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Investigating Bubble Formation in Butt Seals with the Aid of an Electron Microscope

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Abstract: This paper describes observations of saw cuts in quartz and borosilicate glass with both silicon carbide and diamond wheels. I show which surface topology is prone to bubble formation in subsequent butt joints. The observations are largely phenomenological, with minimal theoretical speculation as to why the bubbles form.

Introduction: Glassblowers have long observed that borosilicate and soft glasses, when cut with a silicon carbide abrasive wheel and left unetched, will form a line of bubbles at the joint when subsequently sealed. Quartz does not suffer the same defect under similar procedures. When diamond abrasive wheels are used instead of silicon carbide, the bubble formation at the seal is as good as eliminated in the borosilicate glasses as well.

The wheels used in the investigation are 14" diameter 120 grit silicon carbide and 150 grit diamond, cooled with water. The wet saw rotates at 1725 RPM, yielding a face velocity of 6322 surface ft/min. The test samples are 12mm diameter quartz and Pyrex 7740 tubing, standard wall. I made every effort to duplicate the feed rate for each cut; I tried to match the feed rate of that necessary for the silicon carbide wheel, recognizing that it would be somewhat slower than what the diamond could manage. I dressed the wheels before every cut.

Before making the butt seals, I cleaned the samples in an alkali detergent ultrasonic bath for one minute, followed by a hot water and then distilled water rinse. The samples were allowed to air dry. I treated the samples in the micrographs in the same manner.

The micrographs are SEM (Scanning Electron Microscope) images. Without getting into the workings of electron microscopes, let me just add that images were captured using a relatively low (5 kV) acceleration voltage, and that the samples were sputtered with a thin film of gold/palladium to eliminate charging.

Findings: In the interest of comparison, I include only micrographs of the same magnification, in this case, 10,000 X. I hasten to point out, that by themselves, these miniscule perturbations would form bubbles so small that no conventional light microscope could possibly resolve them. These clearly are not the whole cause for the formation of bubbles visible to the naked eye. Rather, these images show the different effects the two abrasives have on the surface of the glass itself, which leads to the formation of bubbles.

Photos 1 and 2 show the effects of the 150 grit diamond wheel. Photo 1 is the quartz sample, photo 2 is the Pyrex. It is important to notice the similarity of the two surfaces.
Fractured features are clearly evident in both the quartz and Pyrex. Characteristic Wallner lines and hackle marks are evident in both samples. In fact, there is nothing here which distinguishes the two from one another. The diamond, being very much harder than either of the glasses, evidently cuts by means of small scale fractures at the interface of the glass and the diamond particles. These micrographs bear out the assertion that glasses belong to a category of materials that "undergo predominantly microbrittle fracture in the process of chip formation in grinding."

The next two photos, showing surfaces left from the 120 grit silicon carbide wheel, are as different from each other as they are from the first two. Photo 3, the quartz, displays much smaller surface features than photo 1. However, close examination still reveals a fractured surface. The Pyrex in photo 4 appears "smeared," perhaps fused at its surface. The speculation here is that the silicon carbide, being not nearly as hard as the diamond, generates enough friction at the interface to melt the glass. The quartz, having a much higher melting range, still must fracture at the interface.

Hardness is a measure of the ability of the surface molecules or atoms to resist being pushed aside or otherwise displaced. Without getting into the intricacies of the various methods of hardness testing, I will use this space to describe the one most commonly used in this country for glasses, ceramics and abrasives. It falls under the general
category of microhardness testing, and is known as Knoop Hardness (HK), named for the inventor of the characteristic indenter. The Knoop indenter is a diamond faceted in pyramidal form in such a way as to leave a diamond shaped indentation in the test material such that one axis of the diamond is longer than the other, the approximate ratio being 7:1. The hardness number (HK) is the ratio of the load applied to the indenter to the unrecovered projected area of indentation. Simply put, the number varies with either the change of force applied, or hardness of the material tested. On this scale, Pyrex has a hardness of 500, quartz 800-1000, silicon carbide 2000-3000, and diamond 7000-10000 HK.

There is another difference in the properties of these two abrasives which should not be ignored. The thermal conductivity of diamond is 50 times better than silicon carbide, diamond being around 2100 (W/m x K) to silicon carbide's 42 (W/m x K). This means that the diamond carries away the heat generated in the grinding process much better than the silicon carbide, further contributing to efficient microfracture chip formation.

Photo 5 is the image of a surface starting out similar to photo 4, but subsequently etched in 50% HF for 90 seconds.

Observations in the glass shop show that only photo 4 represents a surface sure to form
a line of bubbles when fire polished and sealed. It is the only surface which appears to have undergone thermal effects, in this case, melting. This is the major difference apparent in these micrographs. What might also occur during the interaction of the glass with the silicon carbide wheel in this severe process is the incorporation into the glass itself of some impurity present in the wheel, either as abrasive or as binder. It may become trapped behind the fused surface, the ultrasonic cavitation unable to liberate it, or become a component of the glass chemistry itself, reacting poorly upon reheat.

Conclusions: In some ways this investigation gives rise to more questions than it answers. While we can see here an obvious connection to bubble formation with a distinctive surface, it only goes part way toward explaining why. If this were the whole explanation, then why do our tungsten carbide glass knives, when we ply them to Pyrex in a process which seems much less severe, form bubbles in the same manner? Do we in fact, generate as much heat at the interface because the pressure transferred to the surface of the glass, reduced to a knife edge, translates to heat? Why do side ring seals for double and triple walled vessels, having been ground free hand with a silicon carbide belt on a wet sander, not form bubbles?

It occurs to me that we, as glassblowers, know much about glass from experience and observation. While this is valuable knowledge won in legitimate fashion, new methods of investigation, such as electron and atomic force microscopy, available to us only recently, can deepen our understanding of the materials we work. The more we understand about glass, the better able we will be to expand its uses and demand. There is much investigation to be done in this area. This represents only a scratch on the surface, as it were.

Notes:
2. The formula is: $HK = \frac{P}{A} = \frac{P}{C^2}$, where $P$ is the load applied in kilograms; $A$ is the unrecovered projected area in mm$^2$; $L$ is the measured length of the long diagonal in millimeters; and $C$ is 0.07028, a constant of the indenter relating to the square of the length of the long diagonal.