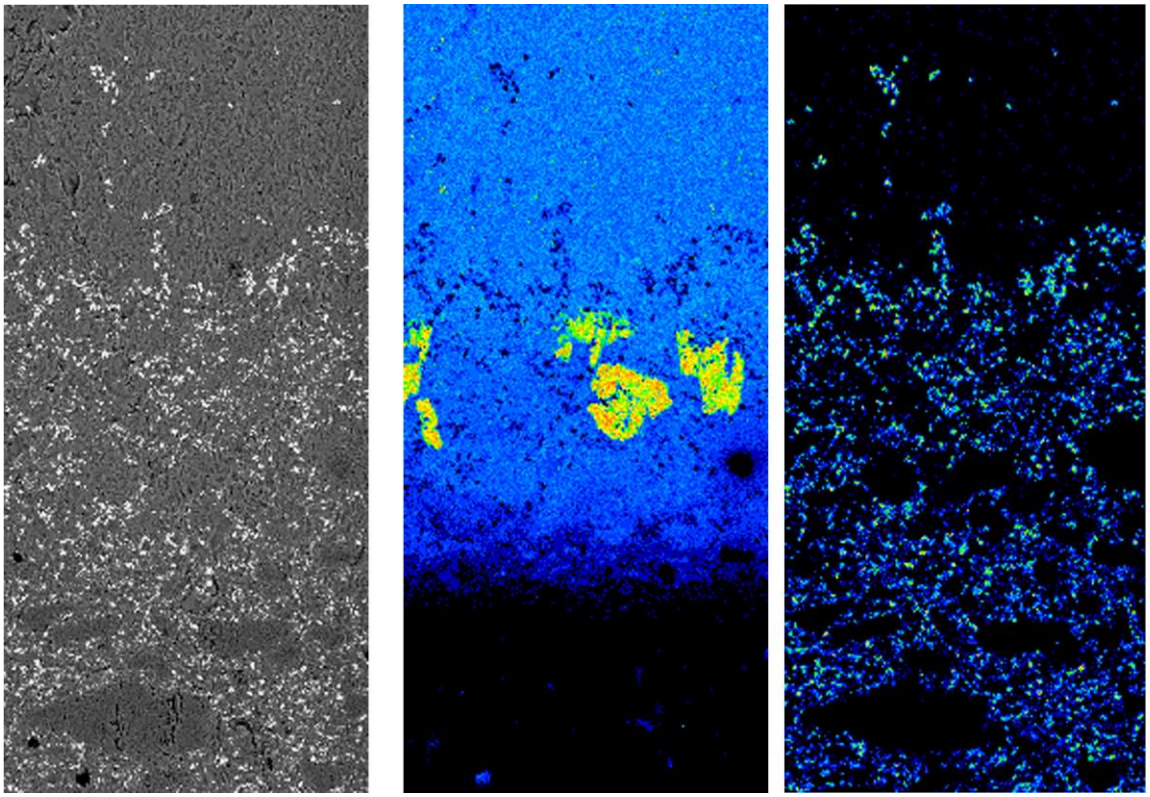

Figure 3. Original Scans Returned from a Typical WDS Scan.

After these individual scans were uploaded into Photoshop (Ver. CS6 Extended, Adobe Systems, Mountain View, CA) the zirconium scan was isolated. The opacity of the zirconium scan was then reduced, so both the zirconium and calcium scans could be seen together. If necessary the hue/saturation of the zirconium scan was adjusted to improve differentiation. Aligning the size bar associated with the respective scans enabled placing one scan directly on top of the other, as the WDS scans exactly the same field for all three scans. Figure 4 shows a finished composite image, and outlines the measuring of the glaze penetration depth.

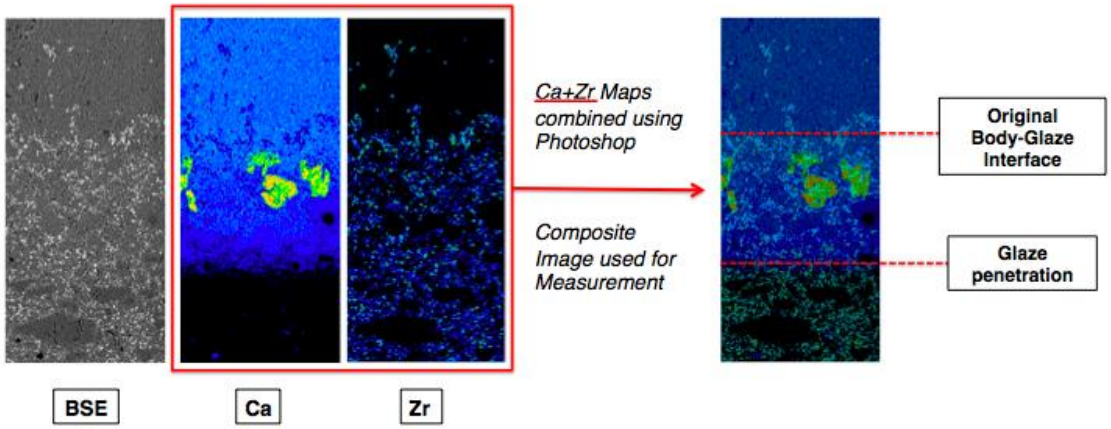

Figure 4. Creating and Measuring a Composite Image. Composite Image for 32 hours at 1250°C with 0.2 boron (UMF).

3 Results

Figures 5 and 6 contain the composite images for both the 0.2 B_2O_3 level and 0.5 B_2O_3 level, respectively. In viewing these images, notice the general trend of increasing glaze penetration depth with increasing sintering time, as outlined by Rein.[3] Another visible trend is an increase of penetration with an increase in sintering temperature.

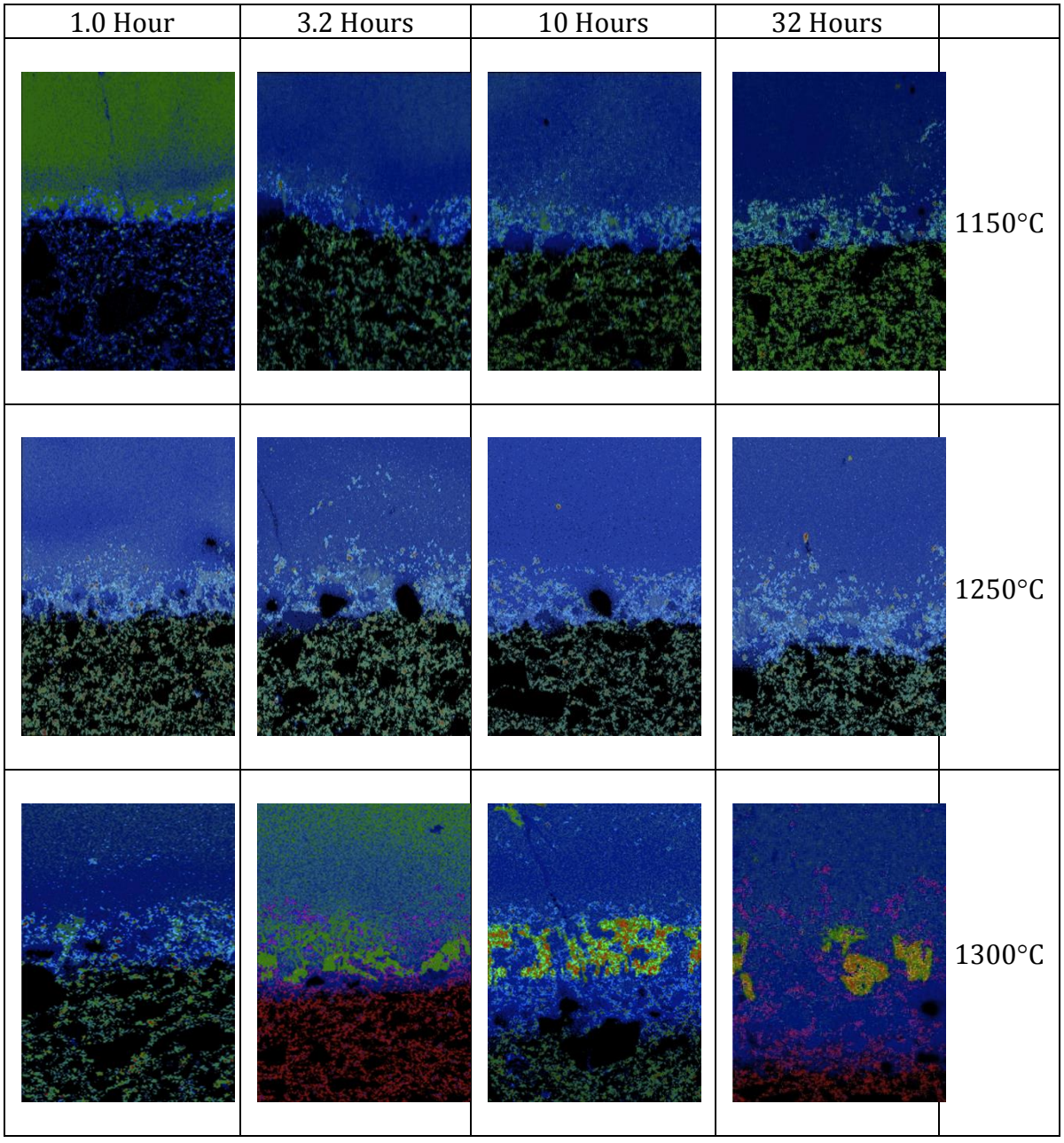

Figure 5. WDS Composite Images for all 0.2 Data Points.

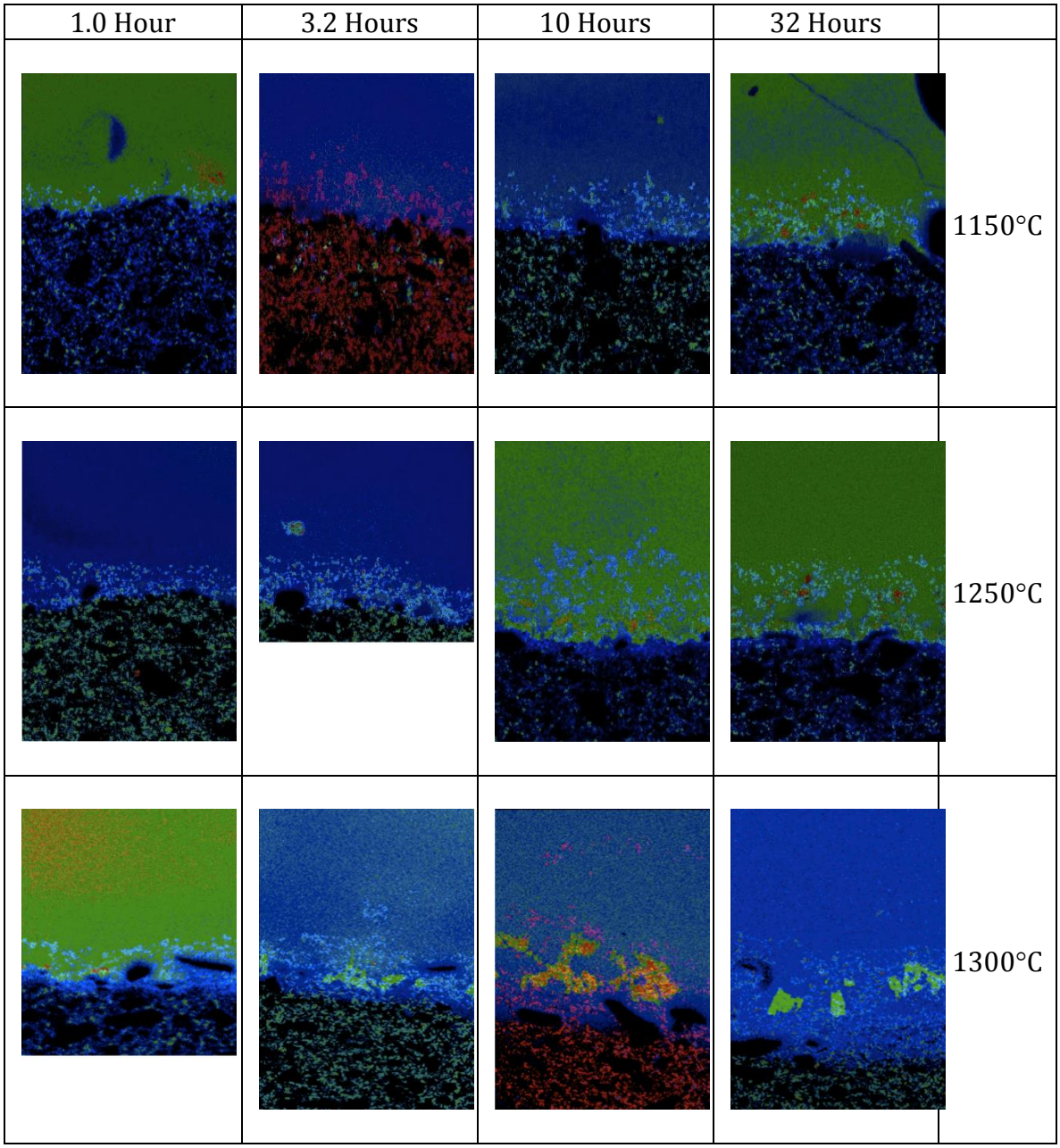

Figure 6. WDS Composite Images for all 0.5 Data Points.

Tables V and VI compile the measurements taken for both the WDS and BSE images. These measurements are represented by a single average, with an associated standard deviation. Figures 7 and 8 plot the measured penetration depth on a log-log scale with sintering time for different levels of B_2O_3 for the WDS images.

Table V. Glaze Penetration Measurements from WDS Images (μm±St. Dev.).

	0.2 B_2O_3			
1150°C	1.0 Hour	3.2 Hours	10 Hours	32 Hours
Average	20.70±3.82	25.86±5.40	40.44±6.82	44.55±8.90
1250°C				
Average	36.42±4.74	47.94±9.64	59.66±8.16	80.64±16.49
1300°C				
Average	45.96±5.92	95.79±11.58	142.5±17.44	210.0±14.32
	0.5 B_2O_3			
1150°C	1.0 Hour	3.2 Hours	10 Hours	32 Hours
Average	16.82±4.45	29.92±6.34	39.73±7.12	45.91±8.99
1250°C				
Average	35.34±7.37	42.56±9.80	77.90±14.98	71.62±6.89
1300°C				
Average	59.07±7.17	74.24±12.49	133.2±22.47	157.5±5.53

Table VI. Glaze Penetration Measurements from BSE Images (μm±St. Dev.).

	0.2 B_2O_3			
	1.0 Hour	3.2 Hours	10 Hours	32 Hours
1150°C	31.47±5.66	39.05±8.28	52.90±13.12	88.37±16.13
1250°C	51.89±10.00	49.90±11.43	86.12±16.89	125.3±21.01
1300°C	136.7±34.37	149.6±32.60	200.5±48.68	233.2±36.33
	0.5 B_2O_3			
	1.0 Hour	3.2 Hours	10 Hours	32 Hours
1150°C	31.50±5.67	43.42±14.90	59.94±9.28	124.5±34.87
1250°C	44.54±15.32	67.89±12.11	-	132.0±25.15
1300°C	83.85±25.31	124.2±31.38	-	256.5±52.26

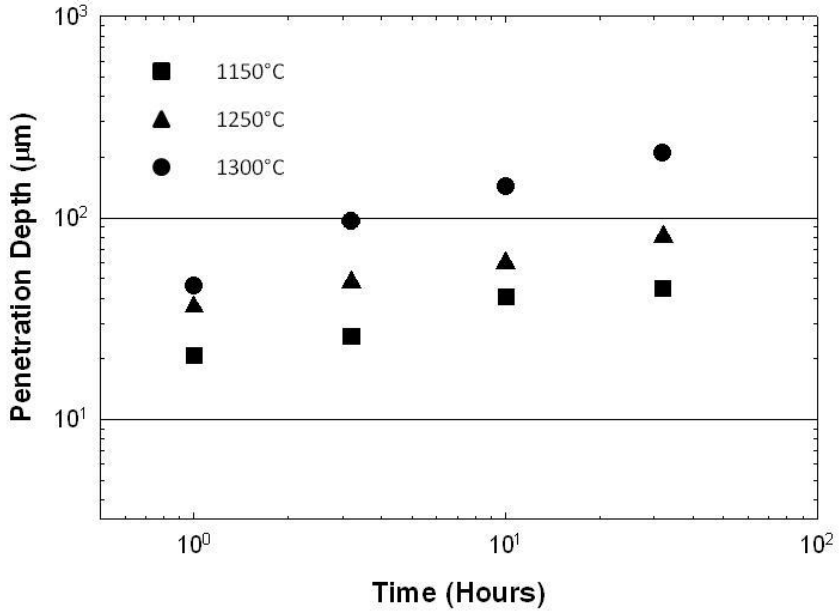

Figure 7. Measurements Taken from WDS Scans for 0.2 B_2O_3 (UMF).

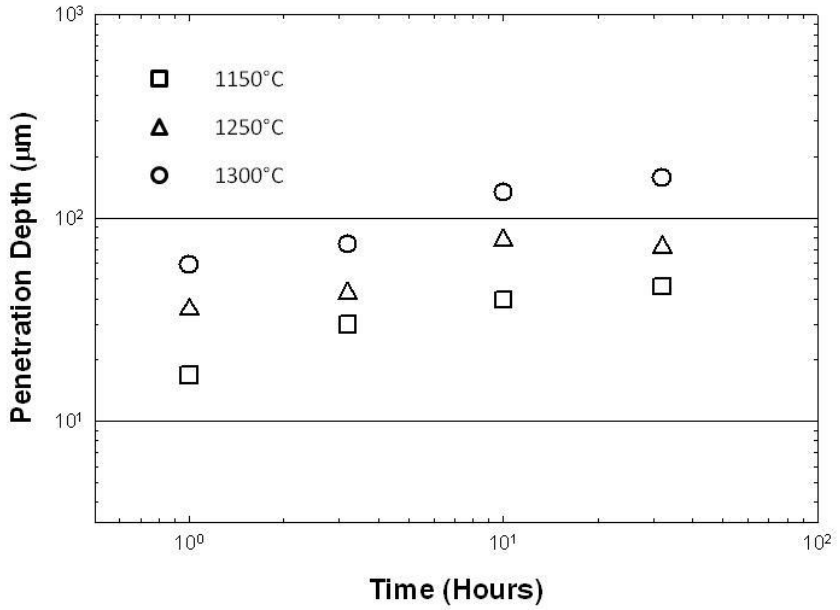

Figure 8. Measurements Taken from WDS Scans for 0.5 B_2O_3 (UMF).

The BSE measurements are not included in the final results. This is because there was too much error associated with taking the measurements. The WDS images are considered to be very accurate because of the ease of differentiation between the calcium and zircon levels. Figure 9 illustrates that the BSE image measurements

were repeatedly greater than the WDS measurements because of the judgment that had to be made using porosity and un-dissolved quartz grains.

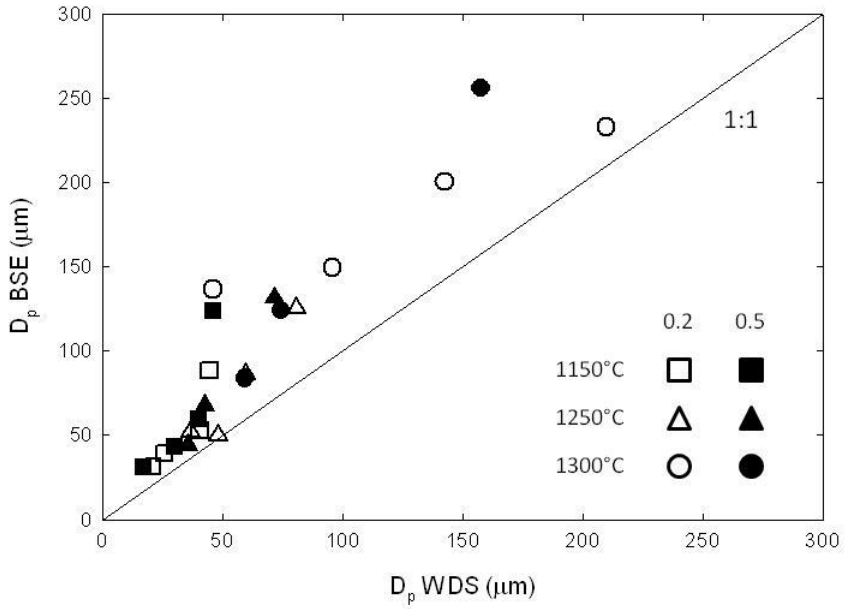

Figure 9. Illustration of BSE Measurement Error.

Figure 10 plots both B_2O_3 levels on the same graph, along with regression lines that represent all eight points for a temperature. The components of these regression lines can be found in Table VII.

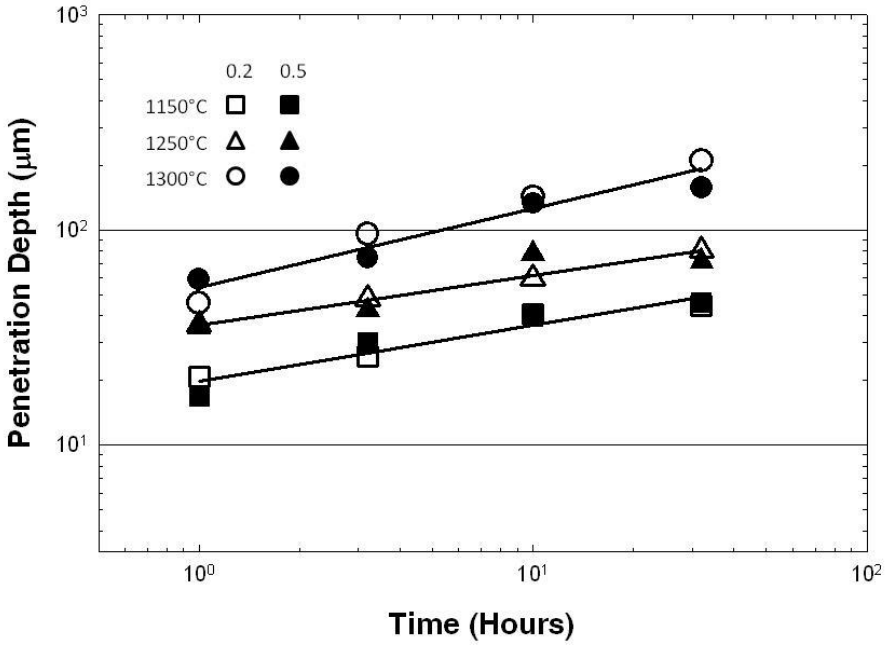

Figure 10. All Measurements Taken from WDS Images with Regression Lines.

Table VII. Components of Regression Lines for WDS Images.

Temperature	Slope	Intercept	RSQ
1300°C	0.37	54.18	0.93
1250°C	0.23	36.09	0.90
1150°C	0.26	19.79	0.92

4 Discussion

4.1 Zircon Migration – An Argument for Reduced Viscosity

For both types of images, the measurements had to be taken from the general zircon layer to the end of the glaze penetration. The measurement cannot always be taken from the first seen zircon because of mechanical migration. This would cause a measurement that was greater than the actual penetration depth. Figure 11 illustrates the effect of zircon migration.

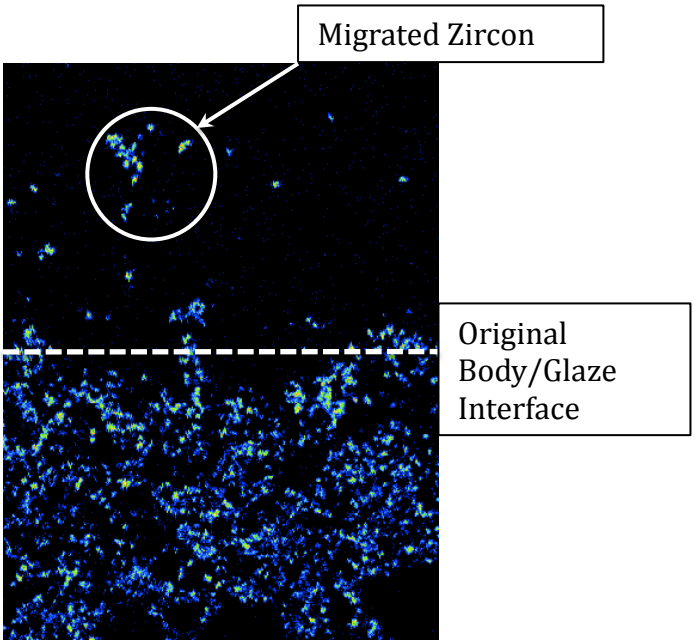

Figure 11. WDS Scan Showing Zircon Migration as a Problem for Measurements.

This migration is caused by zircon traveling with bubbles formed within the glaze during sintering. The prevalence of this migration confirms that there is a reduction in viscosity of the glaze, which can only be attributed to the presence of boron. As described earlier, boron is a high temperature viscosity modifier. Therefore, it would be expected that at higher temperatures, the migration would be more pronounced. Figure 12 shows that this is the case, with the compiled scan on the left being 10 hours at 1150°C, and the scan on the right being 10 hours at 1300°C.

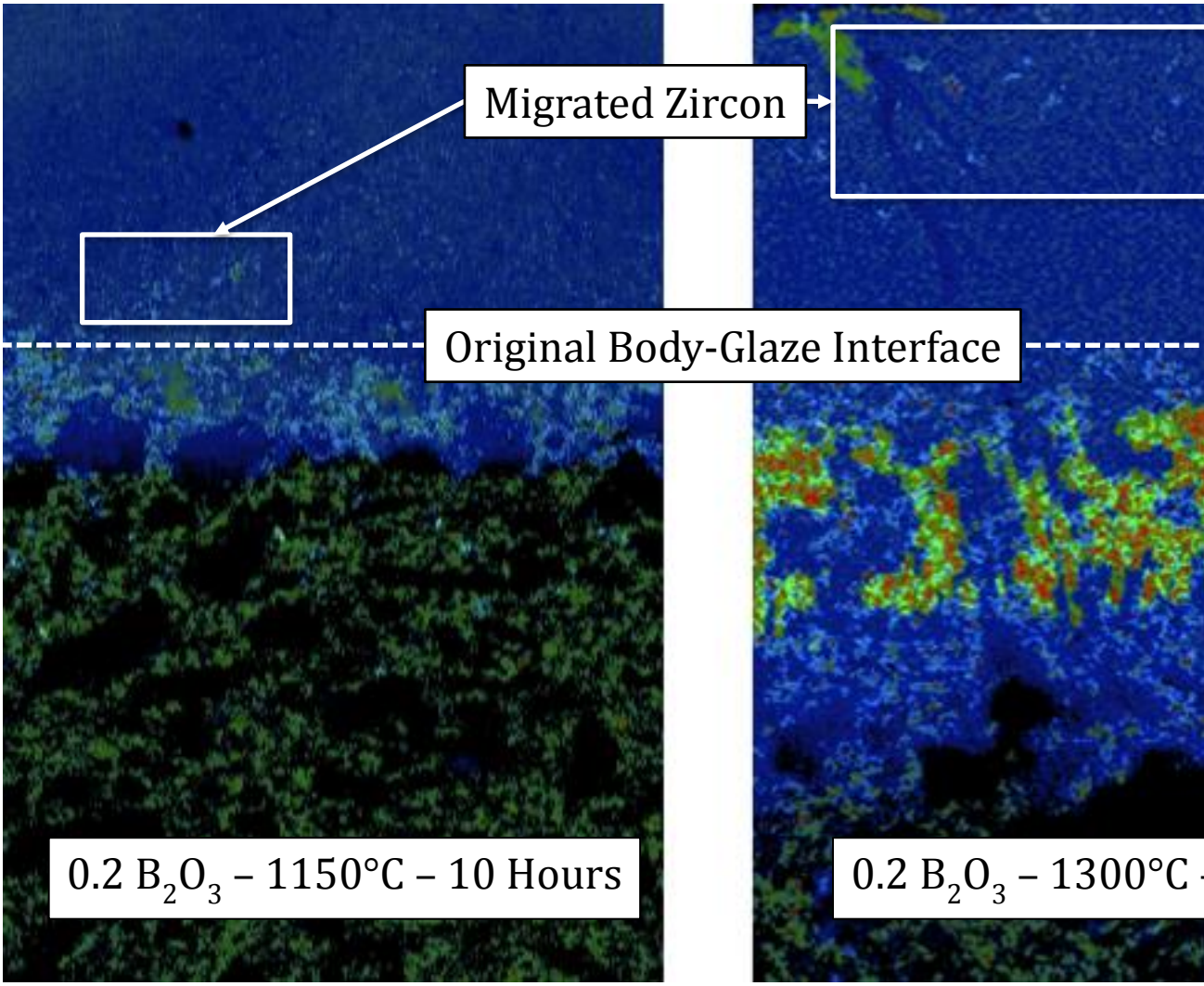

Figure 12. Illustration of Increasing Zircon Migration with Increasing Temperature.

4.2 Comparison of BSE to WDS Measurements

Originally, based off of the work done by Goldberg, measurements were taken using BSE images. The zircon provided a marker that was easily identifiable within the body. However, distinguishing the body from the glaze was difficult. This is because of the fact that the body and glaze have very similar average molecular weights. Goldstein et al. stated that the backscattering coefficient of a material increases as atomic number increased.[5] This trend with atomic number also correlates with increasing molecular weight, that backscattering coefficient also

increases with increasing molecular weight. The molecular weight of the porcelain body is 66.8 g/mol, whereas the molecular weights of the glazes are 64.7 g/mol and 65.0 g/mol for the 0.2 B_2O_3 and 0.5 B_2O_3 levels respectively. Therefore, as the body and glazes molecular weights are very similar (within 3% of each other) they are almost impossible to differentiate. This is also why the Zircon shows up so brightly on the BSE images, as the average atomic mass of Zircon is 190.31 g/mol, which is much higher than the atomic mass of both the body and glaze.

Therefore, measuring the BSE images required estimating where the glaze had stopped penetrating the body by looking at markers such as un-dissolved quartz grains or porosity. Figure 13 shows the lack of definition inherent with the BSE images.

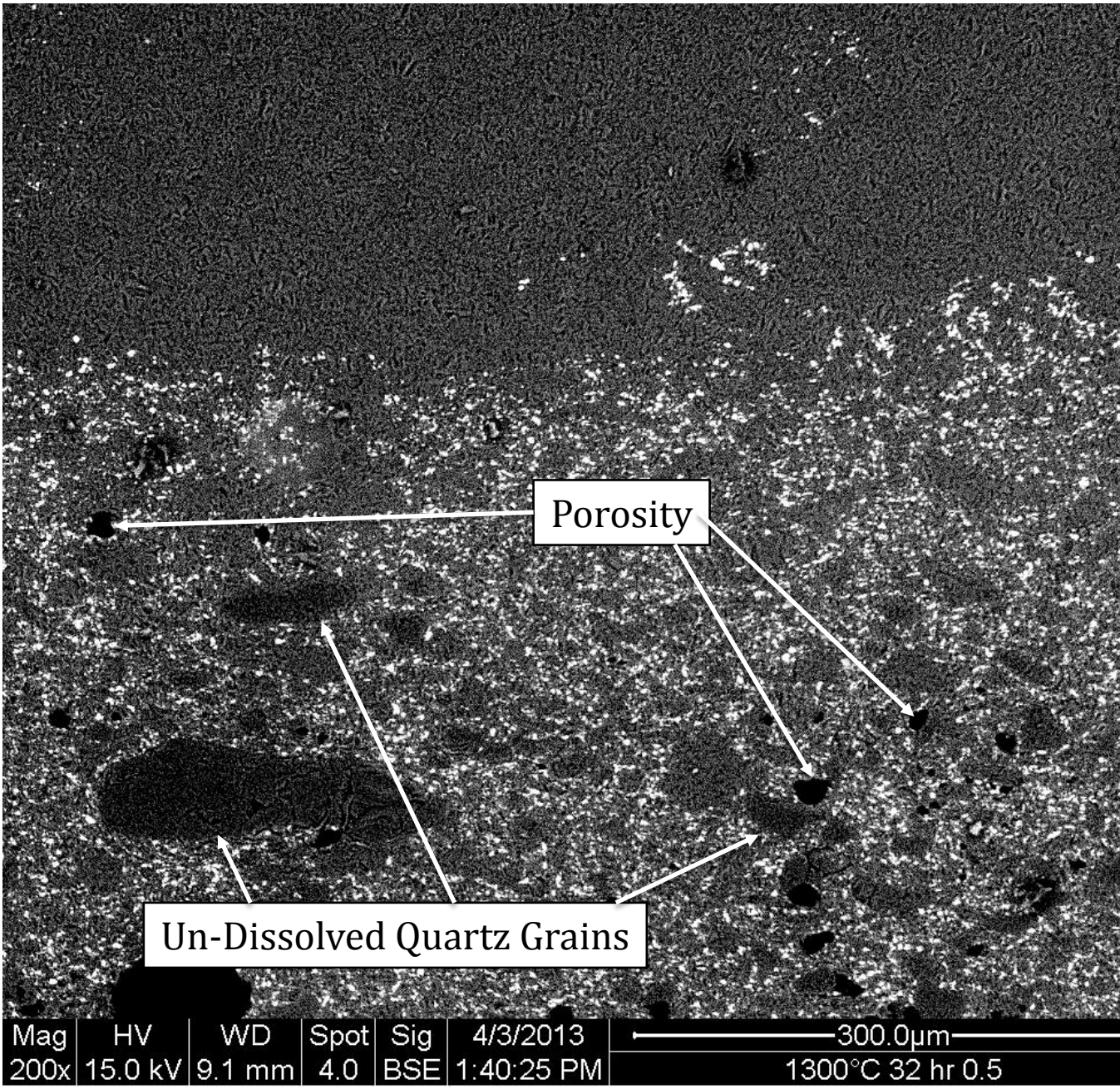

Figure 13. 1300°C for 32 hours, 0.5 B_2O_3 BSE image Illustrating Lack of Definition.

As seen in Figure 13, it is extremely difficult to estimate what point the glaze penetrates to. As can be seen in the figure, the porosity is not evenly distributed, and ranges from what would be only a few tens of microns from the zircon layer, to almost the full length of the size bar from the zircon layer. This uncertainty lead to very large deviations in the data, especially at higher temperatures and dwell times, as recorded in the results section of this thesis. Therefore, the measurements taken

from the WDS images were used in making conclusions. While the BSE images were not necessarily incorrect, the WDS images provided an unarguable penetration depth with small amounts of deviation, and were found to be worth the extra time needed to take the measurements.

4.3 Affect of Boron Level within Glaze

As outlined in the experimental procedure, two different glaze compositions were tested in this work to determine whether the level of boron within the glaze would change the way the boron affected the glaze. It was originally thought that adding more boron to the glaze would increase the affect that boron had on the glaze, i.e. if boron increased glaze penetration, adding more boron would further increase the glaze penetration depth. However, it was seen that there was no significant difference in the body-glaze interactions between the two boron levels tested. Figure 14 illustrates this point.

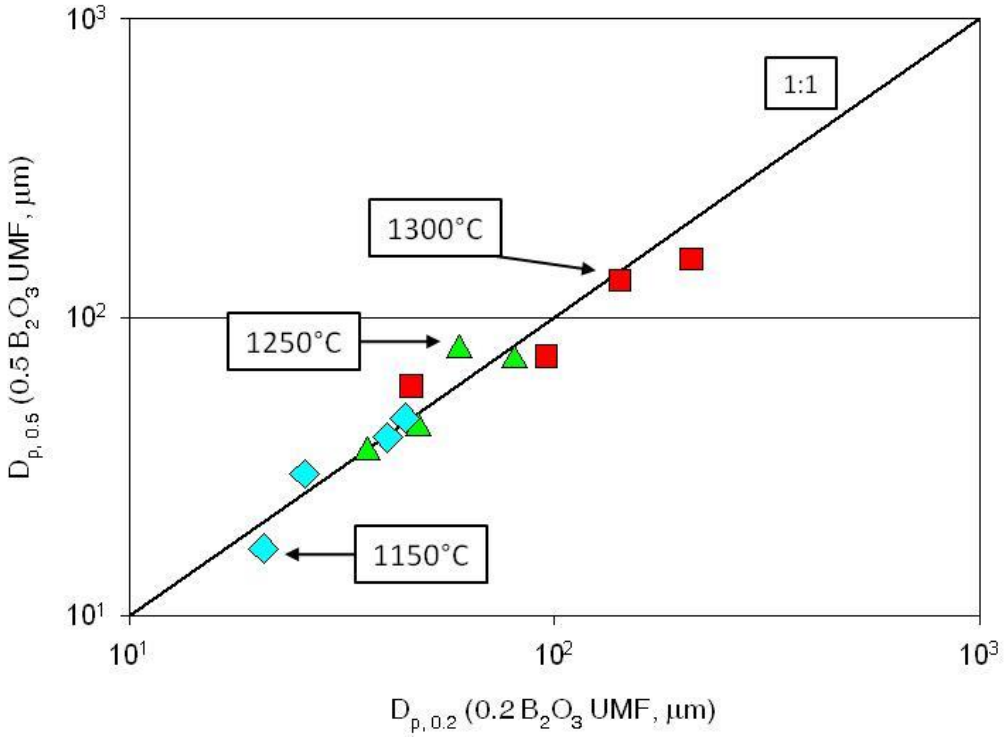

Figure 14. Comparison of 0.2 B_2O_3 level and 0.5 B_2O_3 Level Data Points.

The 0.2 B_2O_3 level is plotted on the X-axis, while the 0.5 B_2O_3 level is plotted on the Y-axis. If there was a significant difference between the two levels, the points would tend to shift to one side of the 1:1 line, however, the points are scattered around the 1:1. This scatter indicates no clear trend about the difference in levels and therefore the conclusion can be made that there is no significant difference.

4.4 Boron's Affect on Body-Glaze Interactions

To demonstrate whether boron had an effect on the body-glaze interactions the data found in this thesis was compared to data collected previously, which contained no boron. Rein's data for a twice-fired system was plotted in conjunction with the 1300°C data from this study and is shown in Figure 15.[3] As there was found to be no difference between the two boron levels, all eight points were used to compare to the previous work. Regression lines for both the previous and current work help to illustrate the general trend of the data.

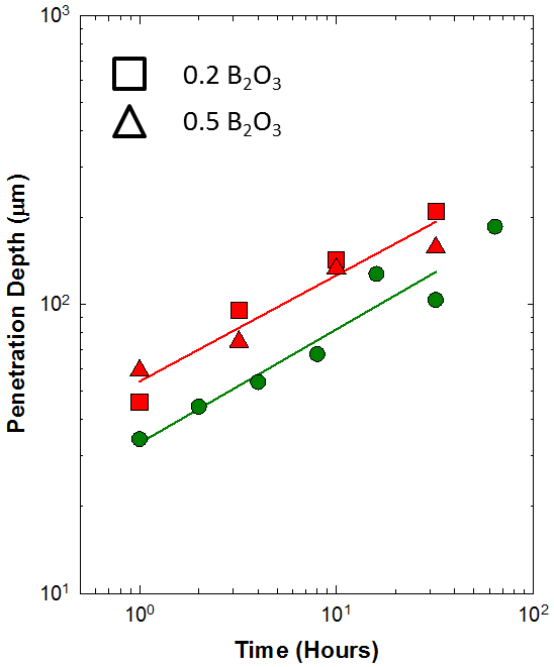

Figure 15. Comparison of 1300°C Data to Previous Data.[3]

The regression lines describing the data sets are almost parallel, indicating that the two glazes interacted similarly. Also, illustrated by the vertical shift of the

regression line, it is apparent that the presence of boron does affect the body glaze interactions, proving the hypothesis of this thesis correct. The components of the regression lines can be found in Table VIII.

Table VIII. Regression Line Components for 1300°C Data and Previous Data.[3]

Data Set	**Slope**	**Intercept**	**RSQ**
B_2O_3	0.36	54.18	0.93
No B_2O_3	0.39	33.13	0.94

The equations for the regression lines help to determine the effect that the boron had on the glaze penetration. By taking a ratio of the intercept values, it can be determined that the boron increased the penetration depth of the glaze by about 1.6 times for a given sintering time.

5 Conclusions

Based on the comparison with previous data, boron effectively increases the interactions between a ceramic body and the applied glaze. Specifically, it increases the penetration depth for a given sintering time. Therefore, the hypothesis of this thesis is correct.

Boron effectively shifted the viscosity of the glaze, as demonstrated by the amount of zircon migration within the body. The migration increased with both increasing temperature and increasing sintering time.

Differences in boron levels were found to be insignificant. While it is clear that the presence of boron does affect glaze-body interactions, the levels tested in this work behave similarly with no appreciable differences.

It was also determined that BSE images did not provide enough definition to accurately and reliably measure the glaze penetration depth. WDS images provide a clear representation of both the zircon and calcium scans, and are more accurate and reliable for taking measurements.

6 References

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