Fracture Analysis, a Basic Tool to Solve Breakage Issues

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Biography

T. Ono received his B. S. and M. S. degrees in inorganic chemistry from Nagaoka University of Technology, Niigata, Japan, in 1988 and 1990, respectively. In 1990, he joined the Shizuoka Technical Center of Corning Japan K. K. where he has been engaged in research and development of the finishing technologies of LCD glass substrates and other glasses. He has also provided extensive fracture analysis support to resolve breakage problems in customer processes.

Abstract

Breakage of glass substrates in Flat Panel Display (FPD) manufacturing processes can cause serious problems of productivity and product quality/ reliability. It is known that the mechanism associated with breakage is crack propagation by tensile stress concentration at a damaged point on the glass, which serves as an origin of the breakage [1]. Elimination of breakage requires the elimination of the origin and/or reducing the tensile stress. However, this solution can be applied only if the location of origins and the kind of the tensile stresses is known.

Fracture analysis can provide information on both the tensile stress and the origin of breakage [2, 3]. This analytical technique gives important information in determining mechanism of breakage, such as direction

of crack propagation, type of the stress, direction of impact and friction, location of the origin. All important information is "memorized" on the broken pieces and can be obtained through microscopic observation. A process where an origin is created is often different from the process where tensile stress is applied. The breakage mechanism can be understood by combining the information from fracture analysis with process information.

The analysis method will be explained using actual cases. Also, the application of the method for optimization of cell-cutting process will be explained

Glass Breakage

The mechanism of glass breakage is that the crack propagates by tensile stress that is concentrated at the origin, which is a small damage site or crack on the glass surface, or in the glass body. The relation of the failure stress and size of origin is explained with Eq. 1 [4],

$$\sigma = Y \frac{K_{IC}}{\sqrt{c}} \quad (1)$$

where Y is constant depending on the crack and sample shape, K_{IC} is fracture toughness and c is crack size. Glass with a larger crack size, c, could be broken at lower failure stress.

Although the stress applied for glass is lower than the failure stress, a crack can be propagated in the atmospheric condition, especially by water. This phenomenon is called Subcritical Crack Growth (SCG) caused by stress corrosion, in which the molecular of water cut the Si-O bonding chemically [5]. The durability of the stress corrosion of glass is indicated with n, crack growth parameter. The glass having large n value is durable for SCG, which means it has long life reliability. The reliability of several different FPD substrates were studied by Gulati et al. [6].

Origin

Origins are typically generated by such mechanisms as indentation, impact, or friction during FPD processes. There are many opportunities to generate these damages during FPD manufacturing. Indentation damage is irregularly generated when substrates are clamped or chucked on process stages where foreign particles, especially glass particles, are present. Therefore, once the breakage occurs, appropriate cleaning of the glass chips, etc. in the process is essential to prevent secondary breakage. The size of the damage has been characterized using Vickers point indentation [7]. The general relation between the crack size and load is explained with Eq. 2, Eq. 2,

$$c^{\frac{3}{2}} = \chi \frac{P}{K_{IC}} \quad (2)$$

where c is crack size, P is load, K_{IC} is fracture toughness.

χ is a constant which relates to the material properties and geometry of the indenter.

Damage by impact, which is principally the same as indentation but with loading speed much faster than that of indentation, is generated mainly on the substrate edge by rapid contact with the supporting rods of the cassette or the alignment pins. This damage is easily generated because stress is localized by point contact. Friction damage is created when placing glass into cassettes, rubbing with the alignment pins, and sliding on the lithography stage. This damage mode usually occurs after impact when the loading direction of the impact is not perpendicular to the surface or edge. When the friction damage is created, it can often be invisible, but becomes visible after acid etching.

Stress

Tensile stress is the force needed to propagate damage during substrate breakage. Thermal and mechanical stresses are the main sources of tensile stress in the FPD process. Thermal stress for the crack propagation is generated when a heated glass substrate is cooled, which can create tensile stress along the substrate edge. The magnitude of the stress is a function of temperature difference, ΔT, as shown in Eq. 3 [8],

$$\sigma = \lambda \frac{E\alpha\Delta T}{(1-\nu)} \quad (3)$$

where α is Coefficient of Thermal Expansion (CTE), E is Young's modulus, ΔT is temperature difference between inside and surface of the glass and ν is Poisson's ratio. λ is a constant that depends on Bio's constant that relates to the thermal conductivity of the glass and thermal diffusibility of coolant. The constant is 0.7 when the coolant is air.

In addition, CTE mismatch between glass and deposited film material increases the magnitude of stress. Mechanical stress is usually due to bending during handling of the substrate, which is generated when flat glass substrate is warped, frequently due to deposited film – substrate CTE mismatch. Bending stress is also generated when warped substrates become flat by vacuum chucking, or clamping. Panel sag during handling also generates bending stresses. Tensile stress by bending is generated not only at the edges, but also on glass surfaces. The tensile stress σ created by bending can be also calculated with Eq. 4 [9],

$$\sigma = \beta \frac{wa^2}{t^2} \quad (4)$$

where w is uniform load, a and t are short edge length and thickness of the glass substrate, respectively. β is the constant which decided ratio of short/long edges length.

Another source of mechanical stress is centrifugal stress by rotation with spin-drying or spin-coating processes. Stress increases with substrate size and with rotational speed.

In general, the failure stress has been used to evaluate mechanical strength of glass. However, the failure stress is strongly influenced by the size of the origin (non-conformity) on the glass substrate as indicated with Eq. 1. Understanding origin size and mechanism of breakage is useful information for identifying and eliminating breakage during FPD process. In the actual case, glass can be broken under the failure stress as calculated with Eq. 1 due to the residual stress created by damage such as impact or friction. These residual stresses accelerate the crack opening. .

A. Fracture Analysis

When glass is broken, "footprints" of cracks are "memorized" on the fracture surfaces. These "footprints" map the fracture event and are strongly related to the origin creation, crack propagation and applied stress. Fracture analysis is structured with two parts, (1) observe the"footprints" on fracture surface to bring the information of origin and tensile stress, and (2) analyze the information with process information to identify the breakage mechanism. Table 1 shows the steps of the fracture analysis until obtaining the solution of the breakage issue [10].

Table 1. Process steps of fracture analysis

A. Obtain information from glass surface Forking, Hertzian cone, Chatter mark, Residue	**B. Obtain information from fracture surface** Wallner lines, Hackle marks, Mirror region, Sharra scarp, Origin
C. Process information Contact point/material, Contact speed/direction, Thermal cycle, especially cooling condition, Breakage loss/trend	
D. Permanent Solution	

Fracture analysis has been applied to various glass breakage events encountered in FPD manufacturing processes and has provided enough information to identify where the breakage originated and how the flaw propagated. The source of the information is on two surfaces, the glass and fracture surfaces, shown as A and B in Table 1.

The details of information taken with the fracture analysis is well summarized in references [1-3, 10,12,13]. A brief explanation of this information is provided below.

A. Information from Glass Surface

Forking: Forking is crack branching. Qualitative magnitude of applied stress can be known from the number of cracks forking as shown in Fig. 1.

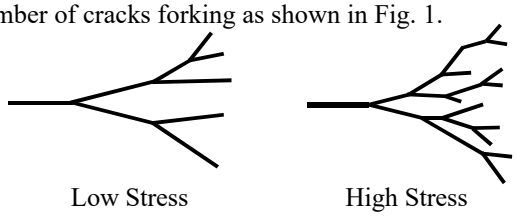

Fig. 1 Forking with high and low stresses.

Hertzian cone: Hertzian cone crack is created by localized impact with a blunt, hard object. Schematic illustration of the Hertzian cone creation is shown in Fig. 2.

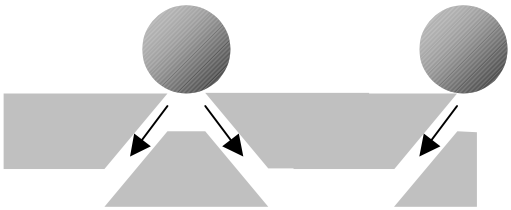

Fig. 2. Schematic illustration of Hertzian cone crack creation from cross sectional view.

Chatter mark: Chatter marks are created by friction, and their shape indicates direction of the friction. The cracks are concave and curve toward the rubbing direction as shown in Fig. 3.

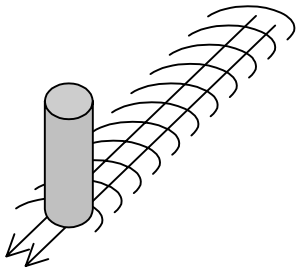

Fig. 3. Schematic illustration of relation between friction direction and shapes of Chatter marks. Drawing shows that the glass is stationary and the object is moving in the direction indicated by the arrows.

Residue: Residue provides information not only on the kind of material contacted, but also the timing when the origin was created or when the crack was run. Patterns on the glass surface such as TFT are ones of residue from the viewpoint of fracture analysis.

B. Information from Fracture Surface

Wallner lines: Wallner lines are rib shaped marks as shown in Fig. 4, which indicate crack propagation direction. The Wallner line is perpendicular to the crack propagation direction. There are three types of Wallner lines that are very useful in determining the course of events. These are primary, secondary and tertiary. Primary Wallner lines indicate that a surface or internal flaw was present prior to the failure event. Secondary Wallner lines occur when the fracture approaches terminal velocity, and tertiary occurs as a result of a mechanical shock, vibration or impact outside the crack front.

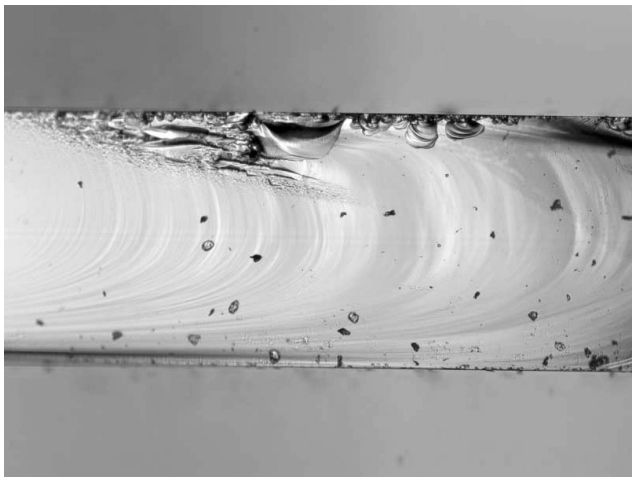

Fig. 4. Secondary Wallner lines created by crack propagation with bending stress. Cracks run left to right (arrow mark).

Hackle marks: Hackle marks are a definitive indication of the crack propagation direction. Typically two kinds of hackle marks can been seen, which are (1) twist hackle, appearing in the region where the tensile stress tilted (twisted) from the crack surface (Fig. 5) , and (2) mist hackle around the origin (Fig. 6). Mist hackles are commonly referred to as velocity hackles.

Mirror region: Around an origin, especially when tensile stress is high, a mirror region can be seen. The applied tensile stress can be calculated from radius of the mirror region [11].

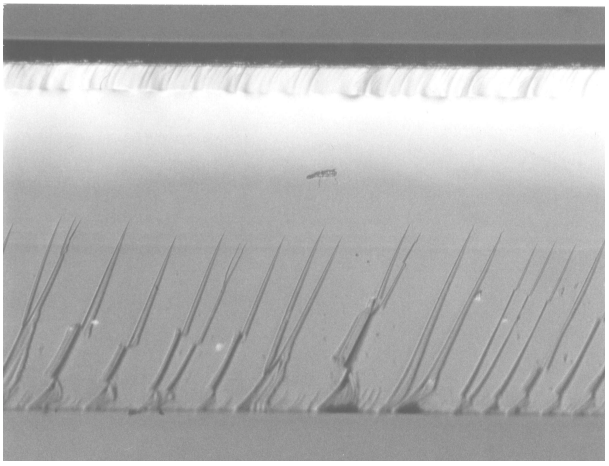

Fig. 5. Typical twist hackle mark created by breaking process following scribe process. The crack front was perpendicular to the Twist hackle marks, and crack propagation direction was parallel to the mark.

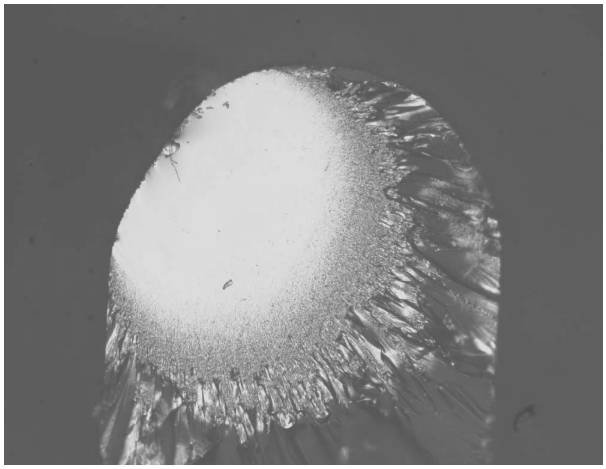

Fig. 6. Fracture surface around origin. Origin is surrounded with mirror region, which is covered with mist Hackle mark. Failure stress can be estimated using radius of the mirror region.

Median Crack Sierra Scarp

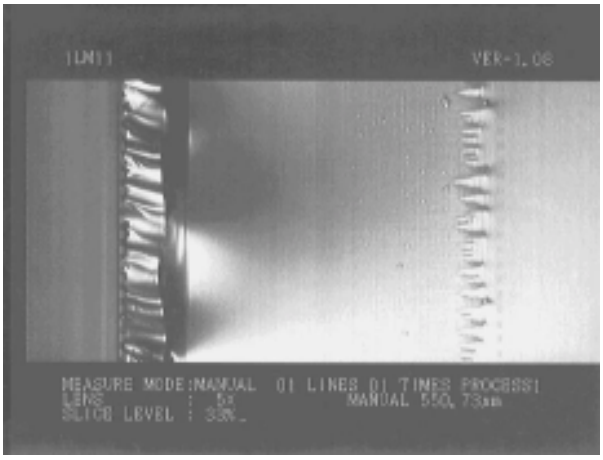

Fig. 7. Sierra Scarp appears on a fracture surface created by scribe/break process. Water was present during median crack prorogation.

Sierra scarp: When water exists at crack front during the crack propagation, Sierra scarp appeared.

Actual Step of Fracture Analysis

Procedure of the fracture analysis when a breakage is reported is explained using a typical case [12]. This case is that breakage on three substrates was found when the sheets were unloaded from the film deposition chamber. In all cases, the cracks separated the sheets into two pieces. The crack shape of three sheets is shown in Fig. 8.

(1) Collect all broken pieces

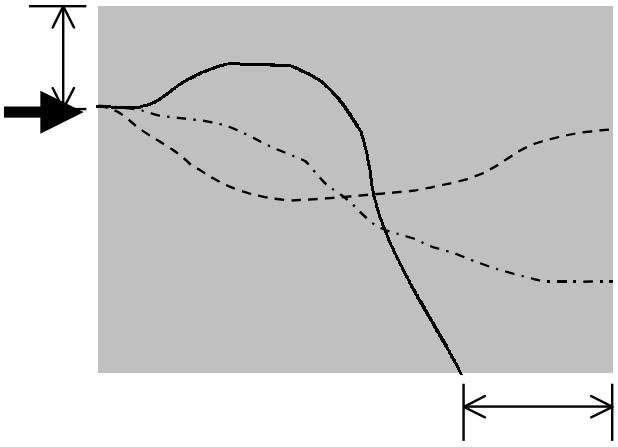

Fig. 8. Crack map of three broken sheets.

All important information is on the broken pieces. It is very important for the fracture analysis that the fracture surface be kept from any additional mechanical damages to retain information. Additional damage such as chipping by friction can erase the information easily. In this case, a sheet was separated already, so two pieces were separately kept.

(1) Make crack maps

Mapping the crack running feature is important to make the analysis easily. It is important to note that you don't play jig-saw puzzling of broken pieces, because the puzzling causes secondary damages. A rough sketch of the crack forking is enough. Also, the distance between the crack end crossing an edge and the nearest corner is important information for identifying the contact point on the edges in the processes.

In this case, the crack map of three broken pieces was obtained as shown in Fig. 8. One of two cracks ending on each broken sheet were located at the same position (arrow mark). This information indicates that the probability of existing origin at this point is high. If a machine creates damage, its location is precisely the same due to its high repeatability compared to a manual process.

(2) Identify crack propagation direction

The crack propagation direction is identified with observation of the Wallner lines or hackle marks to look for a location of origin of the breakage. Not only the crack propagation direction, but also the type of applied stress can be identified from the results of this observation. In this case, Wallner lines which are almost perpendicular to the surface are seen in the middle of the fracture surface as shown in Fig. 9. These Wallner lines indicate two things, (1) that the thermal stress possibly contributed to propagation of this crack and (2) the crack runs right to left.

Fig. 9. Wallner lines on fracture surface Thermal stress is one stress that can generate this shape of Wallner lines. the crack runs right to left.

(3) Identify origin

By following the opposite direction of crack propagation, the origin should be found. The way to identify the origin is three:

1. Find the defect at the center of the mirror region, which is surrounded by a mist hackle mark,
2. Find the defect at the center of hackle marks running dial,
3. Find the defect at the center of semicircle Wallner lines.

Fig. 10 shows around the origin. Origin can be identified at the apex of round edge (arrow mark) by (1) center of radial hackle marks and (2) center of semi circular Wallner lines. This point was the same as the point that the mapping result indicated was a potential location of origin.

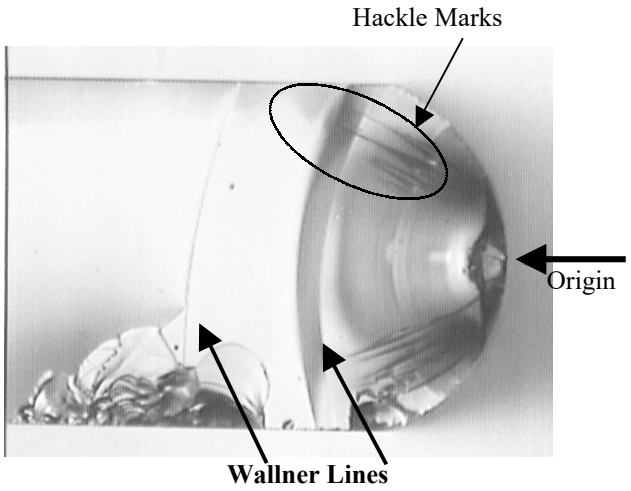

Fig. 10. Fracture surface around origin. Radial hackle marks, symmetrical Wallner lines with arrest lines are present.

(4) Identify root cause of origin creation

After the identifying the origin, observation around the origin is performed to obtain the information related to origin creation. If residue exists, material identification should be done using SEM/EDX or FTIR. Small cracks by mechanical damage become visible after appropriate etching.

In this case, the ground edge around the origin was observed, and the result is shown in Fig. 11. Obtained information was:

1. There is residue, which shines brightly at the apex near the origin.
2. Due to the residue position, only at apex, it is assumed that the sheet rubbed a bar or pin, and its direction is perpendicular to the edge direction.
3. The shape of origin is curved, and hidden cracks having same shape as that of the origin was confirmed (it is difficult to see in this picture). This information implies that the origin was one of Chatter marks, created by friction. Rubbing direction was identified parallel to the edge by the shape of the chatter mark.

Generally, metallic material will appear shiny in an optical microscope with a reflective light source. The residue was analyzed with SEM/EDX to identify its material. The result indicated that the metallic material was stainless steel. Preliminary summary of the mechanism of origin creation is that something made of stainless steel rubbed apex of the ground edge, created a Chatter mark and left metal residue. The chatter mark became the origin of breakage when thermal stress was applied.

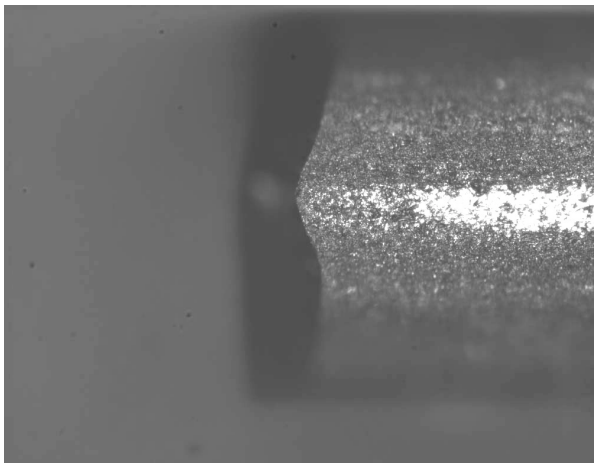

Fig. 11. Around the origin. This picture was taken from apex of the ground edge. Vertical axis in this picture is thickness direction.

(5) Combine with process information

From the preliminary summary of the mechanism of the breakage, other necessary information for the analysis was acquired, such as:

1. The position of the contact point(s) which is pointed out by mapping,
2. Contact was initiated mechanically, not manually, as indicated by the high repeatability,
3. Contacted material is likely stainless steel,
4. The shape of the metal is a pin or bar,
5. The contact happened before the CVD cooling process.

From these conditions, the customer found a process where a stainless steel pin was used for alignment of the sheet. The surface of the pin was abraded by the ground edge.

(6) Identify the mechanism of the breakage

Finally, information obtained from the broken piece and processes is considered to identify the breakage mechanism. In summary of this case, damage was generated by the glass edge rubbing with the alignment pin when the substrate was pushed toward the pin as shown in Fig. 12. After the film deposition, the crack propagated from the origin by tensile stress, which was created parallel to the edge when the substrate cooled down from 200-300°C to room temperature.

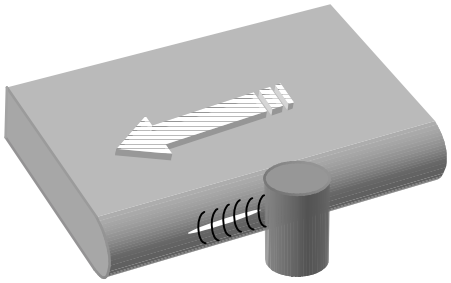

Fig 12. Schematic illustration of the identified mechanism of origin creation on the case. The glass sheet slides in the direction of the arrow against the fixed pin and the relative rubbing direction by the pin is in the opposite direction (left to right in this figure).

Application of the Fracture Analysis for the Cutting Process Optimization

The fracture analysis also can be applied to the optimization of cell/glass cutting process. Scribe/ break method is widely used as a glass cutting method. The glass sheet was first scribed to create a median crack with a tungsten carbide wheel. Tensile stress is then applied with a force on the back side of the scribed surface, or, bending to separate the glass sheet by propagation of the median cracks.

Therefore, the glass cutting process using scribe/ break method is a controlled breakage, where the crack propagates only along the scribe line. Cracks of the glass sheet cutting is the controlled breakage shown in Fig. 13. The scribe line is an origin of the breakage, which crack propagation along with the scribe line is an origin of separation of the sheet. The fracture analysis is a useful tool for the optimization of the process, and/or to analyze the cause of faulty cutting, where cracks run uncontrolled.ncontrolled.

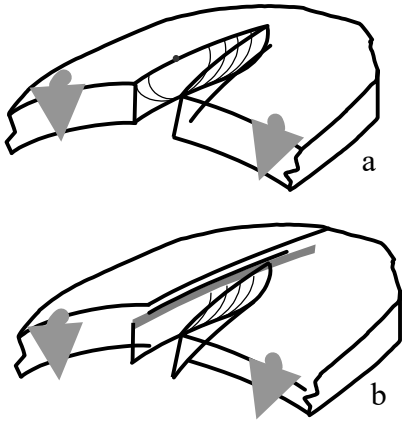

Fig. 13. Schematic illustration of (a) breakage and (b) cutting. The only difference between (a) and (b) is the origin of crack propagation. Irregularly created damage as origin for breakage (a) and median cracks created with scribing as controlled origin for the cutting (b).

The typical fracture surface by good cutting (called "Cut surface") is shown in Fig. 14. Median crack, approximately 100μm in depth is uniformly created, and the median crack propagated portion was smooth, due to low and stable breaking force.

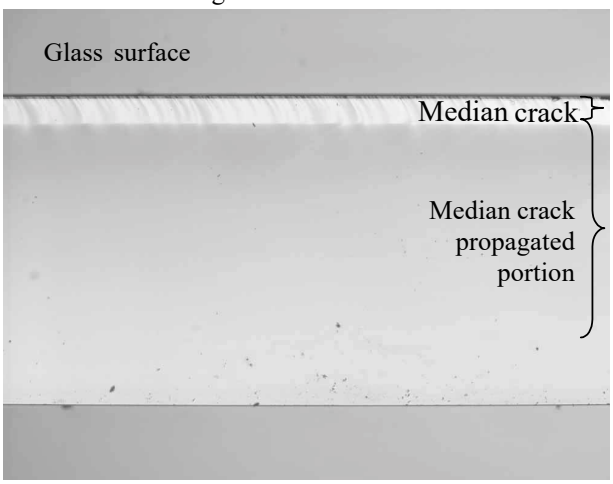

Fig. 14. Typical fracture surface by good cutting. Median crack depth is stable and the median crack.

In contrast to that, Figs. 15 and 16 show some faulty cut surfaces [13]. When individual FPD cells are separated in the final stages of FPD manufacture, the fracture plane sometimes deviates from the score-line. Even when scoring is done properly, i.e., the median crack is deep enough, this break failure is (also called mis-break) can result from inappropriately applied stress during separation. In Fig. 16, although the median crack is deep enough, the fracture surface deviates from the median crack. This fact shows that the tensile stress on the opposite surface of the scribed side is higher than that on the scribed side. The mechanical stress of impact during the cell separation process could also provide sufficient additional stress to cause mis-break.

Fig. 16 shows how insufficient median cracks are often created when the scribe wheel becomes worn or the scribe force is too low to maintain the median crack. Left side median cracks were created uniformly, but on the right side, radial cracks were created. The radial cracks are created just in front of the scribe wheel contact portion. They are shallower than median crack depths at the same scribe load, and have a wavy surface. This crack easily deviates from the scribe line. The scribe wheel runs from left to right.

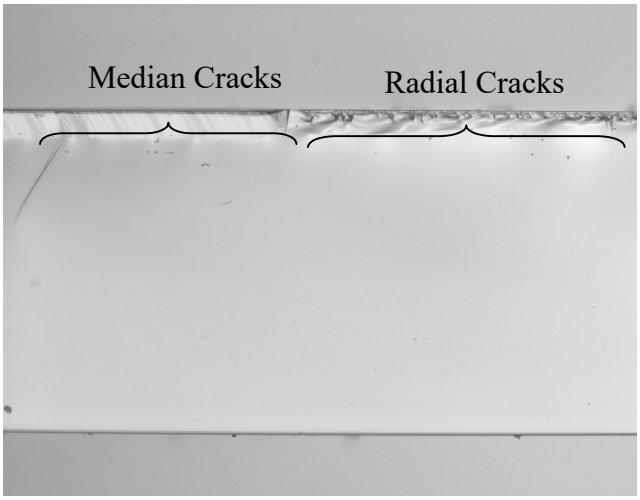

Fig. 15. Cut surface where the median crack stopped. Scribe wheel runs left to right.

Key information for the optimization of the cutting process are Wallner lines and hackle marks at the median crack propagated portion, as well as median crack feature.

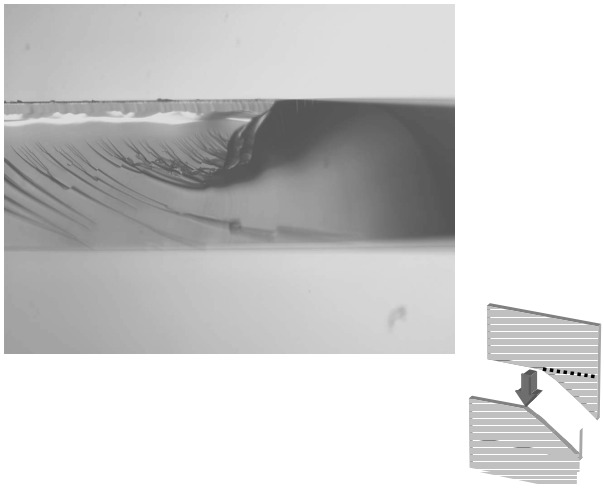

Fig. 16. Fracture surface of miss-breaking sheet by inappropriate separation [13].

Conclusion

1. Fundamentals and applications of fracture analysis, the effective tool to solve the breakage problem, is explained. The fracture analysis can bring reliable evidence of breakage from broken pieces. Once the mechanism of the breakage can be identified with the collected evidence, an appropriate solution can be secured.
2. Also, the effectiveness of the fracture analysis for the cutting process optimization is explained. Fracture analysis is applicable for the optimization, because the cutting process using scribe/break method is a controlled breakage.

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This paper was first presented at 2000 SEMI FPD Expo Taiwan.

The Mystery of Softening, Annealing and Strain Points

Posted on 23 March, 2014 by chatterglass

The question "Why?" rather than "What?" motivates this posting. Let me explain because I want to be more specific…

When I bought an electric kiln it came with some pre-programmed schedules. They worked but I wanted to know why. I then read widely on the Internet and found a million more schedules that did the same and added a few more of my own creation. But they are all wildly different, even though they do the same thing. But why?

So I stopped asking the questions like "What schedule can I use to do a bottle slump?" and started to ask questions like "Why do these schedules do a bottle slump?". I had to change from "What?" to "Why?" because I could not see any consistency in what I was reading. It seemed to me that I was living in an era of "superstition and blind faith" rather than "the age of enlightenment".

Because the many physical properties of glass are well defined and simple physical processes are deterministic, and despite glass being rather "weird" as an amorphous crystalline substance, it struck me that the design of kiln schedules seemed to be more about trial and error, and "rule of thumb", all of which can be taken to be a euphemism for guesswork.

So I have to ask why are we behaving like alchemists when we should be behaving like scientists? Why are we asking "How?" when we should be asking "Why?"

So, this posting is one small step towards answering a big question: "Why are the kiln schedules I'm using the way they are?" and I start by looking into the "important temperatures" relating to glass work.

It turns out that the real starting point is to look at the viscosity of glass at different temperatures.

Viscosity Points

There are several notable viscosities for glass that are widely recognised. For each of them there is an associated temperature and standardised tests that can be used to determine them. But you will probably recognise the names as being related to temperatures rather than viscosities.

These are the widely recognised viscosities for glass that I found:

- Working point 10^{4} Poise
- Flow point 10^{5} Poise
- Softening point $10^{7.6}$ Poise (Bullseye & Wiki) or $10^{7.65}$ Poise (Britannica)
- Annealing point 10^{13} Poise

- Glass transition 10^{12} Poise
- Strain point $10^{14.5}$ Poise

Exactly what "Poise" means as a measure of viscosity doesn't really matter so much as recognising that smaller numbers mean less viscous which means "runny". This in turn means higher temperatures.

If you're "mathematically challenged" then terms like "10^{4}" may leave you baffled. Don't worry. It is an "exponential" or shorthand notation that means "10 multiplied by 10 four times, or 10000". So, the superscript number tells you how many zeroes are involved and saves time and confusion when writing very big numbers.
So, the reason for choosing these specific viscosities as standards is because we can compare different types of glass. And the reason we have to be a bit "arbitrary" about choosing these standard viscosities is because glass does not have a sharply defined melting point.

Temperature Equivalents
Temperatures associated with the viscosities depends on several factors, such as the formulation of the glass, including additives such as metal oxides that produce colours. Therefore the temperature at which one particular piece of glass will have a particular viscosity will differ from another piece of glass. Consequently there will be a range of temperatures for each of the viscosities even for a "compatible" range of glass such as the Bullseye and Uroboros FX90 products offerings.

To give a flavour and substance to the significant temperatures relating to Bulleye "COE90" glass and compatible Uroboros "FX90" glass I will work through each of the "special" viscosities in turn. I will also show you that you have to be careful with information you find on the Internet and try to reach some conclusions of my own that will help me understand why kiln schedules are the way they are. I hope you find it useful for your own purposes too.

But a word of warning. I haven't visited every web site and every forum on the Internet which means I haven't gathered together every piece of information that is available. But I do hope I found enough information with which to reach come rational conclusions.

Working point
The working point is the temperature corresponding to the viscosity of 10^{4} Poise. At this viscosity glass is sufficiently soft for shaping activities such as blowing or pressing in a glass forming process.
A forum poster with an un-identified Bullseye Glass Rod chart says "Working Temp 1750°F" (954°C) and a graph from Bullseye (comparing Spectrum and Bullseye working ranges) was used to estimate the working point as about 934°C (1713°F) within a margin of couple of degrees either way. But this is all I could find.

A tentative conclusion is that the working point temperature is towards the upper temperature limit for a typical glass kiln, so somewhere in the range 1713°F (934°C) to 1750°F (954°C).

Flow point

The flow point is the temperature corresponding to the viscosity of 10^5 Poise. At this viscosity glass begins to flow freely if unrestrained. In practical terms this represents the upper temperature of the fusing range.
No useful information was found about flow point temperatures other than a general description in Bullseye Technote 4 that says glass flows under the force of gravity at temperatures above 1500°F (816°C).

A tentative conclusion is that temperatures in the approximate range 800°C (1472°F) to 850°C (1562°F) may be appropriate.

Softening point

The softening point is the temperature corresponding to a viscosity of $10^{7.6}$ Poise (Bullseye & Wiki) or maybe $10^{7.65}$ poise (Britannica). I didn't figure out why I found two definitions for the softening point. At this viscosity glass softens sufficiently to be worked. It represents a relatively low temperature within the fusing range in which glass will sag in a kiln or can be bent in the flame of a lamp-blowing burner.
Someone on a Forum with an un-identified Bullseye Glass Rod chart says "Softening Point 1250 F" and another says "The softening point is from 1180-1270 for Bullseye glasses"

Three people forum quote, without providing a reference, to Bullseye published information that I could not find, suggesting softening points as being 1250°F (677°C) for transparents, 1270°F (688°C) for Opals and 1180°F (638°C) for Gold Pink transparents.

In Technote 5 Bullseye say that a soak in the 1150°F (621°C) to 1250°F (677°C) range is often used to squeeze or minimize trapped air from between layers. This implies that at 621°C glass has softened enough to slowly sag, implying the softening point has been reached.

A graph from Bullseye (comparing Spectrum and Bullseye working ranges) was used to estimate the softening point as being about 1265°F (685°C) within a couple of degrees either way. From a graph in Bullseye Technote 4 a softening temperature of 1090°F (588°C) was estimated.

Based on a suggestion to do bubble squeeze by a slow ramp of about 200°F per hour from 1000°F up to 1200°F, and a comment saying glass won't fire polish until 1300°F, implies that the softening point temperature is no higher than 1300°F.

Several published Bullseye schedules (including from Warm Glass UK) have segment 1 of their kiln schedules with a target temperature of 1250°F (677°C).

As an aside, Spectrum's own basic schedules have segment 1 rising to target temperature of 1050°F (566°C) and 1250°F (677°C) when heating. This combines nicely with a suggestion at Glass Campus that "If firing to a specific temperature produces a desired effect in Spectrum glass, you will have to fire Bullseye to a higher temperature (about 25°F) to produce the identical effect in Bullseye glass." This leads to an equivalent for Bullseye glass to be approximately 1030°F and 1275°F, which seems to mirror expectations.

Several sets of numbers can be used for analysis of what I've just found. There is reasonable confidence that Bullseye have defined 1250°F for opals, 1270°F for transparents and exceptionally 1180°F for Gold Pink transparent. Several forum sources hint at this same range and claim Bullseye as the source but I could not find the primary source at Bullseye. Graphs from Bullseye imply approximately 1090°F and 1265°F as softening temperatures. Bubble soaks suggest softening temperatures somewhere between 1150°F and 1250°F or somewhere between 1000°F and 1200°F with an upper limit of 1300F as used for fire polishing.

A conclusion is that the softening point temperature might be taken to be an average of around 1260°F (682°C), a narrow range as 1250°F (677°C) to 1270°F (688°C) and an all-encompassing range as about 1090°F (588°C) to 1270°F (688°C).

Annealing point

The annealing point is the temperature corresponding to a viscosity of 10^{13} Poise. At this viscosity glass will hold its shape quite well because it is "not quite solid". The annealing point represents a good temperature at which to anneal glass quickly such that internal stresses are relieved in minutes, as opposed to hours near the strain point.
The annealing point is defined by a specific viscosity so there can only be one annealing temperature for a particular piece of glass. This means that "upper" and "lower" annealing points do not exist, despite what some people are suggesting. More about such "confusional help" towards the end of this posting!

A forum contributor with an unknown Bullseye Glass Rod chart says the "Annealing Temp 940°F". Another says "Bullseye says their glass anneals at 960°F, (though James Kervin lists it as around 980°F.)" without identifying either source. Three people quote information from Bulleye, without providing a verifiable reference, that suggest average annealing points for transparents as 990°F (532°C), opalescents as 935°F (501°C) and Gold-Pink transparents as 882°F (472°C).

Bullseye Technote 5 says glass is rigid with no visible shape changes under 1000°F (538°C) and expands subject to COE. The same Technote also suggests an anneal soak temperature of 900°F (482°C) and that glass will be subject to thermal shock below 900°F (482°C). A graph in Technote 4 suggest the anneal soak temp is at 900°F (482°C)

but another at Warm-Glass claiming to be from Bullseye suggests a temperature of about 970°F (521°C). The older temperature is a pre-2009 recommendation from Bullseye… "As of June 2009, Bullseye has changed its chart for annealing thick slabs. Specifically, the recommended anneal soak temperature has been lowered from 960°F/516°C to 900°F/482°C."

Information at Uroboros for FX90 glass (compatible with Bullseye) is "Use these measured points as 'typical' or an average over the FX 90 colors made by Uroboros": TYPICAL ANNEALING POINTS: Transparents: 977°F (525°C) and Opals: 955° F (512°C)

My analysis of what I found is as follows. Less reliable sources for anneal point temperatures suggest 940°F or 960°F or 980°F. Several sources point to an unidentified publication from Bullseye suggesting anneal point temperatures in the range 935-990°F, exceptionally to 882°F for Gold Pink transparents.

Bullseye Technote information suggests glass is rigid at 1000°F/538°C yet simultaneously suggests an anneal soak temperature of 900°F/482°C which is also quoted as being a temperature beneath which glass is subject to thermal shock. Also consider Bullseye's change from 960°F to 900°F for anneal soaks and the reasoning behind it. But there are reliable averages for Uroboros in the 955°F to 977°F range.

My conclusion it that with no clearly stated average temperature to use as the anneal point, 960°F (516°C) is reasonable midpoint capturing both Bullseye and Uroboros FX90 glass types and matches the pre-2009 Bullseye anneal soak temperature. But for a narrow range 935°F (502°C) to 990°F (532°C) is reasonable, with an all-encompassing range 882°F (472°C) to 990°F (532°C).

Glass transition

The glass transition temperature (or simply "glass transition") corresponds to a viscosity of 10^{12} Poise. However, in reality, the glass transition happens over a temperature range because it relates to reversible changes in which there is smooth but sudden change in physical properties of glass such as thermal expansion coefficient, specific heat and viscosity. In this sense the glass transition can be seen as a change from a hard and relatively brittle state into a viscous molten or rubbery plastic state (or the reverse). Unlike other "special" temperatures for glass work, the glass transition temperature is the only one where a very clear and scientifically observable change is taking place in glass so far as I could tell. All the other "special" temperatures seem to be arbitrarily defined by common consent. It is a rather curious situation to note that glass transition temperature is about the only temperature for art glass workers that does not ever seem to be mentioned!

I have read that "by general agreement it is considered that a liquid on being cooled becomes practically a glass when the viscosity equals 10^{13} poise or where the relaxation time is 10^{2} s." This is an interesting sentence because the viscosity suggests the

annealing point whereas describing a “liquid on being cooled becomes practically a glass” suggests the glass transition point.
This conundrum seems to be resolved by a comment in Encyclopaedia Britannica which says “the annealing point and the strain point lie in the glass transformation range; often, the glass transition temperature and the annealing point are used synonymously, and the strain point marks the low-temperature end of the range.”

So, perhaps, the glass transition temperature is a single measurable point on a temperature-viscosity graph around which is a less-well defined glass transition temperature range that is bounded at the upper end by an annealing point and at the lower end by a strain point. But are the anneal point and strain points coincident with the bounds of the glass transition temperature range?

To add to my confusion, Bullseye Technote 4 say that in the 538°C to 677°C temperature range glass holds its shape but is beginning to soften and calls this the transformation range. So, on the one hand the words “transformation range” hint at the glass transition range but the description and temperature range both hint at a the temperature range being bounded by the anneal point and the softening point.

My analysis of this confusion is that because the 538°C to 677°C range is equivalent to the 1000°F to 1250°F range then the “transformation range” can not be the “glass transition” range because 538°C is within what appears to be the range of expected anneal point temperatures and 677°C is within the softening point temperature range. With no other information than viscosities, the glass transformation temperature is somewhere in the middle of a glass transformation temperature range which appears to be bounded by an anneal point and a strain point.

My conclusion is that I’m still not sure what’s happening here!

Strain point
The strain point is the temperature corresponding to a viscosity of $10^{14.5}$ Poise. At this viscosity glass will hold its shape because it is a solid in practical terms. At the strain point it takes several hours for stresses to relax within as opposed to minutes at the annealing point. Furthermore, stresses that remain in glass at the end of the annealing process become permanent below the strain point.
A practical consequence is that initial heating up to the stress point (and final cooling down from the stress point) for a piece of glass will introduce temporary stresses that will result in thermal shock and breakage if temperate changes are too dramatic.

The strain point is defined by a specific viscosity so there can only be one strain temperature for a particular piece of glass. This means that “upper” and “lower” strain points do not exist, despite what some people are suggesting. More about such “confusional help” towards the end of this posting!

One contributor to a forum with an un-identified Bullseye Glass Rod chart says "Straining point 820°F." but another contributor says the "strain point is 750 F." And on another web site I find 800° F for everything but Satake glass.

I also found three people quote the same information from Bullseye, without providing a reference to information that I cannot find at Bullseye, that suggest an average strain point for transparents as 920°F (493°C), opalescents as 865°F (463°C) and Gold-Pink transparents as 820°F (438°C).

I also found a suggestion that because the strain point is the "solid – not solid" boundary a slow ramping to avoid thermal shock cracks is only needed below 1000°F (515°C). Although I like reasoned arguments I am always suspicious of "round numbers" when physical measures are concerned. I get doubly suspicious when the temperature quoted lies in the anneal point range rather than the strain point range.

At Uroboros I found explicit information for FX90 glass (compatible with Bullseye) as "Use these measured points as 'typical' or an average over the FX 90 colors made by Uroboros": TYPICAL STRAIN POINTS: Transparents: 910°F (488°C) and Opals: 875°F (468°C)".

The only primary source information I could find relating to Bullseye glass was an explicit statement in Technote 4 that says "Subject to thermal shock below approximately 850°F (454°C)" but there are much higher strain point temperatures for some many kinds of Bullseye and Uroboros glass suggesting that some kinds of glass are susceptible to thermal shock at higher temperatures.

My analysis is that the unreliable specifications for the strain point are 750°F, 800°F, 820°F and 1000°F. Because the original source can not be found, I am cautiously confident that the Bullseye strain range lies around 865°F to 920°F, with 820°F for Gold Pink transparent.

Slightly worryingly, Bullseye warn about thermal shock for temperatures under 850°F (454°C). But Uroboros are quite explicit with typical strain points 875°F (468°C) to 910°F (488°C).

My conclusion is that a single average strain point might be somewhere around 890°F with a minimal range of around 850°F (454°C) to 920°F (493°C) and an all-encompassing range of about 820°F (438°C) to 920°F (493°C).

And as a final point, I noticed as I browsed the Internet a reminder that glass doesn't suddenly change from liquid to solid on cooling: "Upon further cooling, below the stress point, viscosity increases rapidly to well beyond 10^{18} poise, where it can no longer be measured meaningfully." What this is saying is that on cooling glass becomes so viscous that you can't measure it!

Working Range
At http://www.bullseyeglass.com/the-working-range-and-what-it-means-to-fusing.html Bullseye compare Spectrum and Bullseye glass in terms of "working range" in the context of an old (but still interesting) legal dispute. They say the working range is "The range of temperatures in which glass is formed into ware in a specific process. For comparison purposes, when no specific process is considered, the working range of glass is assumed to correspond to a viscosity range from the working point to the softening point. (10^4 to $10^{7.6}$ Poise)"
The graph on that web page shows that there's not so much of a difference between Spectrum and Bullseye glass.

In Bullseye Technote 4 they describe the working range as usually between 1000°F (538°C) and 1700°F (927°C).

Buyer Beware
I have now passed on what I've learned but I'm not done with you yet!

You've already seen hints of misleading and inconsistent information that I encountered as I did my research. I want to give you a few more examples that relate to what I call "confusional help" because you get confused when you read more than one of them!

My first example comes from http://www.arrowsprings.com/html/annealing.html and you should now be equipped to spot the problems:
The annealing temperature for any glass is actually a range. The higher end of the range is a temperature set to be safely below any possible chance of distortion. The lower end of the range is a temperature high enough for heat soaking to be effective within a reasonable amount of time. The commonly used temperatures for any particular glass is actually just a temperature chosen as a compromise between the higher and lower ends of the range. In other words, a temperature in about the middle of the range. An exact temperature is not what is important. What is important is that you keep the temperature steady for a period of time before slowly cooling the glass to room temperature.

The clue to one reason why that text is wrong is in the meaning of "annealing point". A second reason is that what the quote is describing is an annealing process, not the annealing point. It's easy to create confusion by using the wrong words (and I'm sure I do it myself).

My second example is different but equally confusional. It is found at http://www.warmglass.com/Basic_Process2.htm:
Unlike many substances, glass does not melt or harden at a single temperature. Instead, it gradually softens and hardens as the temperature changes. The phase during which this transition from liquid to solid occurs is called the "annealing zone." There are three critical points within this zone.

- *The Upper Annealing Point – this is the upper end of the annealing zone, where the glass begins to return to solid form.*

- *The Annealing Point – this is the temperature where the molecules in the glass optimally realign themselves evenly throughout the glass. It's always between the upper annealing point and the strain point.*

- *The Strain Point – this is the lower end of the annealing zone. It's the place where the glass solidifies. The stress (or strain) remaining in the glass at this point is unlikely to be changed or relieved unless the glass is heated up again and annealed again.*

The problem is that we know there is just one annealing point!

My third example I found at http://www.glass-fusing-made-easy.com/strain-point.html:
Annealing glass occurs between the upper and lower strain points, therefore finding the annealing point of a piece of glass is important. The annealing is accomplished by a slow cooling of the glass to and beyond the lower stain point of the glass.

This time we have two strain points when we know there is just one.

My fourth example I reluctantly mention because, on the whole, I like what the author is trying to achieve and it is otherwise a very helpful article. Again we seem to have two strain points. You will find it at http://glasstips.blogspot.co.uk/2010/02/strain-points-and-annealing-ranges.html
There is an upper and lower strain point, although this is disputed by some. There are mathematical definitions for these as well as observational definitions. I do not understand the mathematics of either. In lay terms, the lower strain point is that temperature below which no further annealing can take place. It is safe to assume this is 50C below the annealing point (I think it actually is 43C, but I'm not certain of this number).

So it is safest to control the cooling to at least 5C below the lower strain point. Bullseye find that cooling from the annealing soak to 370C is best – this is much more conservative than is theoretically required – 146C below the annealing soak point. This does take care of any problem of thermal shocking of the glass during the cooling.

The upper strain point might be more properly described as a softening point. This also has scientific definitions. The way I think of it is as being the temperature above which no annealing can occur. Another is to think of it as a point beyond which the molecules of the glass are in relatively free motion – which increases with temperature. This again can be considered (on the rise) as 50C above annealing. However on the way down it is safer to consider it to be not more than 30C above

annealing. This is because the glass temperature lags behind the air temperature (which is what our controllers measure).

As we know, there is only one strain point. I therefore reach a conclusion that when the author says “lower strain point” he actually means “the strain point”; and when he says “upper strain point” he really means “the softening point”. And there’s more I’d argue about too, such as whether it is safe to assume 50°C either side of the anneal point.

So, our tally is now two anneal points and two strain point if you’re prepared to take what you read on the Internet at face value about annealing glass. So, to balance the books, let me describe the annealing process briefly and in a way that does not need “upper and lower” anneal or strain points.

To begin the process of annealing glass you have to choose a temperature to “soak” the glass to dissipate internal stresses. If the chosen temperature is “sensible” it will relieve the bulk of the stresses in all the kinds of glass you have used and will achieve it quickly.

To ensure that stresses are not re-introduced during the subsequent cooling process, and to further reduce the internal stresses, you then have to slowly cool down the glass to beyond the stain point ensuring that temperature differences within the glass object are minimised. This slow cooling is the anneal cooling period in your kiln schedule. Below the strain point any residual stresses from annealing are permanent because the glass has become rigid.

Cooling down from the strain point to room temperature can then happen at any rate that does not cause thermal shock breakage.

So, no mention of “upper” or “lower” anneal or strain points.

But stop for a moment and think. This does not mean you do not have more than one anneal and strain points to consider when designing a kiln schedule. Remember that when you have more than one kind of glass in your creation (eg pink opal and some purple transparent) then each of them with have their own particular anneal and strain points. Life is never simple. You need to take their differences into account. You must choose an anneal “soak” temperature that is able to allow all the glass to anneal at a sensible rate. You also want the anneal cooling step of your schedule to end when the whole piece is at or below the strain point for all the component glass parts.

Some Conclusions

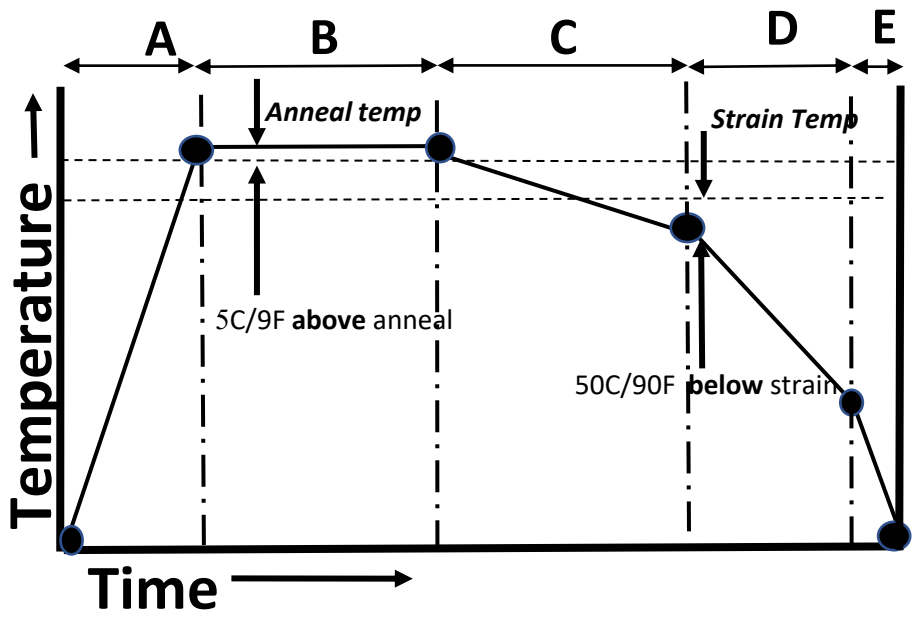
The properties of glass may be downright weird and unusual but is does not mean that the design of kiln schedules should be in the realms of guesswork, naïve superstition and blind faith in someone else’s kiln schedule suggestions. Start asking the question “Why?” rather than “How?”

Make use of what you've just learned. Compare your schedules with what is happening to the glass. Be more scientific.

And finally, my experience over the last few days suggests you must have a healthy mistrust of what you read on the Internet. Look for corroboration but take steps to verify that corroboration is not actually cut-and-paste copying. Look for primary sources of information (eg from Bullseye or Uroboros) rather than trust the guesswork and vague recollections of secondary sources (eg in Forums).

Happy fusing.

Generic Schedule for 33 COE 560C/1050F Strain 525C/950F



A Heat to 5C/9F above anneal temp	**D** Secondary cool rate 50C/90F below strain point
B Hold for X min	**E** Final cool down
C Initial cool rate through strain point	

Borosilicate Cooling on One side (Solid Work)

Thickness		A	A	B	C	C	D	D	E	E
Inch	MM	C/Min	F/Min	Min	C/Min	F/Min	C/Min	F/Min	C/Min	F/Min
1/8"	3.1	130	234	5	5	9	24	43	130	234
1/4"	6.3	30	54	15	10	18	6	11	30	54
1/2"	12.7	8	14.4	30	20	36	1.6	3	8	14.4

Borosilicate Cooling on Two sides (Hollow Work)

Thickness		A	A	B	C	C	D	D	E	E
Inch	MM	Rate C/M	F/Min	Min	C/Min	F/Min	C/Min	F/Min	C/Min	F/Min
1/8"	3.1	400	720	5	5	9	78	140	400	720
1/4"	6.3	130	234	15	10	18	24	43	130	234
1/2"	12.7	30	54	30	20	36	6	11	30	54

Taken from "The Glass Engineering Handbook" By EB Shand