Looking at glass from a different angle: new insights into fracture patterns through transmitted light microscopy

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Abstract

This paper shows the benefit of using transmitted light microscopy together with a Z-scanning software in fractographical analyses of glass. The strength of glass is largely dependent on processes that happen at the microscale. In this research, 52 plates were fractured in a biaxial tensile test. These were divided into five categories according to their fracture pattern. 6 plates were examined with a polarised light microscope and photographed with the Z-axis scanning function. This revealed fracture markings that are barely visible with the naked eye and overlooked when only performing a microscopic analysis of the fracture surface. This led to the conclusion that transmitted light microscopy on glass' fracture pattern is a valuable addition in glass fractography. It gives the researcher an overview of all fracture markings and flaws in one image. This can be used as a guide to find the fracture origin and it gives new information on the crack propagation and local failure processes.

Keywords: glass, microscopy, fractography

I. Introduction

In the last decades there has been an increase of the use of glass as a building construction material. Next to window panes, glass can also be used to build columns, walls and beams. However, a true glass revolution is impeded by its uncertainty in strength. Strength tests performed on glass can yield values as high as 150 MPa and as low as 20 MPa (Veer, 2007). Mostly the lowest value is assumed to be the strength of glass in structural design. This is a waste of material when the true strength of the glass is actually much higher. Therefore, more research in the strength of glass is desirable. Glass researchers generally agree that these lower strengths are caused by flaws that weaken the glass (Bourhis, 2014, Quinn, 2007, Veer, 2007, Wurm, 2007). The types of flaws that occur in glass can be categorised by their location, see Figure 1. This paper follows this classification (Molnár et al. 2012):

- Surface, such as scratches from mishandling or pits from weathering
- Edge, such as grinding or polishing damage
- Volume, such as air bubbles or inclusion formed in the glass during its manufacturig process.

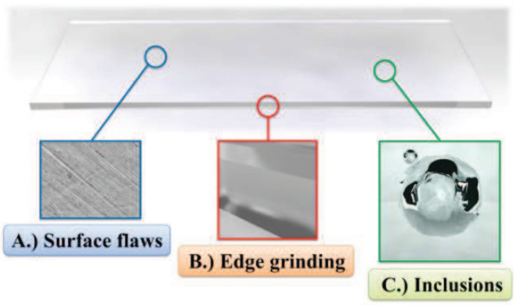


Figure 1: Flaws categorised by location (Molnár et al. 2012)

In this research 52 plates were tested in a biaxial tensile test. Traditionally, the microscopy part of a fractographical analysis is only concerned with the fracture surface of the broken pieces. This paper explores the addition of observing the fracture pattern underneath the microscope. In other words, the angle of observation is turned 90 degrees.

Glass fractography

Quinn (Quinn, 2007) stated that the fractographical analysis of a specimen is just as important as the fracture stress found in the strength test. He argued that a fracture caused by one type of flaw cannot

Figure 2: Wallner lines (Shinkai, 1994)

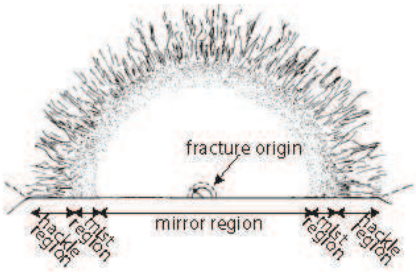


Figure 3: Regions marking the origin of fracture (Quinn, 2007)

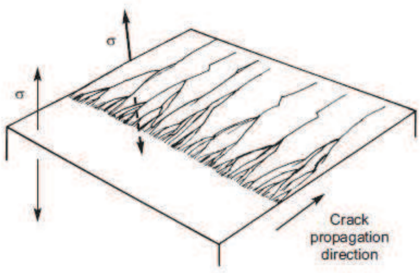


Figure 4: Twist hackle (Quinn, 2007)

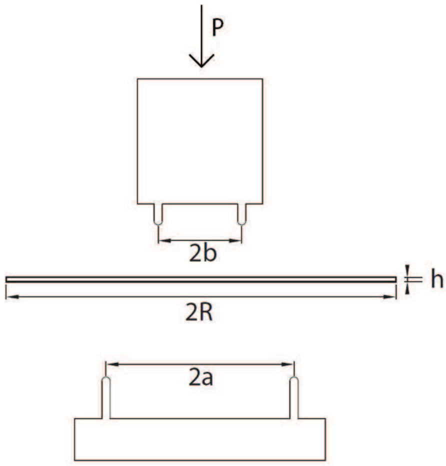


Figure 5: Ring on ring test values

be statistically compared to one caused by another type. He promoted fractographical research to find the origin and fully understand the strength. Such an analysis of glass usually starts with a study of the fracture pattern. Either the specimens are taped to hold the fragments together or the shards are reassembled after failure. From the pattern, the origin crack can be determined by finding the crack that started branching first. The analysis finishes with collecting the fragments at that crack and examining the fracture surface. The flaw that caused the failure can be found by looking for certain marks. Wallner lines at this surface are oriented away from the fracture origin, see Figure 2. Following them back will lead to the flaw of origin (Shinkai, 1994). Around this flaw special marks can be found that prove it is the origin, see Figure 3 (Quinn, 2007). Lastly, twist hackle is sometimes reported, see Figure 4. Twist hackle shows the local direction of crack propagation.

This is the ideal situation, but sometimes the fracture mechanism is more complex. Therefore, an additional procedure is suggested in this research: examining the fracture pattern underneath the microscope before collecting the fragments. The expectation is this will give more insight into the crack propagation and failure process.

II. Experimental

The strength values of the glass in this research were determined by a Ring-on-Ring (RoR) test. It was chosen because the fracture stress is not influenced by the flaw orientation and because it introduces a large area in the glass under constant bending stress. This stress can be calculated by the formula below (Morrel, 1998). Also see Figure 5.

$$\sigma_b = \frac{3P}{2\pi h^2}\{(1-\nu)\frac{a^2-b^2}{2R^2} + (1+\nu)ln\frac{a}{b}\} \qquad (1)$$

The dimensions used are a = 45 mm, b = 20 mm, R = 141 mm, h = 3.9 mm, ν = 0.22. P is the load at failure. The dimensions are based on recommended ratios (Fessler and Fricker, 1984, Morrel, 1998). 52 soda lime silica glass plates were RoR tested in an Instron 8874 loading device. The specimen dimensions are shown in Figure 6. The cruciform shape was chosen to be able to introduce compressive stresses in a later stage of the total research. The specimens were taped with transparent foil, so that the fracture pattern could be studied after failure. Friction is known to be an issue in RoR tests. Various precautions were tried to minimise this. A full description and evaluation of them can be found in this research's corresponding thesis (van der Velde, in preparation).

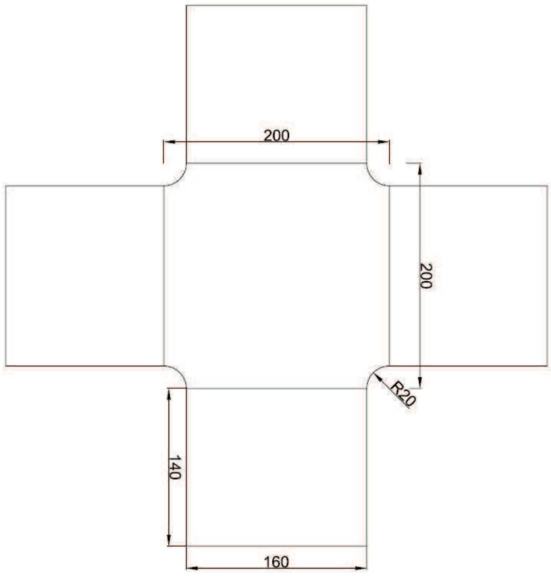


Figure 6: Dimensions of the glass specimen

After the RoR test, six of the fractured plates were examined by a Leica DM2500P petrographic microscope equipped with polarization accessories and semi-apochromat (fluorite) objectives. Optical photomicrographs of the glass specimens were acquired with a Leica DFC310FX digital camera at 1392 × 1040 uninterpolated resolution. In order to overcome the limited *depth of field* of the compound microscope, images were acquired through Leica LAS Live Z-Builder software which enabled dynamic emergence of the single in-focus image. The resulting image has a significantly large depth-of-field which renders the microstructural features of a glass specimen on a two dimensional final image. The advantage of this technique is that it makes all flaws and fracture markings that exist throughout the depth of the glass visible in a single field of view.
Two light settings were used:

- Transmitted light: polarised light enters the specimen from underneath illuminating all flaws inside the glass,
- Reflected light: the specimen is lighted from above, mainly illuminating the surface.

Unless mentioned otherwise, the transmitted light setting was used. All specimens were observed with their tensile side up.

III. Results

As is the case in most strength tests on glass, the results were quite variable, with strength values ranging from 42 MPa to 143 MPa. What was notable is that there was a difference in fracture pattern among the results. Some plates had the expected pattern: fracture origin inside the loading ring (category I) or at or just outside the loading ring (category R). Some of these were accompanied by a circumferential cracking pattern at the loading ring, as is reported in literature (**?**), see Figure 7. However some also showed such a pattern at the support ring, which is not mentioned in literature (noted by an additional S). This might have two explanations: either the cracking pattern is created by a punching shear mechanism caused by friction between the glass and rings or the pattern is created by shock waves brought along by unstable crack growth interacting with the branching cracks. Fractographical analysis might conclude this. Lastly, a few plates had a line-shaped fracture origin which is an indicator for a line-shaped flaw, such as a scratch on the surface (noted by an additional L). The plates that were examined with the microscope are depicted in the pictures on the next pages. The white circles mark the contact of the loading and support ring. The red arrows mark the likely origin crack. TT3, TT40 and TT10 were chosen because they had the smallest failure stress: 41.79 MPa, 56.16 MPa and 57.59 MPa, respectively. This is thought to be the case due to a line defect. Microscopical examination might prove this. The other three plates form a good representation of the fracture pattern categories.

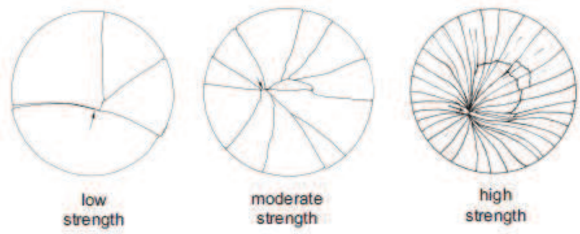
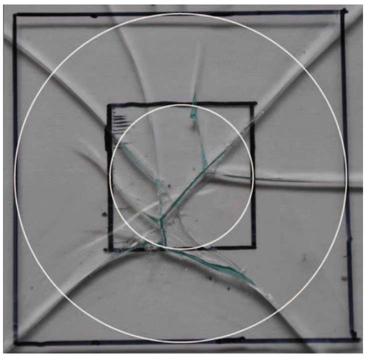


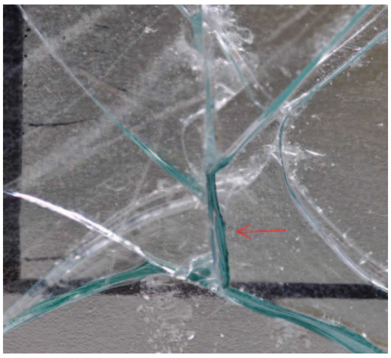
Figure 7: At high failure stress the pattern can include circumferential cracking at the loading ring (Quinn, 2007)

Fractographical analysis

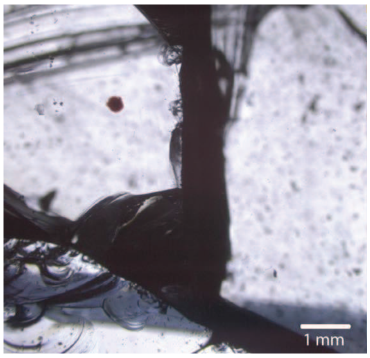
An analysis of each plate is made.
TT3: At this magnification of the likely origin nothing much is seen. However looking at the left bottom, a lot of fracture marks are seen, see Figure 8c and 8d. Quinn states that the greater the failure stress at fracture, the more the stored energy and the richer the fracture marks (Quinn, 2007). Therefore it is concluded that on this line the actual origin point lies. Apparently the branching of the cracks was interpreted wrongly. In Figure 8e several origin like marks are observed.



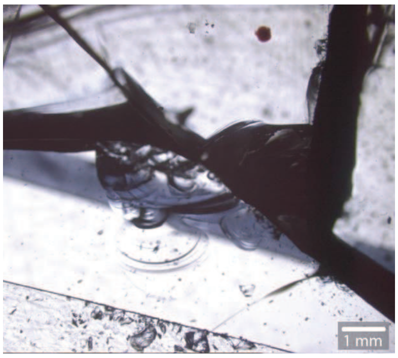
a) Fracture pattern, category IL



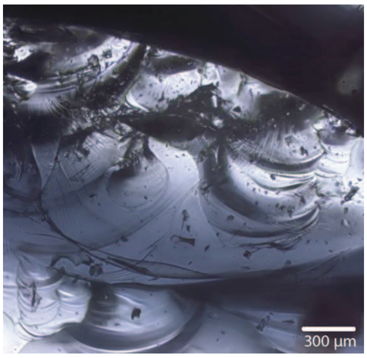
b) Likely origin point



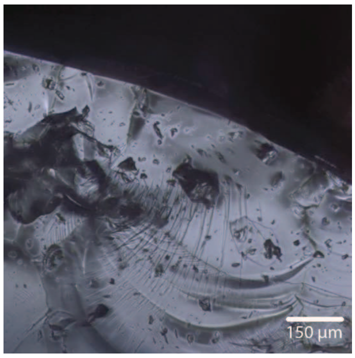
c) magnification



d) magnification

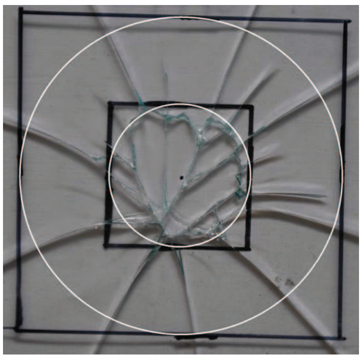


e) magnification



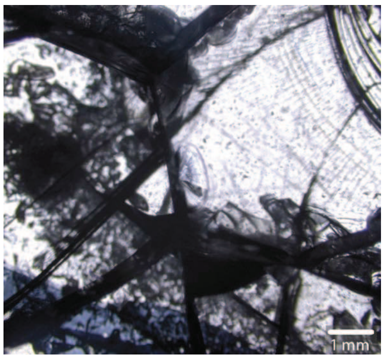
f) magnification

Figure 8: Fractographical analysis of TT3



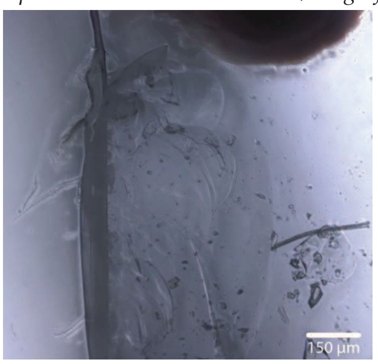
a) Fracture pattern, category IL

b) Likely origin point



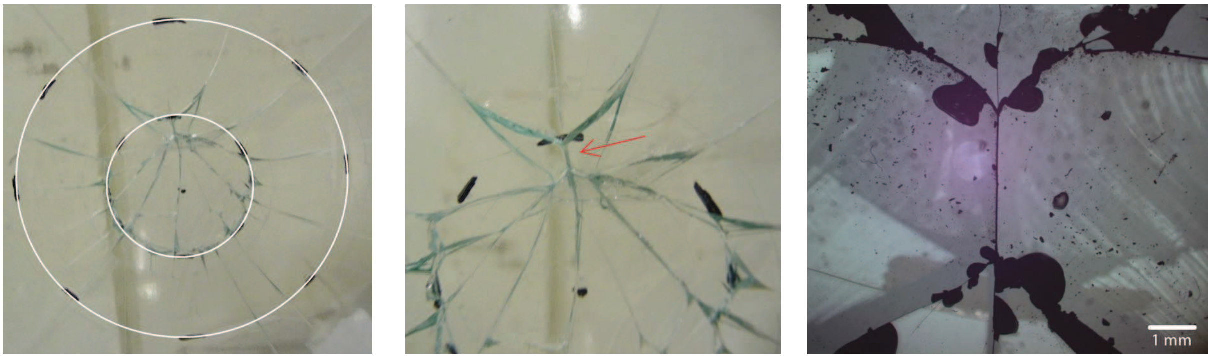
c) magnification

d) magnification

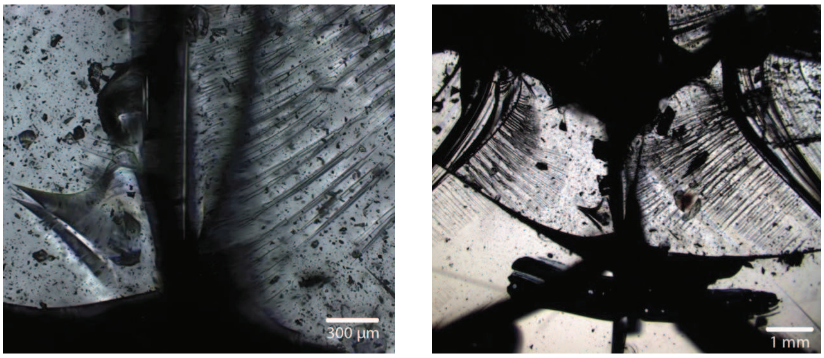


e) magnification

Figure 9: Fractographical analysis of TT10



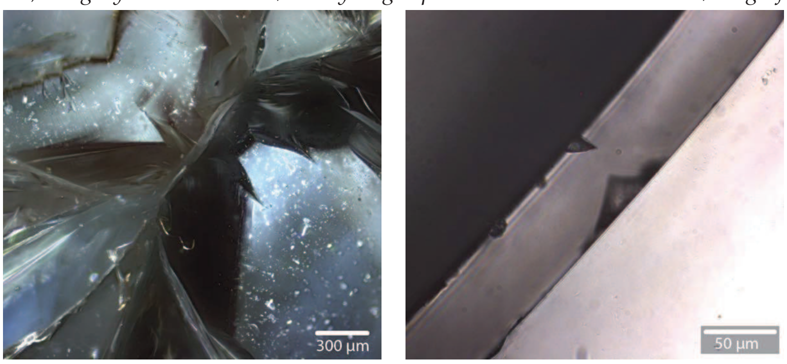
a) Fracture pattern, category IL *b) Likely origin point* *c) magnification*



d) magnification *e) magnification*

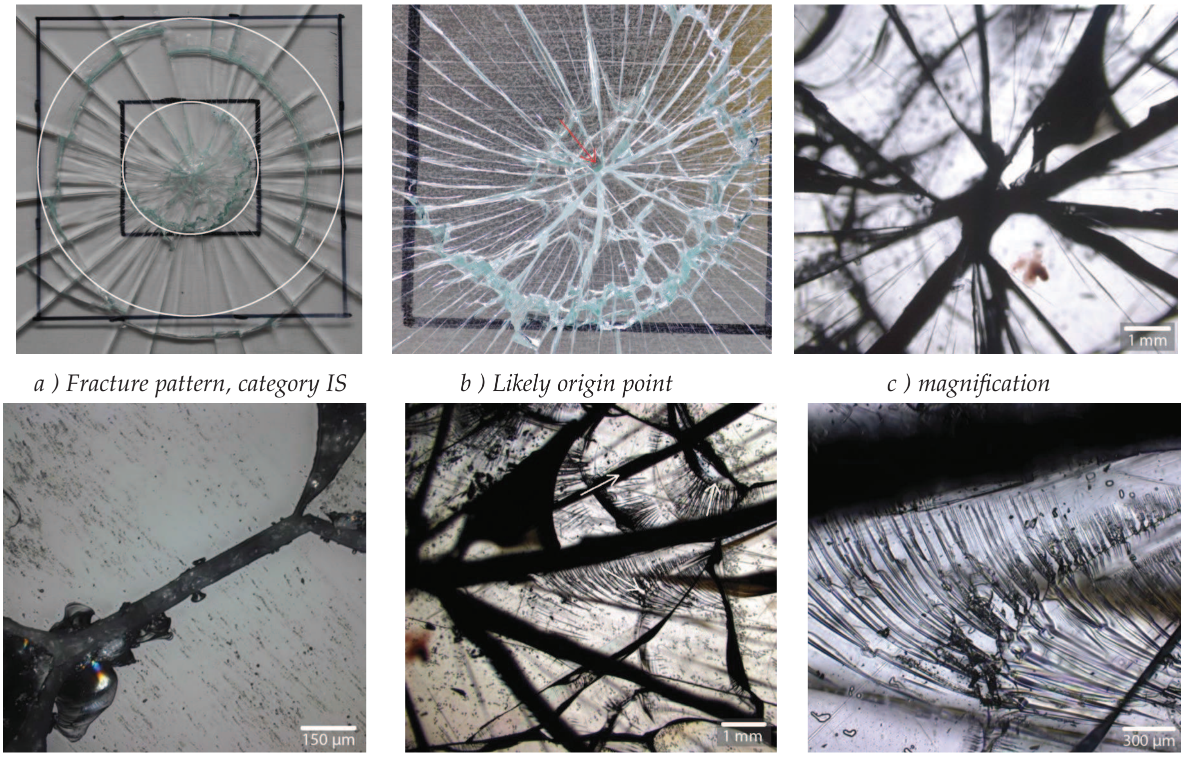
Figure 10: Fractographical analysis of TT40

a) Fracture pattern, category I *b) Likely origin point* *c) magnification*



d) magnification *e) magnification*

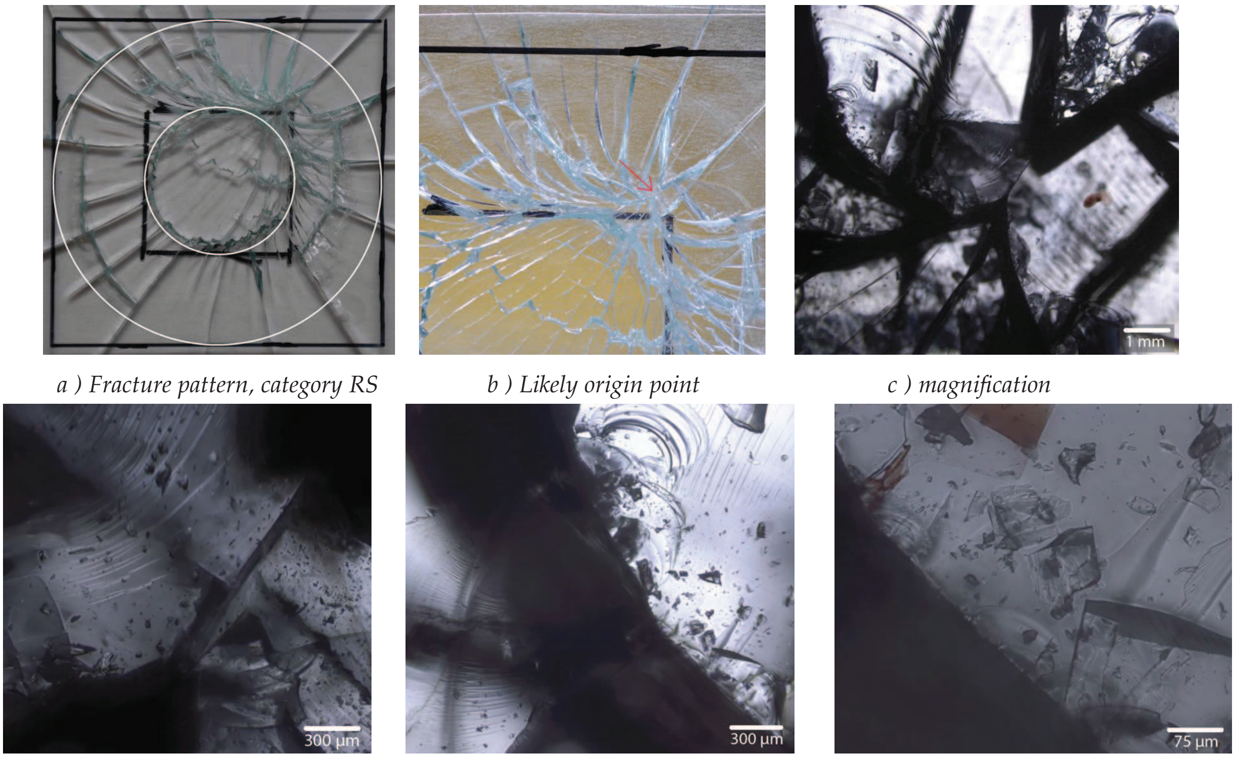
Figure 11: Fractographical analysis of TT6



a) Fracture pattern, category IS   b) Likely origin point   c) magnification

d) magnification   e) magnification   f) magnification

Figure 12: Fractographical analysis of TT8



a) Fracture pattern, category RS   b) Likely origin point   c) magnification

d) magnification   e) magnification   f) magnification

Figure 13: Fractographical analysis of TT16

Which is the actual first one cannot be said from this image, but it does show multiple cracking processes followed up on one another. The last figure zooms in on one of them.

TT10: This specimen gave quite a straightforward analysis. At the first crack a clear shell-shaped mark can be seen in Figure 9c. In Figure 9d stable crack growth is observed. Interestingly at the arrow a point where unstable crack growth commences is marked. Figure 9e zooms in at a magnification at the centre of shell shape where the cracking process started. Analysis of the fracture surface will have to conclude whether it is the actual point of origin.
TT40: In Figure 10c, made with reflected light instead of transmitted light, the crack branching is clearly seen which suggests this is the origin crack. In transmitted light in Figure 10d no starting point can be marked, but it could be blocked from view by the shadows. Looking at Figure 10e in transmitted light something interesting is going on. Twist hackle marks are observed at the sides, both with their propagation direction (marked by arrows) towards the origin crack. This is special because mostly crack propagation runs away from the origin point. Whether this is a coincidence or the origin point is actually located somewhere else cannot be concluded from this analysis alone.
TT6: The location of the fracture origin is clearly visible in this specimen. However, under the microscope so much light was coming through a gap at the crack of interest that the image was too bright. Therefore Figure 11c was taken in crossed polarised light. Despite giving a spectacular picture, in this light it is harder to distinguish the fracture marks, so it is less suitable to draw conclusions about the fracture pattern. On a sidenote, crossed polarised light can be useful in determining the composition of a material's components.
TT8: In this specimen the transmitted light was blocked at the crack of interest. Therefore Figure 12d was taken with reflected light only. Especially to the left, crack growth marks are observed. The origin of fracture might be found there. Figure 12e is taken to the right from the origin crack (the red dot can be used as a reference). Here twist hackle is captured that gives an indication of the ongoing crack propagation
TT16: The origin crack seemed to have been found but under the microscope hardly any fracture marks were observed, nor in Figure 13d. Looking back at Figure 13c a lot more marks are observed to the left, which are shown in Figure 13e. Interestingly, stable crack growth is seen in the form of beach marks but they seem to be taken over by unstable crack growth in the form of hackle. At the centre of this hackle, next to the crack more stable crack growth marks were captured in Figure 13f. The fracture may have originated there instead.

IV. Discussion

Firstly it should be stressed that these are all 2D images of features that are located at different depths in the glass. While interpreting them this should be borne in mind to make sure the right conclusions are drawn. This can be aided by making photographs of just the glass surface before building the 3D images, so that it can be checked whether a certain feature is at the surface or inside the glass.

It is the opinion of the authors that this technique works best in combination with fracture surface analysis, but definitely is a valuable addition. For TT3 and TT16 it seems the wrong origin crack was concluded from the visual fracture pattern analysis. In a fracture surface analysis this would have been discovered by following the Wallner lines at the fracture surface to the right origin point. However, by looking at the fracture pattern by transmitted light microscopy this can be seen in one glance and immediately gives a direction at which point to look in the fracture surface analysis. Therefore it is recommended to use these Z-axis scanned images as an overview to contemplate during the fracture surface analysis. For example for TT10 a very clear spot is marked where the fracture origin can probably be found. When having found the fracture origin at the fracture surface the researcher then can use the Z-axis scanned image to gain an understanding of the rest of the cracking process. This process cannot be deduced in one view by examining only the fracture surface. And interpreting the fracture pattern by eye can lead to wrong assumptions. This is nicely seen in the case of TT8 where twist hackle marks the crack propagation at several location in the glass in one image.

The authors recognise that this technique on its own does not give the final conclusion or even the desired images as was the case for TT40 and TT6. It turns out it depends on the position of the shards in the glass whether enough or too much light is let through and a good image can be made. Although it might be possible to improve this by exploring all settings of the polarised microscope.

These results represent a first try out of this technique. Further research of more plates including fracture surface analysis and significant strength data is expected to show the value of transmitted light microscopy even better.

V. Conclusion

A transmitted light microscopy analysis combined with Z-axis scanning software gives the glass researcher an overview of all fracture markings throughout the depth of the glass plate in one image. In combination with the fracture surface analysis this image can be used as a map which guides the researcher in their search of the fracture origin. More importantly, it reveals fracture markings that give new insights into the crack propagation and the local failure process that are overlooked in traditional glass fractography.

Based on the findings of this study, it can be concluded that transmitted light microscopy, together with Z-axis scanning software is a valuable tool for glass fractography and its use needs to be further explored.

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