March 26, 2010

To: File

From: Michael J. Minot

RE: Evaluation of GO18-223 Frit Applied to B33 Glass (Borofloat) – UPDATE

This document has been updated to include results based on building small assemblies consisting to a top plate, bottom plate and side walls, using B33 glass and G018-223 Frit. In addition, a sandwich assembly was made, consisting of a silver coated B33 slide, with a top plate frit bonded to the silver. The sandwich remained intact following the frit bonding. Refer to Page 24 for these additional updates.

March 1, 2010

To: File

From: Michael J. Minot

RE: Initial Evaluation of GO18-223 Frit Applied to B33 Glass (Borofloat)

These trials were initiated around February 3d, and still remain a work-in-progress.

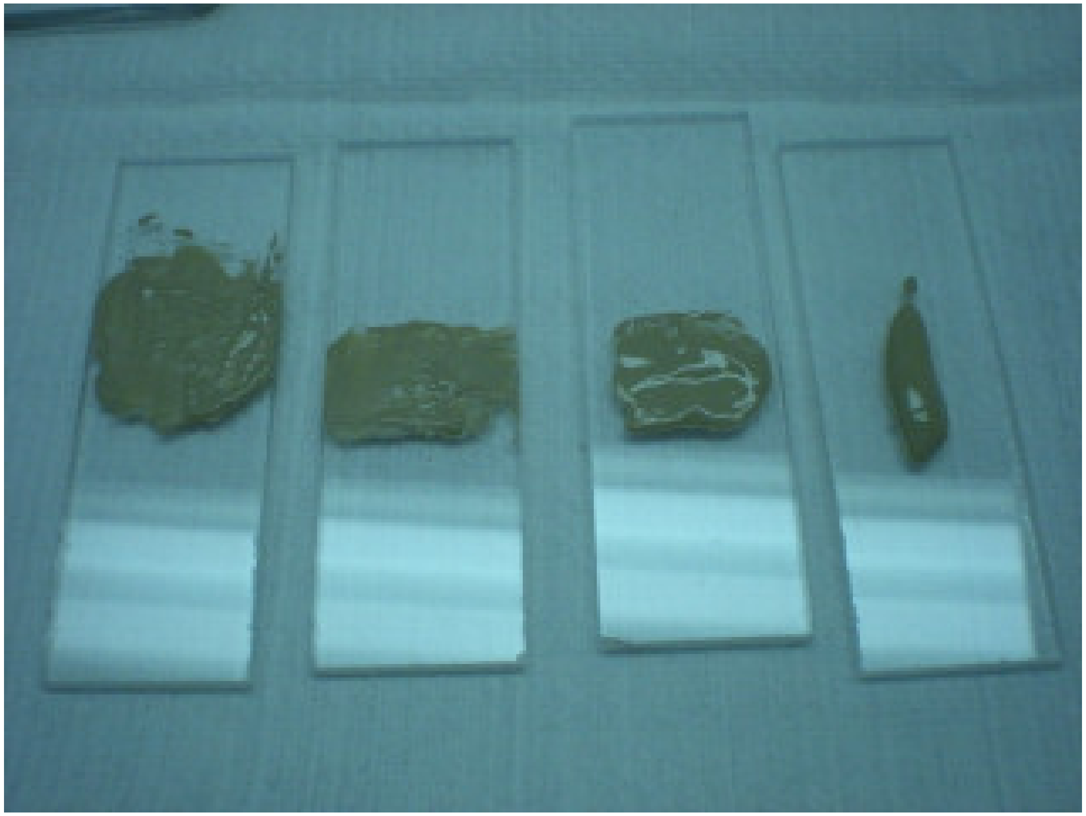
MJM

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 1 (832) – Frit, as received from Schott, was pre-mixed with binder; however it had settled, and was lumpy when stirred with a Teflon spatula.

Figure 2 (833) – After 10 minutes of vigorous mixing, the frit & binder were smooth and free flowing.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

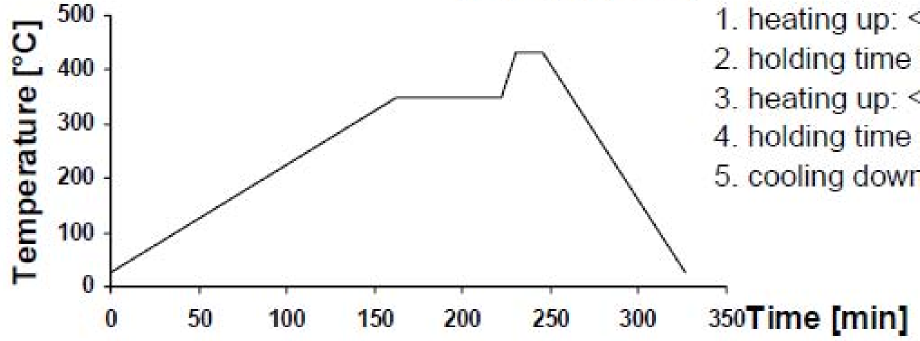
Figure 3 (834) – Frit was applied using Teflon spatula to four 1" X 3" pieces of Borofloat (B33) glass. Samples #1 and #2 on the left were thinner, compared to #3 and #4 on the right, which were thicker.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Glass No.: G018-223

Burn out binder and Pre-Glazing

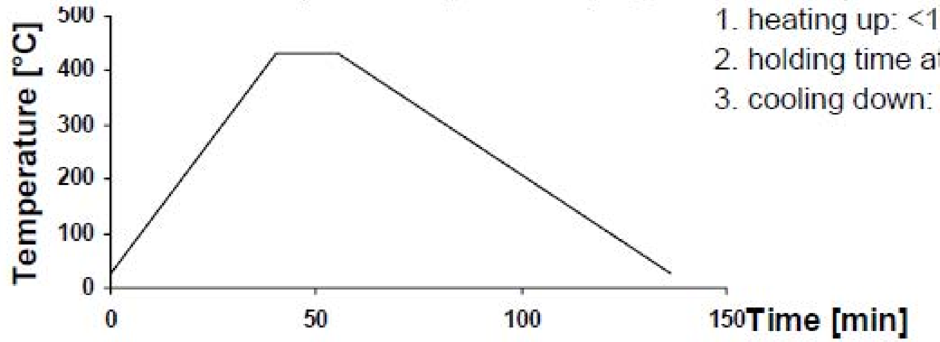
(In case of paste)



1. heating up: <2 K/min to 350°C
2. holding time at 350°C ; 60 min
3. heating up: <10 K/min to 430°C
4. holding time at 430°C ; 15 min
5. cooling down: <5 K/min to 25°C

Vitreous seals and glazings

(In case of powder or preglazed material)



1. heating up: <10 K/min to 430°C
2. holding time at 430°C ; 15 min
3. cooling down: <5 K/min to 25°C

Figure 4 - Showing recommended thermal cycle for binder, pre-glazing and vitreous sealing.

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Table 1 – Showing actual thermal cycle used (furnace was manual, non-programable)

Time	Process Step	Target Temp C	Furnace Set Point F	Elapsed H:Min	Elapsed Minutes	Temp. F at Sample	Temp C	
2:20 PM	1) Ramp UP	350	RT	0:00	0	68	20.0	
2:55 PM	Binder Bake-out	350	250	0:35	35	241	116.1	
3:25 PM	Binder Bake-out	350	400	1:05	65	384	195.6	
3:45 PM	Binder Bake-out	350	600	1:25	85	494	256.7	
4:05 PM	Binder Bake-out	350	600	1:45	105	576	302.2	
5:00 PM	2) Binder Bake-out	350	663	2:40	160	650	343.3	
Note #1: Furnace shut-off overnight, photographed upon cooling, showing a dull non-glazed surface								
8:30 AM	Ramp-up	430	662	0	160	84	28.9	
9:15 AM	Ramp-up	430	662	0:45	205	623	328.3	
9:40 AM	Ramp-up	430	806	1:10	275	778	414.4	
10:00 AM	3) Pre-Glazing	430	836	1:30	365	818	436.7	
Note:	Samples removed from the furnace, photographed, showing a reflective, glazed, glassy surface							
Note:	First two sandwigh samples prepared and re-inserted into the hot furnace							
10:20 AM	Ramp-up	430	836	0	365	775	412.8	
10:40 AM	4) Vitreous Seal	430	836	0:20	385	854	456.7	
Note:	Furance shut-off and samples allowed to cool (some what)							
Note:	Samples removed & second set of sandwich samples prepared and inserted into the furnace							
11:10 AM	Ramp-up	430	836	0	385	716	380.0	I
11:30 AM	5) Vitreous Seal	430	887	0:20	405	887	475.0	
Note:	Samples removed and furnace shut off							

Figure 5 (837) – All four samples were placed on a metal support (lined with aluminum foil) and inserted into the middle of a small (4" X 4") laboratory furnace. The furnace control thermocouple is seen at the top middle (red arrow). Two monitoring thermocouples were inserted along the bottom left and right sidewalls (black arrows) and positioned so that the TC was touching the bottom of the metal plate, in the middle (white arrows).	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 6 (839) – Samples were removed from the furnace following 'binder burn-out' and photographed (Note #1). The samples showed no luster or sheen; they were powder rather than glassy (as expected at this stage of the cycle).

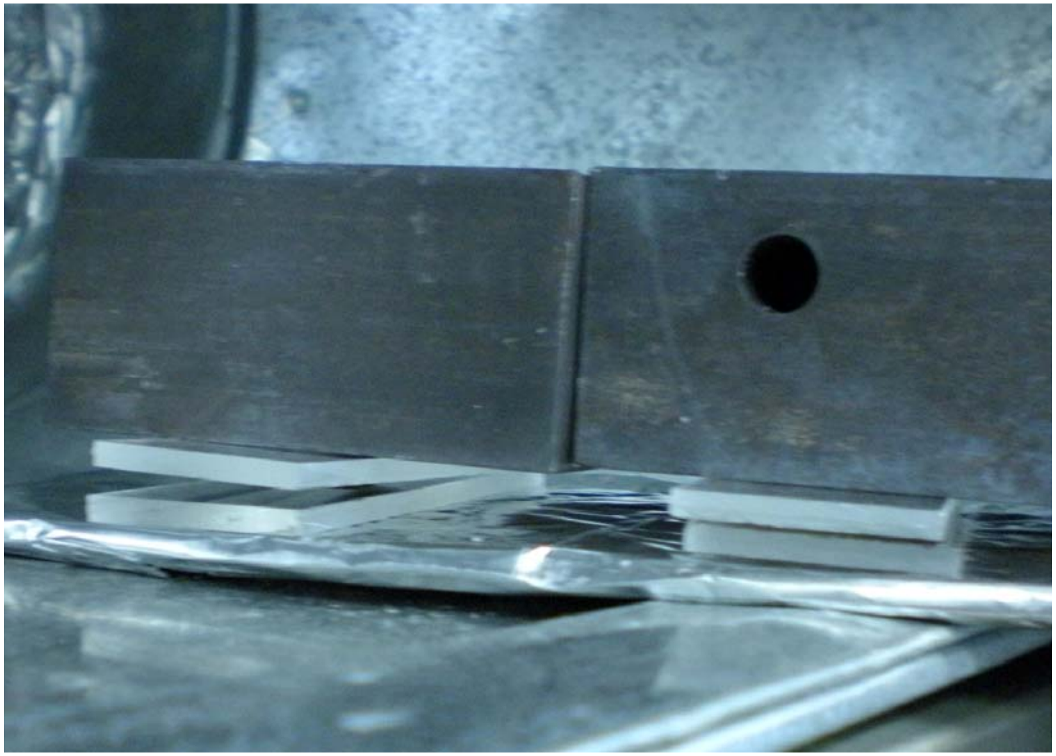
Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com

JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 7 (840) – Close-up of samples (from Figure 6) following 'binder burn-out' (Note #1). The samples showed no luster or sheen; they were powder rather than glassy (as expected at this stage of the cycle)	

Figure 8 (845cropped) – Samples following 'pre-glazing' (Process Step 3). These samples have a glassy luster or sheen, as expected after heating at 437C.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 9 (849cropped) – top slide of Borofloat was stacked on top of the bottom piece with the glazed frit between forming a 'sandwich' construction (B33 – GO18233-B33) with metal weights applied on top. The top plate did not sit evenly onto the glazed frit as seen in the sample (#4) on the left. The two steel weights were 808 gms (1.78 lbs) and 862 gms (1.90 lbs).	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 10 (850 cropped) – Sandwich structures #3 and #4 following vitreous sealing. The frit spread out forming a uniform layer, despite the uneven structure shown in figure 9.

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 11 – showing the reverse sides of samples #3 and #4	

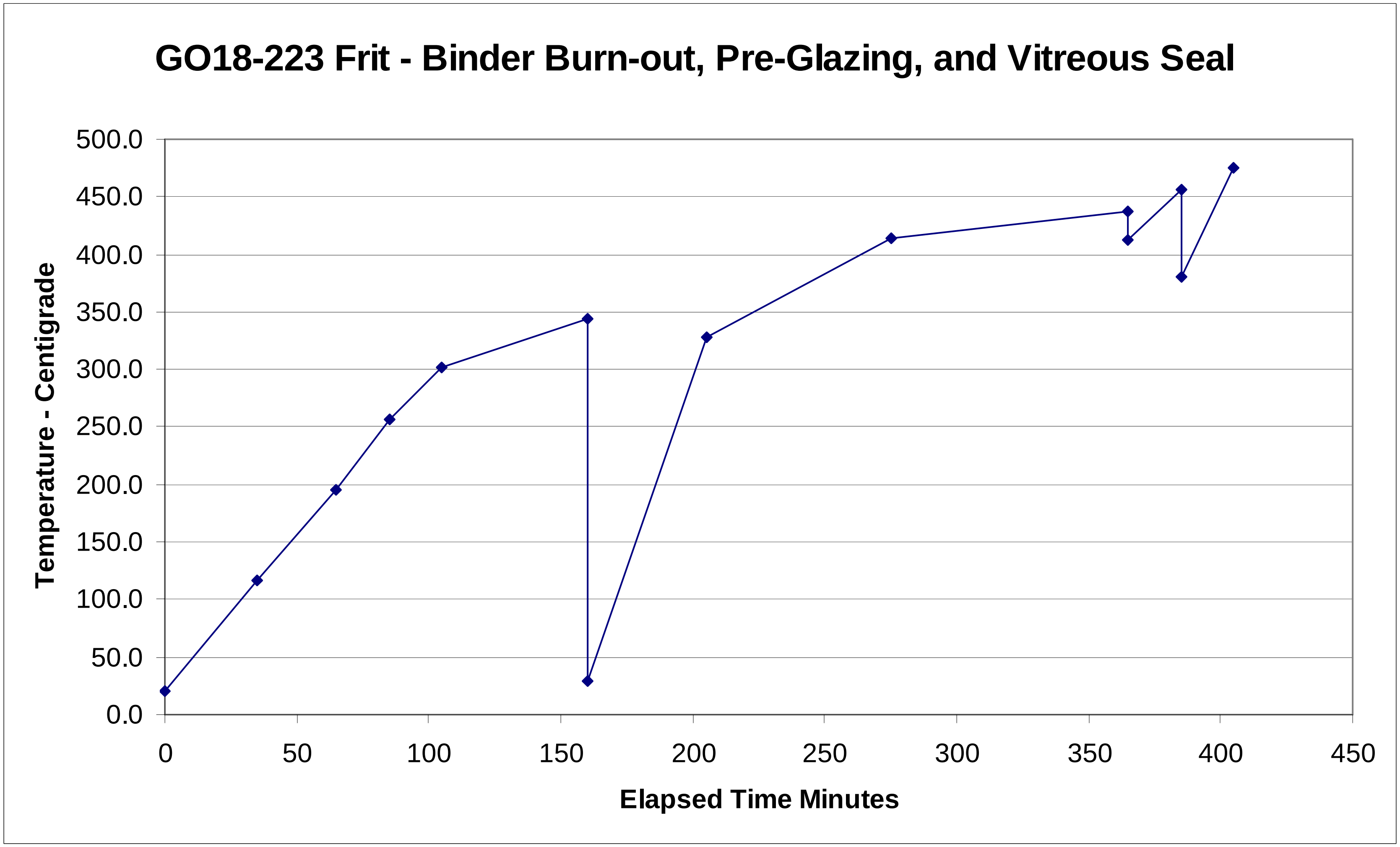


Figure 1 – Showing actual thermal cycle used: Binder burn-out (RT to 350C), Furnace shut-off over night, Ramp to pre-glazing (436C), Samples removed to form first sandwich pair, Vitreous seal (456C), Samples removed and second sandwich pair inserted, Vitreous seal (475C) .

Figure 12 - B33 glass / GO18-223 Frit / B33 Sandwich structure, top down lighting, 50 X magnification, polarized light. The frit is smooth; no voids or bubbles are observed, there is little or no microstructure.

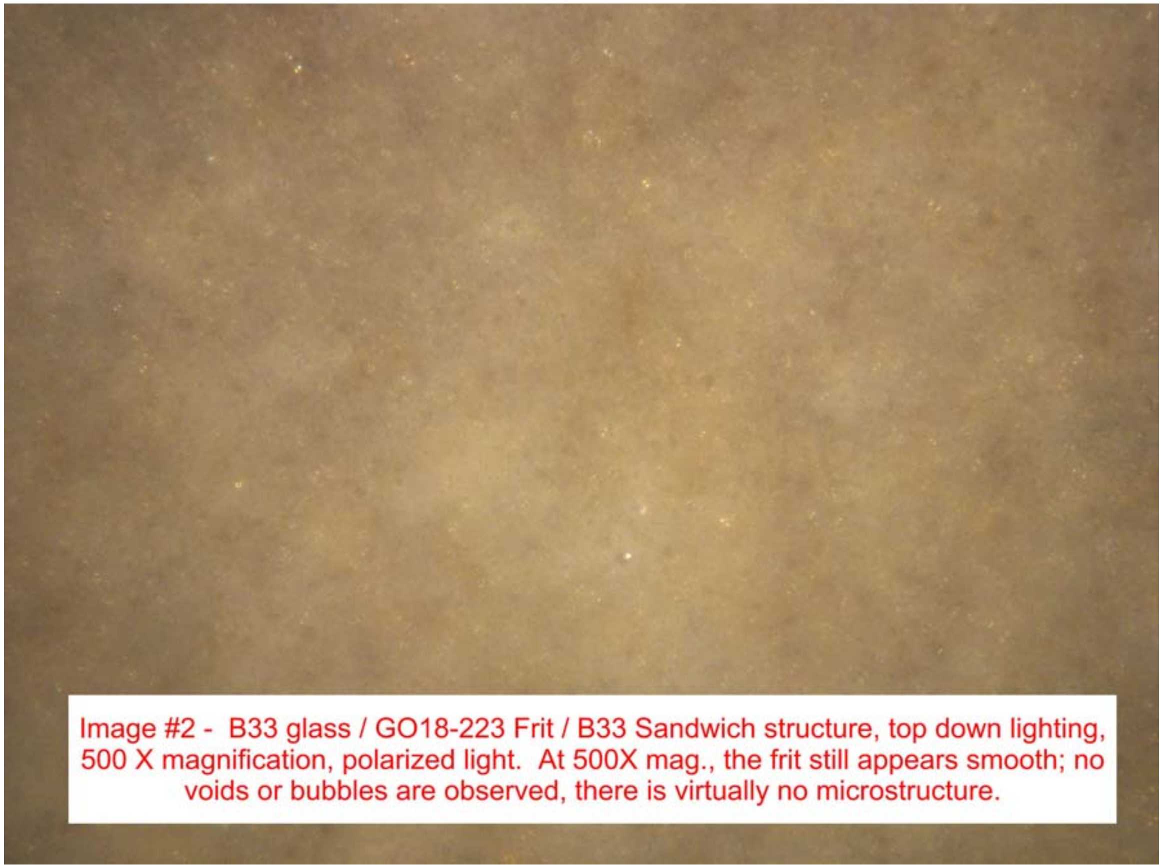
Figure 13 - B33 glass / GO18-223 Frit / B33 Sandwich structure, top down lighting, 500 X magnification, polarized light. At 500X mag., the frit still appears smooth; no voids or bubbles are observed, there is virtually no microstructure.	

Figure 14 – This image is shown for comparison and is from an earlier trial using Borofloat (B33) - Vitta Frit - Borofloat (B33) sandwich that was fused October 6-8, 2009. The image shows extensive bubbles and open porosity. Bubbles are typically 50-micron diameter. (B33-(G-1017)-B33, 50X Magnification, Top down lighting, crossed polarizers)	

II. Application of Frit using the Performus V Pneumatic Dispenser:

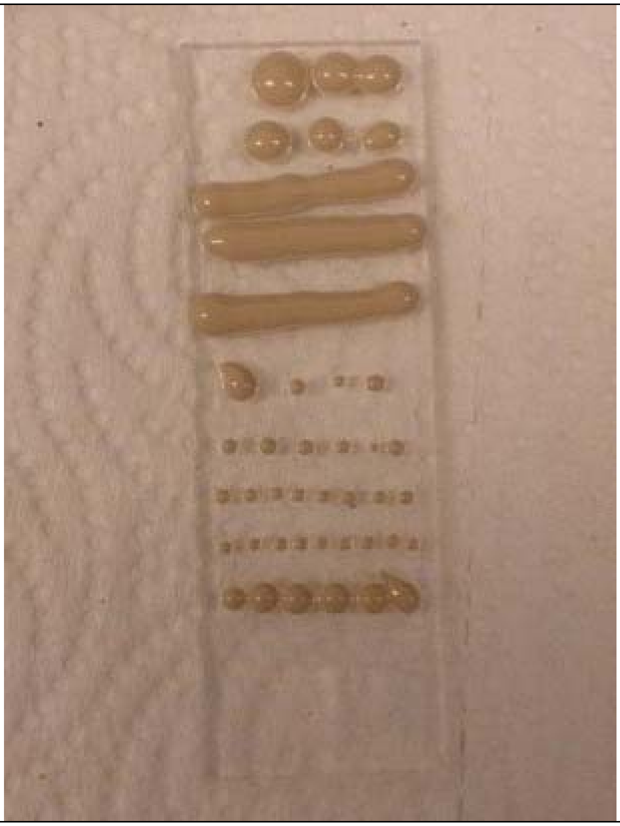
Figure 15 shows the EFD Performus V pneumatic dispenser used for the trials that follow.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 15 - Frit paste has been placed into plastic syringe barrel, the matching (white) piston component has been inserted and is being pushed up with a stick to consolidate frit in the dispensing end of the syringe. The frit is allowed to settle for 1 hour to allow small bubbles to rise to the surface and be vented from the top (blue cap)

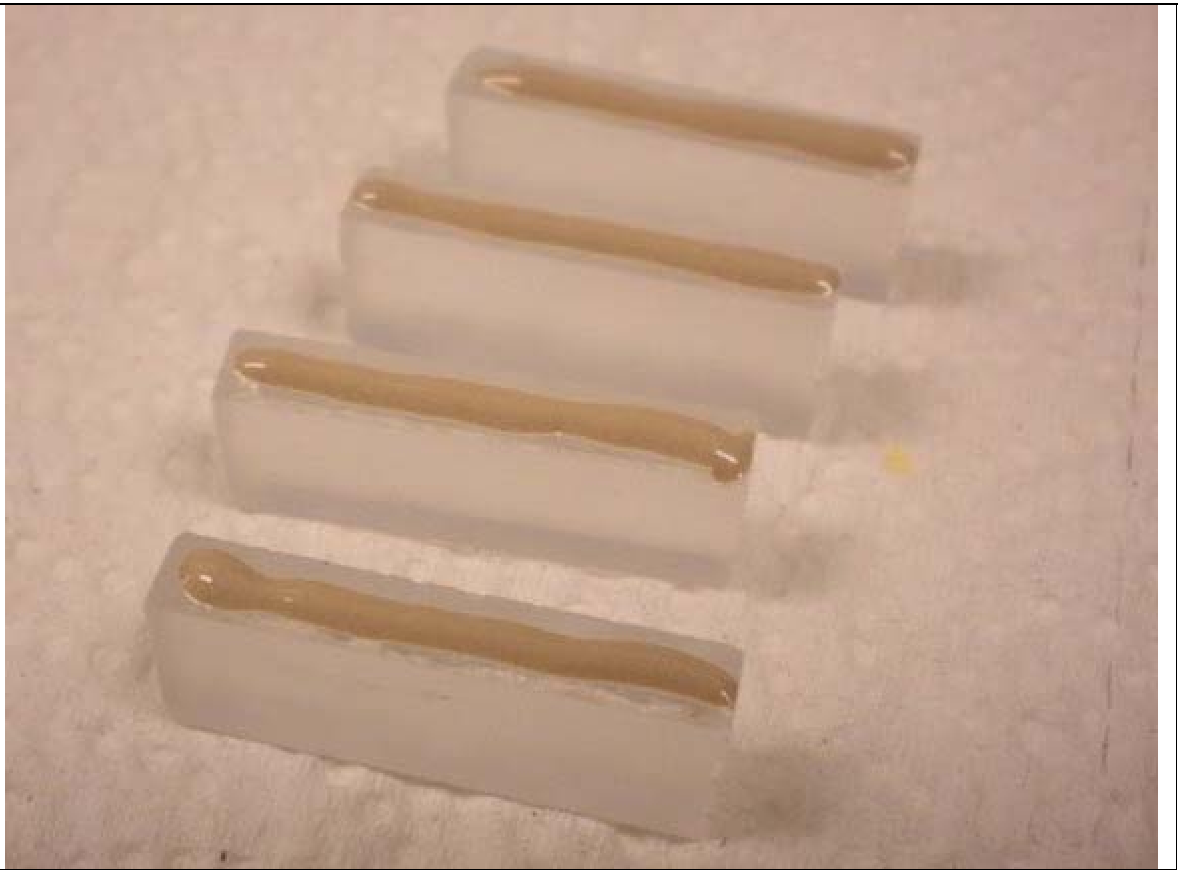
Figure 16 - bubbles are vented from the top orifice. A syringe tip is selected for preliminary application trials. A 16-gauge (grey) plastic syringe tip was used for these trials.

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 17 – Precise control of dispensing pressure and control of the dispensing time (0.001 seconds) allows for deposition of highly repeatable aliquots of frit paste.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 18 – Argonne supplied (B33) 'side walls' are coated with a line of frit.



Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

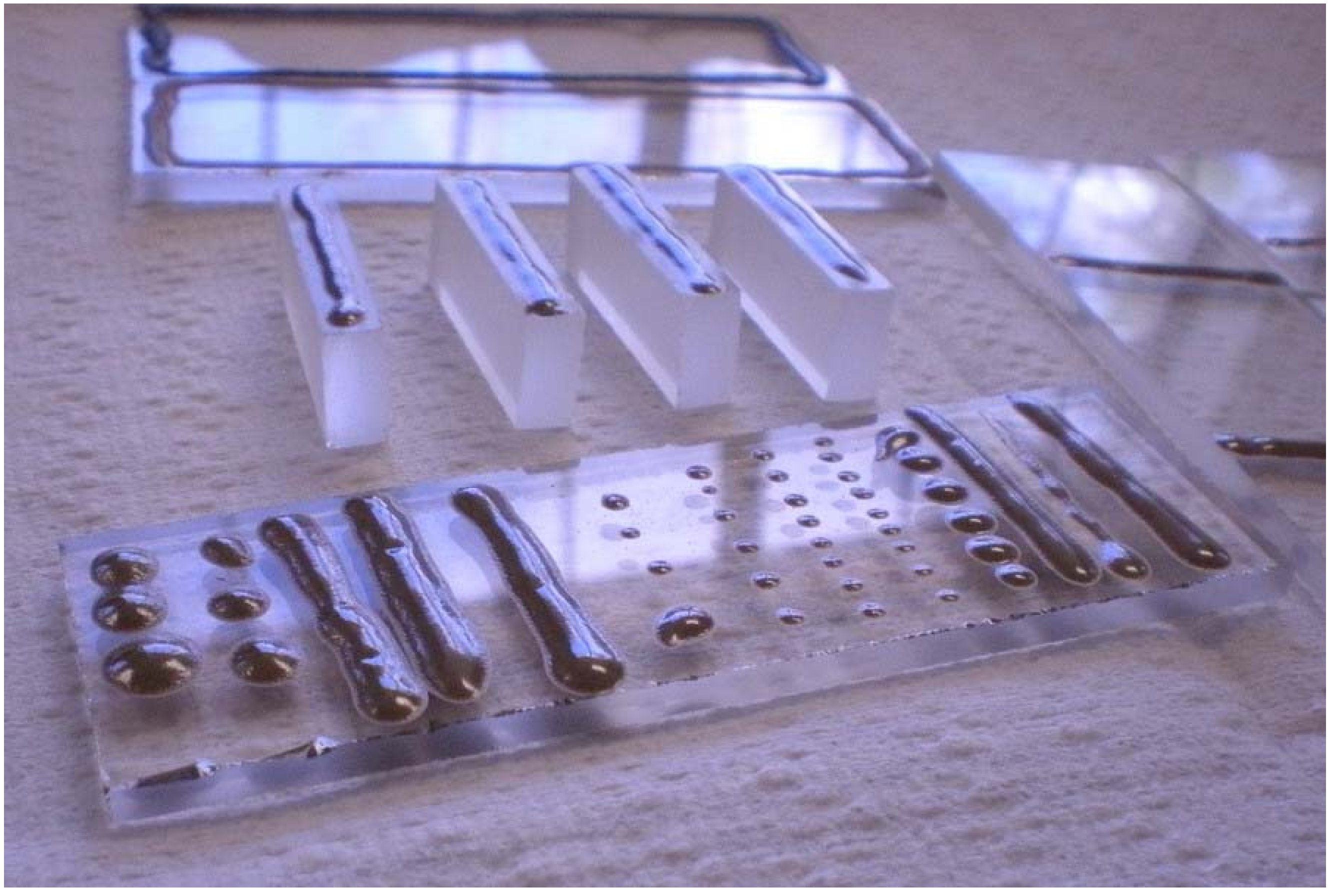
Figure 19 – Argonne supplied (B33) "side walls" are coated with frit. (same samples as Figure 18)	

The samples shown in Figure 18 and 19 were given the 'binder burn-out" and "pre-glaze" heat treatment on Friday, February 19th. Corresponding top and bottom plates were also prepared. I had hoped to prepare full 'sandwich constructions' on the following Monday, but have not been able to follow-though due to illness.

MJM
March 1, 2010

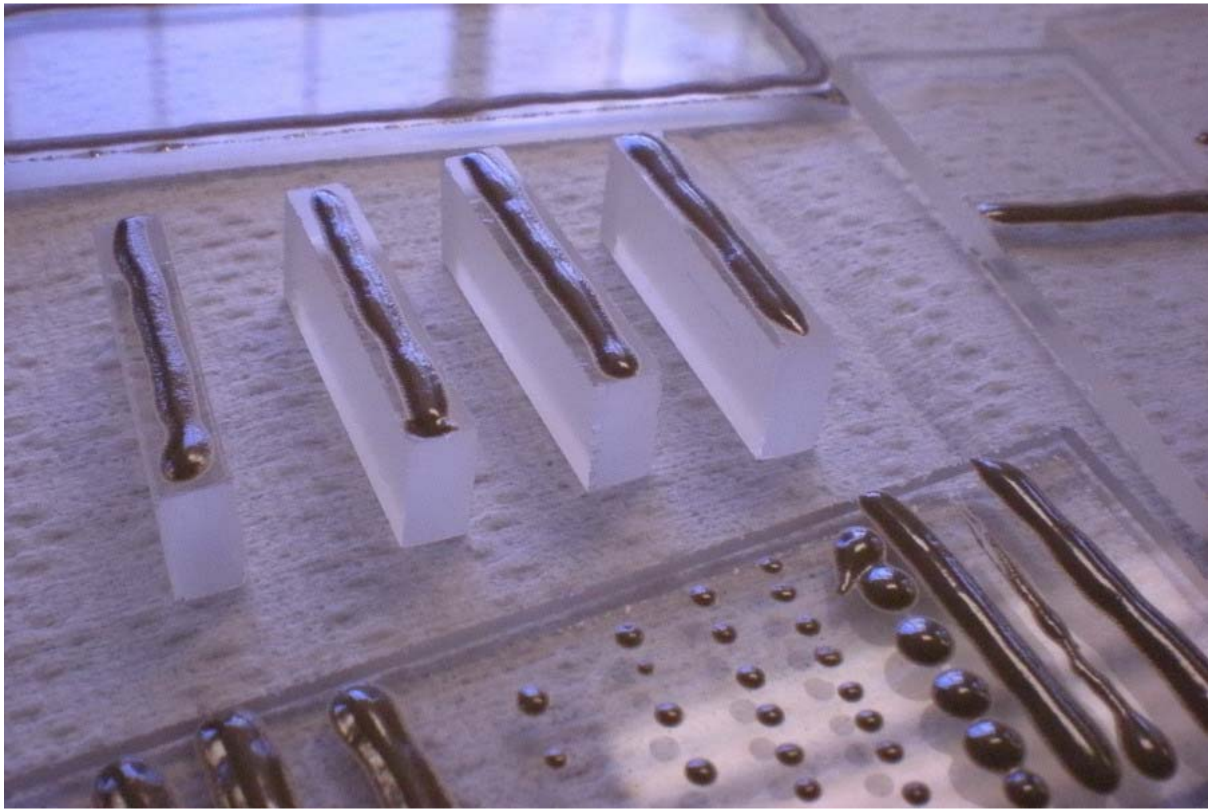
Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

March 26, 2010 - These section includes results based on building small assemblies consisting to a top plate, bottom plate and side walls, using B33 glass and G018-223 Frit. In addition, a sandwich assembly was made, consisting of a silver coated B33 slide, with a top plate frit bonded to the silver. The sandwich remained intact following the frit bonding

Figure 20 - This photo shows the frit after being fired to 'pre-glaze' it. Note the high reflectance of the frit, which shows that it is 'glassy'.	

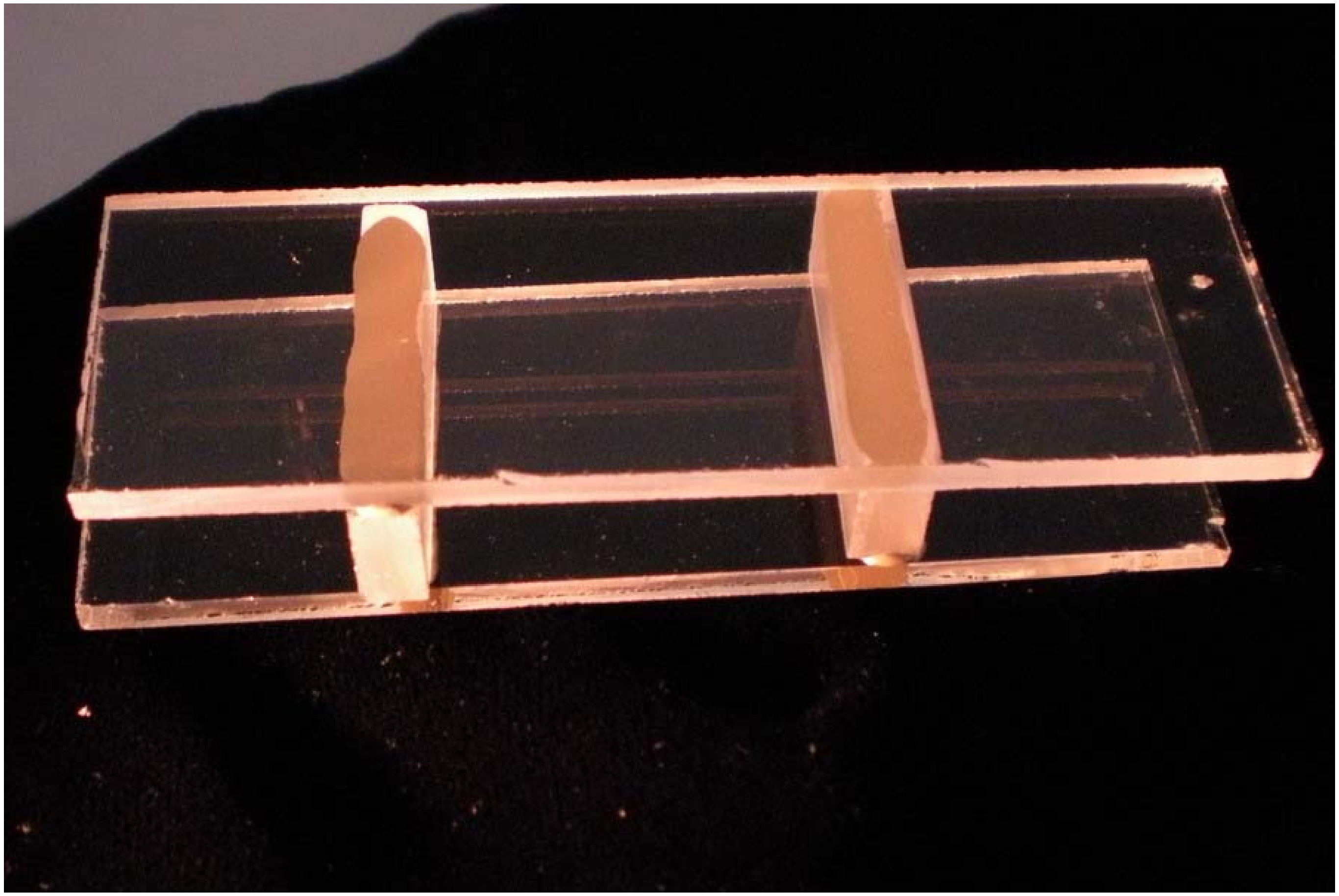
Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com

JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 21 - This photo is similar to the previous image and shows the frit after being fired to 'pre-glaze' it. Note the high reflectance of the frit, which shows that it is 'glassy'. The four pieces in the center of the photo were prepared as 'side walls'	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 22 - This photo shows an overview of the samples prepared February 19th. The 'side wall' samples will be combined with 'bottom plates' seen on the right, and a top plate, not shown, to form a mini-assembly. The sample with multiple strips and dots (bottom of picture) will be sandwiched with a silver coated B33 sample (not shown). The sample at the top of the image with a darker colored frit, is B270 glass with DT430 frit. All other samples are B33 with G018-223 frit.	

Figure 23 - This photo shows the fired assembly of top plate, bottom plate, and side walls (B33) frit fired (G018-223) together. The assembly appeared to be solidly fused; no bubbles were observed in the frit. No cracking was observed in the glass.	

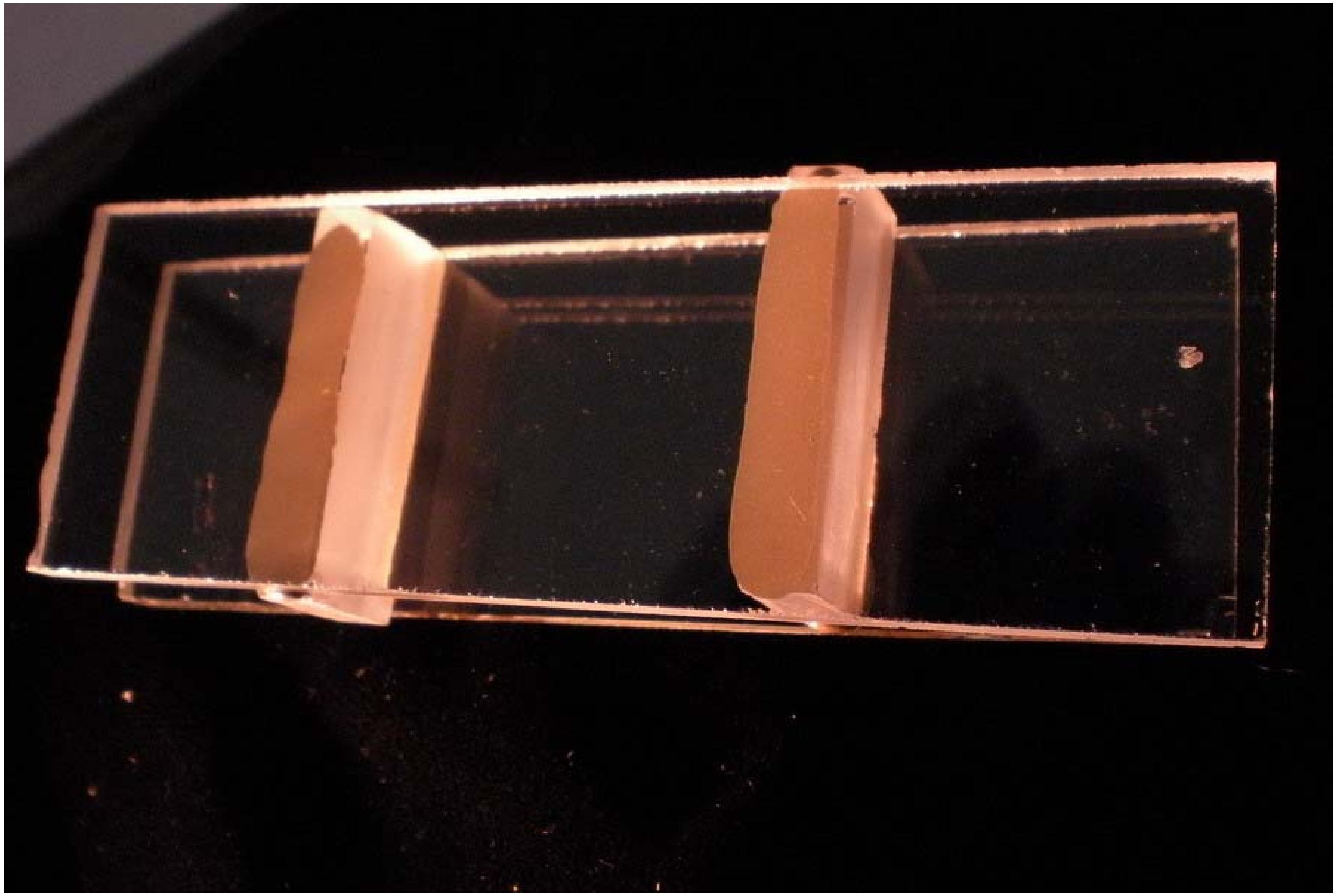
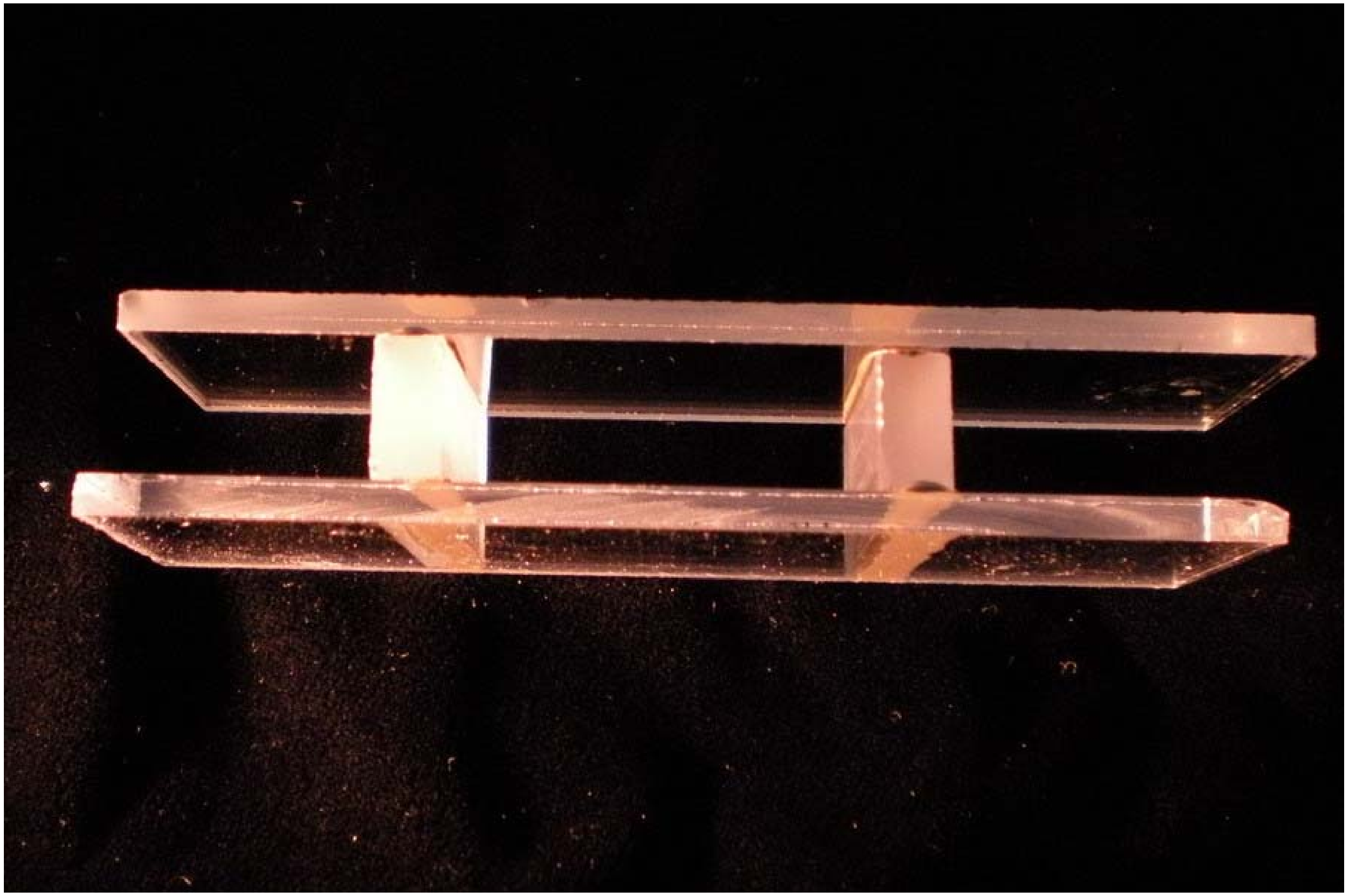
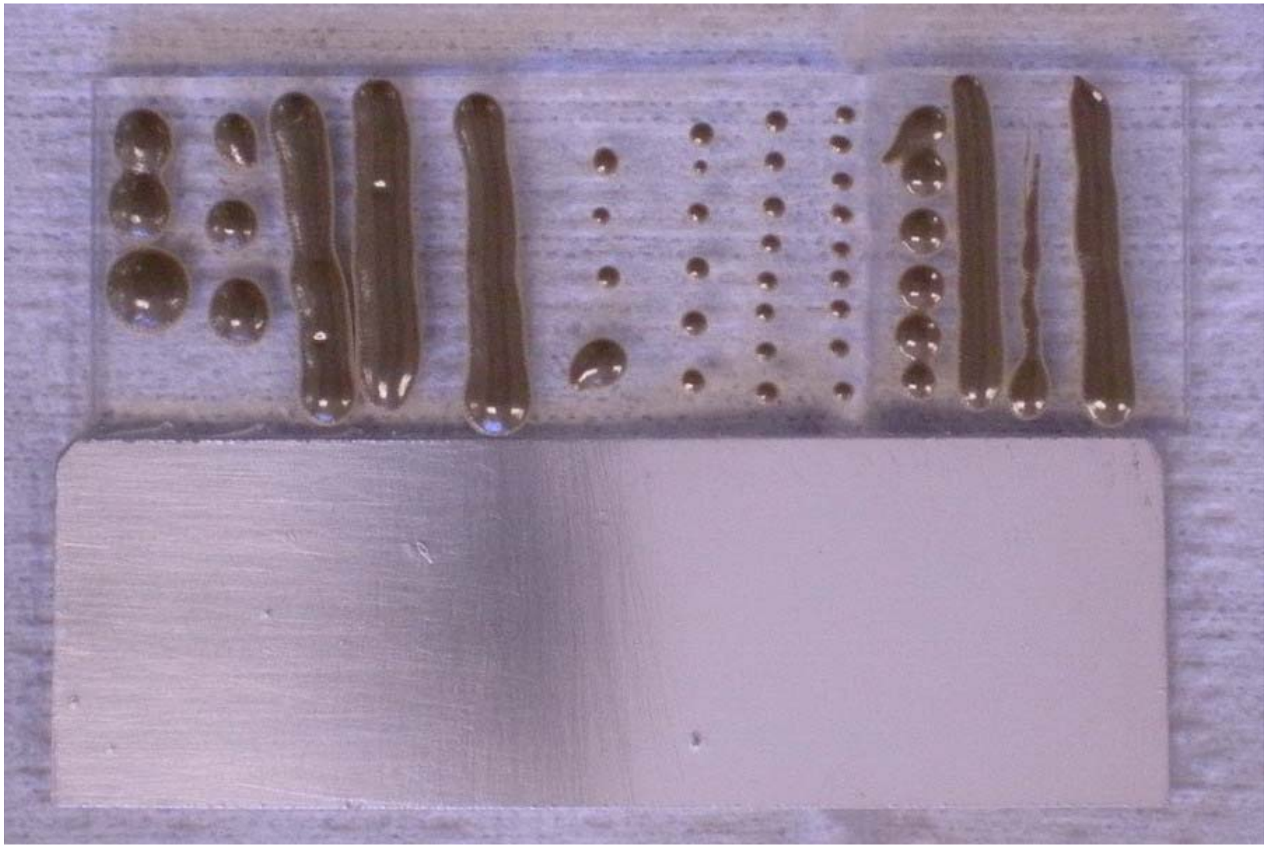


Figure 24 - This photo is another view of the assembly shown in the previous picture.

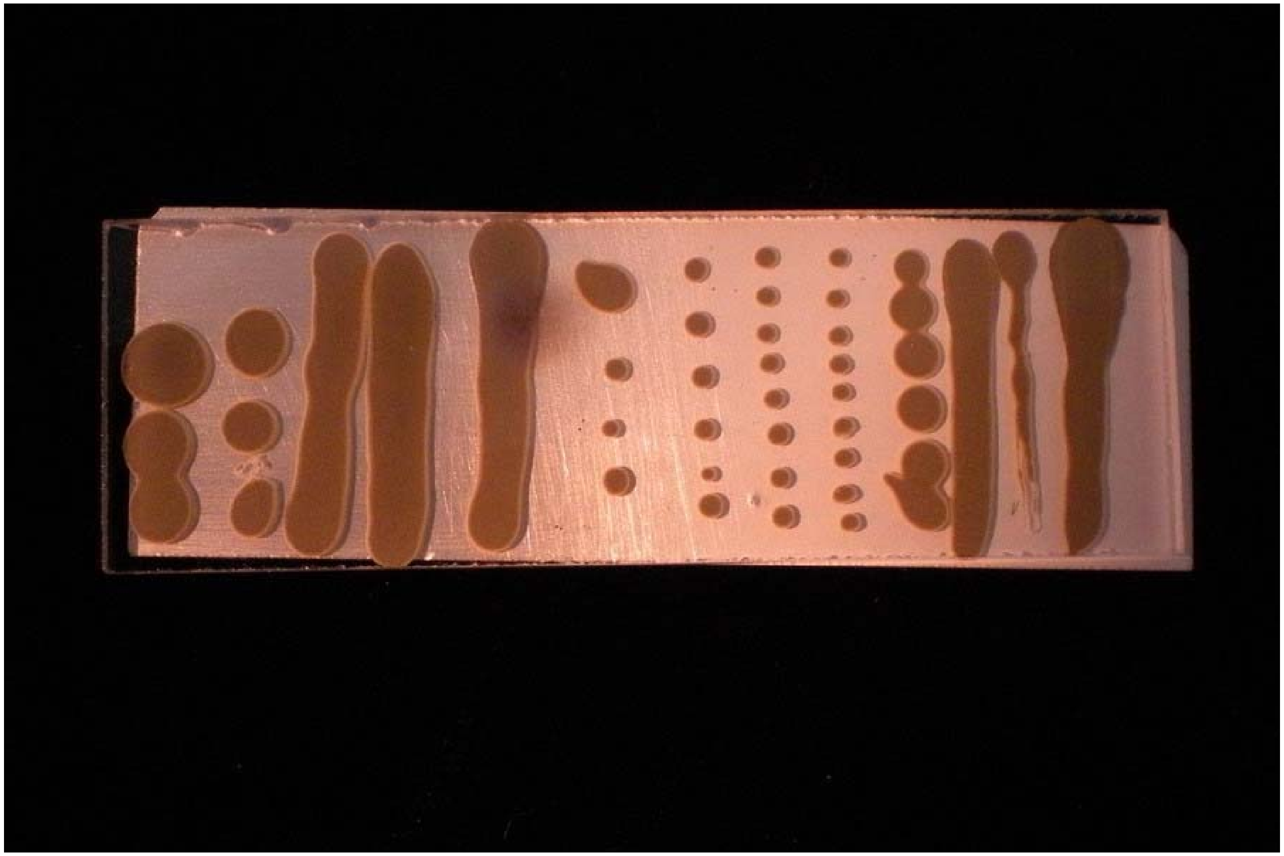
Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 25 - This is a side view of the assembly.	

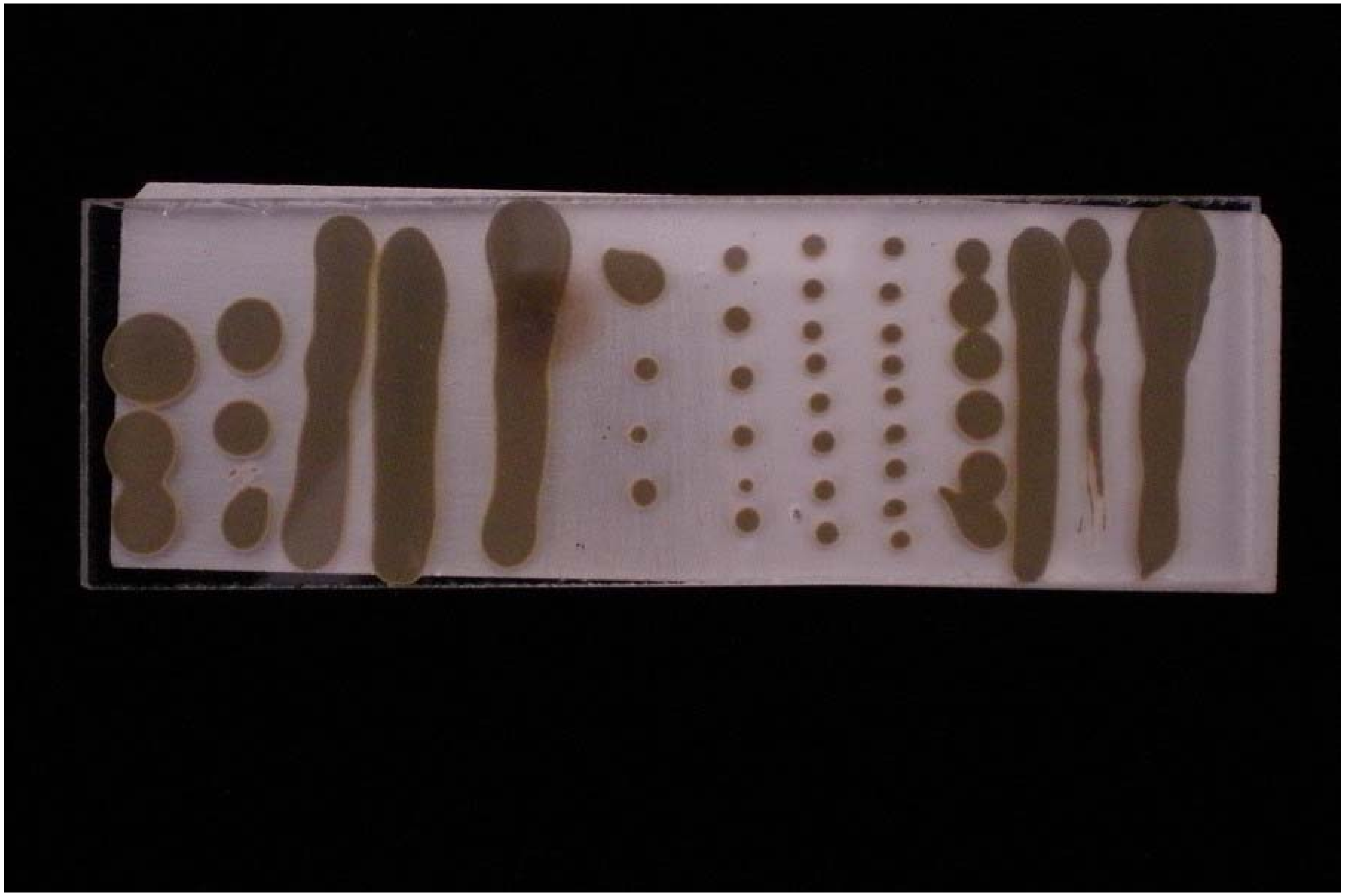
Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 26 - This photo shows a silver plated B33 slide (bottom of photo). The white oxide coating has been buffed from one-half (left side) of the slide. Above is shown a B33 slide with pre-glazed frit applied in the form of strips and dots. These two pieces will be assembled as a sandwich and fired to determine the bonding between frit and silver (both buffed and not buffed).	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 27 - This photo shows the previous sample after being fired to bond the top and bottom plate. The sandwich assembly appears to be well bonded, with no noticeable difference between the bonding in areas (left side) that were buffed vs. those where the silver was not buffed.	

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507

Figure 28 - This photo shows the same sample as the previous figure, with different lighting.	

It is noteworthy that the B33-Silver Coating –Frit-B33 sandwich fused without any evidence of fracture. This seems to be at odds with recent observations from similar trials at Argonne. The samples shown in Figures 27 and 28 will be cross-sections and polished to allow microscopic inspection of the frit bond.

MJM, March 26, 2010

Phone: 978-474-8034 · Fax: 978-475-4047 · www.MinoTechEng.com
JRD Technology Center, 242 Sturbridge Road, Charlton, MA 01507