

MinoTech Engineering™ Inc.

Science & Engineering Consulting

November 13, 2009

To: Henry Frisch,
Robert Wagner

From: Michael Minot and Seungwoo Lee

Subject: Preliminary DT430 Frit Application Trials

Synopsis: DT430 Frit was purchased from Asahi Glass Company (AGC). This frit has a CTE=72 that is appropriately matched to B270 sheet glass, which has a CTE=82 (20C-100C, and 94 20C-300C). This frit-glass combination was specifically recommended (Seungwoo Lee) since DT430 frit is qualified as hermetic for a variety of high vacuum display applications. As part of a first round of preliminary trials, DT430 frit was tested applied to B270 glass as well as Borofloat 33 (CTE= 33) glass.

Frit Preparation: 50.6 grams of DT430 frit was weighed out and combined with 5 grams of alpha-Terpineol (a natural pine oil extract) which serves as vehicle. The combination of frit and vehicle was hand mixed using a Teflon stirring rod forming a smooth paste, which was stored in sealed ball jar until ready for use.

Frit Application: In the initial round, the prepared frit paste was ‘battered’ onto the glass test substrates and smoothed as well as possible. In a later round, the frit paste was loaded into a plastic syringe and applied in thin strips.

Air and Furnace Drying: All samples were left in open air until ready to be furnace dried to remove the vehicle. In some cases, samples were left overnight in air, and in others the samples were inserted into the furnace shortly after being prepared; All samples were heated to 315C (599F) which insures full evaporation of the Terpeneol.

Sealing: Substrates with the dried frit applied were assembled into a ‘sandwich’ consisting of Bottom Glass – Frit – Top Glass. A weight was applied on top of the assembly for most, but not all test assemblies. The assembly was carefully loaded into a furnace pre-heated to 430C (806F). Samples with no weight were fired for 15-minutes. Samples with weight were allowed to return to furnace temperature (about 30 minutes) and then fused for an additional 15-minutes (45-minutes total for most samples).

Results: DT430 frit fired to a smooth, glassy dark colored (black) glass which initially appeared to be well adhered to both the B270 and B33 glass. Closer microscopic inspection of the B33 samples revealed that the fit was micro cracked (as anticipated), likely a result of the poor CTE match between glass and frit. By comparison, inspection of the frit interface between B270 glass and DT430 showed a distinct lack of any visible structure (200X magnification).

An additional trial was run combining B33 glass on one side, and B270 glass on the other with frit between. This was intended as a confirming trial to demonstrate that the micro cracks were substrate related and not related to other variables. In this trial the frit against the B270 was smooth and crack free; the interface with B33 completely delaminated! CTE match matters.

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DT430 frit paste 'battered' onto
B33 glass slides.



DT430 frit paste 'battered' onto
B270 glass slides.



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Close up view of DT430 frit paste 'battered' onto B270 glass slides, showing the variable thickness achieved with this crude technique.



Syringe assembly (30 ml) used for DT430 Paste application



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DT430 frit paste applied to small coupons of B270 glass



DT430 frit after drying. Note some spreading and leveling of the applied frit. At this stage the frit is not glazed. It remains friable and is easily wiped from the glass surface.



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Small laboratory firing furnace, showing sample loaded into the center of the furnace with steel block weight on top.



Borofloat 33 frit sandwich following firing.



B33-DT430 - B33

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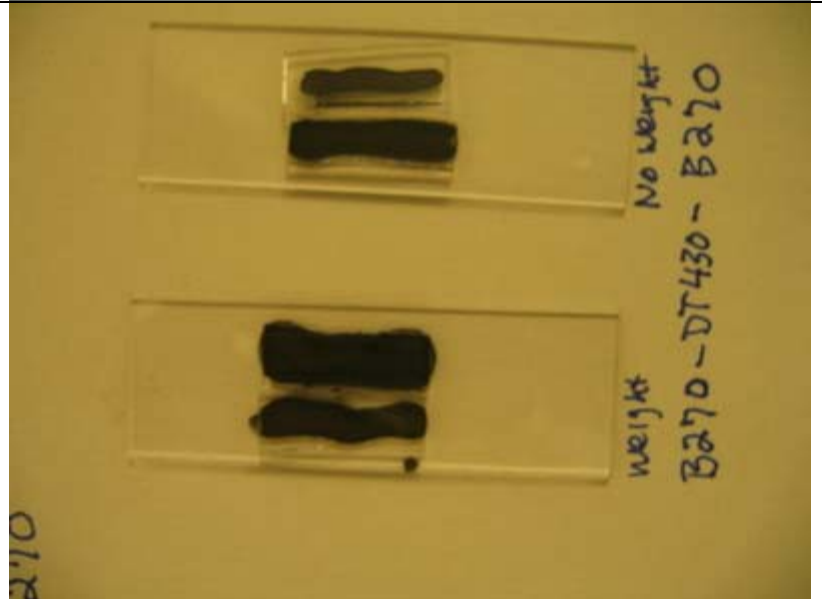
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B270 frit sandwich following firing.



B270 test samples fired with and without weights on top.



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B270-Frit Micro-structure (50X magnification)



B270-Frit Micro-structure (200X magnification)



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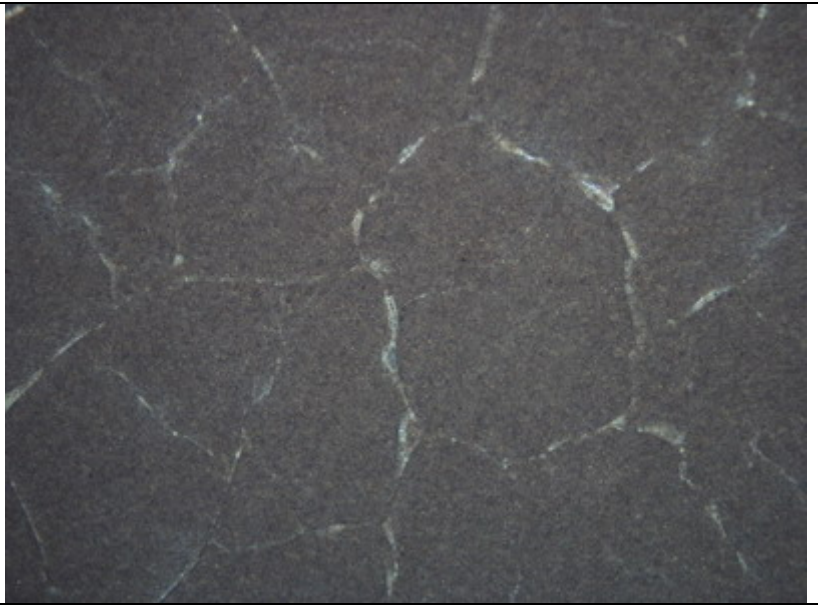
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B33-Frit-B33 Frit
Microstructure 50X Mag top
showing cracks



B33-Frit-B33 Frit
Microstructure for the opposite
side of the sample, 50X Mag
top lighting showing cracks



RESULTS YOU CAN MEASURE

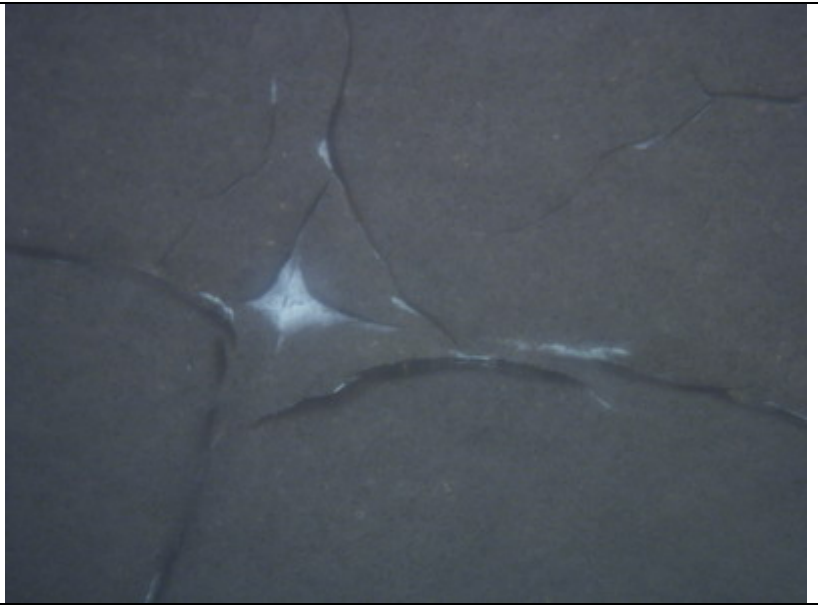
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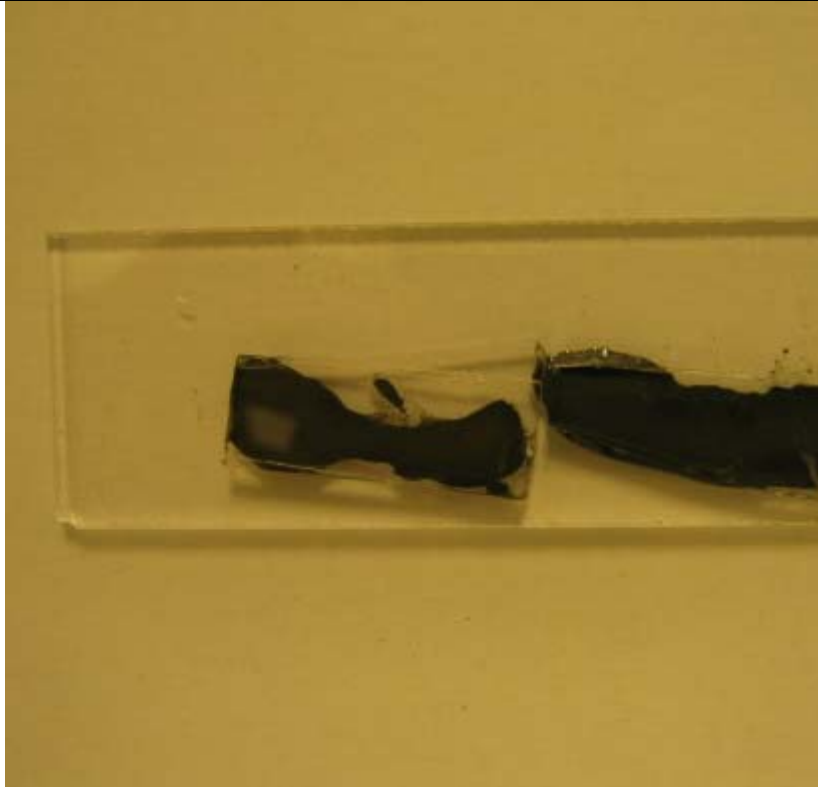
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B33-Frit-B33 Frit
Microstructure for the opposite
side of the sample, 200X Mag
top lighting showing 50-micron
cracks



Borofloat Bottom Glass + Frit +
B270 Top glass



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