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Triggering bulk flaws in glass: Uniaxial tensile testing of glass using theta-specimens

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ABSTRACT: Ongoing research at TU Delft focuses on recycling low-quality glass by casting it into volumetric elements, under the assumption that bulk flaws, and thus compatible contamination, have little influence on the volumetric component's strength. However, to validate the structural behavior of volumetric glass with significant bulk flaws , a corresponding uniform tensile testing method is needed that is not governed by the surface quality of glass and thus, can trigger bulk flaws. This research explores the applicability of the Theta-specimen (Durelli *et al.* 1962), as a method for measuring tensile strength of volumetric cast glass, while avoiding the main drawbacks of a direct uniaxial tensile test.

Three theta-sample geometries are evaluated using FEA, and by mechanical testing of 4-5 CNC waterjet cut float glass specimens per geometry. Polarized light is used to visualize the development of stresses within each sample. Post-fracture fractographic analysis is performed to identify the origins of fracture, and to estimate failure stresses based of the fracture mirror radius using Orr's formula.

The photo-elastic patterns closely match the FEA prediction, confirming that the largest tensile stresses occur within the intended central test strip. Polarized light reveals a sensitivity to eccentric loading for the two newly proposed designs, which can be minimized by introducing a neoprene interlayer between the sample and testing machine. All samples failed at multiple points; stress estimations based on fracture mirror size indicate that the lowest failure stresses consistently occurred within the test strip, further confirming that failure initiated within this area. It is concluded that the Theta-sample has potential as a uni-axial tensile testing method for brittle materials such as glass, though further research is required to fully confirm its reliability.

1 INTRODUCTION

Glass casting enables the manufacturing of volumetric glass elements for use in the built environment (Oikonomopoulou *et al.*, 2022; Oikonomopoulou *et al.*, 2018). Through casting, glass can be elevated from a two- to a three-dimensional material, while allowing full use of the glass' high compressive strength (Oikonomopoulou *et al.*, 2017). More recently, glass casting is being investigated as a method for recycling waste glasses under the assumption that flaws in the bulk of glass have limited effect to its strength. In specific, previous experimental work by TU Delft suggests that flaws restrained in the bulk of volumetric cast glass components, have little influence on its strength, allowing in turn, cast volumetric glass products to tolerate higher contamination compared to thin-walled glass products without a significant compromise to their properties (Bristogianni and Oikonomopoulou, 2023; Bristogianni *et al.*, 2018; Bristogianni *et al.*, 2019; Bristogianni *et al.*, 2020; van Minkelen, 2024; Yu *et al.*, 2020)

Subsequently, cast glass manufactured from waste glass is characterized by a great number of flaws located within the material bulk (Bristogianni and Oikonomopoulou, 2023). Thus, to validate the influence of the bulk flaws on the glass's strength, a suitable testing method is needed that can trigger these flaws by subjecting the entire cross-section of a specimen to a uniform tensile stress . Nonetheless, current testing methods for glass, such as the three- and four point bending and ring-on-ring tests, are flexural tests which primarily test the behavior of the material surface. These tests are valid for homogeneous, flat glass, where failure almost exclusively originates from surface flaws (Shelby, 2005).

The most widely used test for applying a uniform tensile stress is a direct uniaxial tensile test. For brittle materials this method poses some significant drawbacks, notably a substantial risk of contact failure at the tensile grips; and a high sensitivity to unintended bending moments caused by eccentric loading (Christ and Swanson, 1976; Munz and Fett, 1999).

An alternative testing method that could evade the aforementioned drawbacks is the Theta specimen, first introduced by (Durelli *et al.*, 1962). It is an indirect tensile test, where the specimen, of a shape similar to the Greek letter Θ, is tested under direct compression, generating a uniform tensile stress at its central gauge zone. It avoids the complex clamping

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mechanism associated with a traditional uniaxial tensile test; essentially moving the complexity from the testing machine to the sample itself.

This paper aims to explore the validity of the Theta test for cast glass, by conducting preliminary testing in waterjet-cut float glass specimens of three different shape variables, in order to gain insights and propose modifications for improving the reliability of the method for cast glass samples.

2 BACKGROUND

2.1 *Theta testing by Durelli* et al. *(1962)*

The Theta test was first proposed by Durelli *et al.* in 1962. They tested around 70 designs, studying their behavior through the photo-elastic effect, before settling on a single design (Figure 1). Based on fringe counting in polarized light tests, the empirical relation for stress and strain in the gauge section was formulated:

$$\sigma = K\frac{P}{Dt} = 13.8\frac{P}{Dt} \qquad (1)$$

where P is the applied load, D is the outer diameter of the sample, and t its thickness. A dimensionless scaling factor K of 13.8 is used, which is determined by the relation between sample diameter and gauge section height. Strain in the gauge section is described with the following relation:

$$\varepsilon = -0.585\,\frac{\delta_v}{D} = 1.293\,\frac{\delta_h}{D} \qquad (2)$$

where δ_v and δ_h are the changes in length in the vertical and horizontal direction respectively.

The calculation is unaffected by the Elastic Modulus and Poisson ratio. It is based on the assumption that (i) the material is isotropic and homogeneous; (ii) the deformation at failure is relatively small; and (iii) the material exhibits a linear stress-strain relationship. These assumptions can be considered valid for brittle materials such as glass.

All tested samples failed near simultaneously in multiple locations. For two samples the moment of failure was recorded using a camera triggered by the breaking of a conductive strip painted on the sample. For one sample this showed a clear initial failure in the gauge section; for the other sample the initial failure could not be identified. Combined with the polarized light analysis showing maximum tensile stresses in the gauge section, this led to the conclusion that failure did originate in this section as intended.

2.2 *Theta testing by NIST*

The Theta-specimen was further investigated at the National Institute of Standards and Technology (NIST) for the testing of elements used in micro- and nano-electromechanical systems (Fuller *et al.*, 2007; Gaither *et al.*, 2011; Quinn, 2009; Quinn *et al.*, 2005). Samples with a 300 μm diameter were produced from single crystal silicon wafers using deep reactive ion etching (DRIE) (Quinn, 2009; Quinn *et al.*, 2005). Both the original round geometry, and a hexagonal variation were tested. The original geometry was modified by the addition of a flat base to simplify manufacturing and testing at this scale. The failure strength was determined analytically using formula (1), and through FEM. The resulting strength was found to be on the low end of the range commonly reported for miniature single crystal silicon structures, likely due to surface damage left by the DRIE manufacturing process which functioned as crack initiators (Quinn *et al.*, 2005).

A point of attention in the theta-specimen is the intensity of the highest secondary tensile stress outside of the gauge section, as this can result in an invalid test due to fracture originating outside the central gauge section. This can be expressed in the stress ratio R_s = σ\`/ σ, where σ is the gauge section stress, and σ’ the highest stress outside the gauge section (Fuller *et al.*, 2007). For the original design, (Fuller *et al.*, 2007) reported a R_s of approximately 1.00, which implies a risk of failure outside of the gauge section. Several modifications to the geometry have been proposed to reduce the secondary stress, such as the addition of an upper loading tab (Fuller *et al.*, 2007; Gaither *et al.*, 2011), or using a modified geometry with arch-shaped voids (Gaither *et al.*, 2011) (Figure 1).

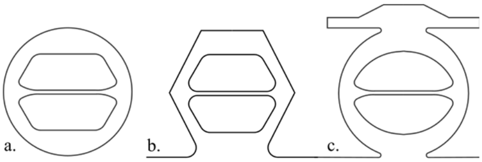

Figure 1. Evolution of Theta-specimen design. A. Durelli *et al.* (1962); b. Quinn *et al.* (2005); c. Gaither *et al.* (2011).

3 METHODOLOGY

The goal of this research is to develop a variation on the theta-specimen that is suitable for testing volumetric glass with bulk flaws. A new geometry is proposed with the aim of minimizing the secondary stresses outside of the central gauge section, i.e. to minimize stress ratio R_s, while increasing the volume of the gauge section. 3 geometries have been tested: the round geometry as proposed by (Durelli *et al.*, 1962) (referred to as RD), and two new square theta designs, with an outer edge with a width of 15 and 20 mm respectively (referred to as

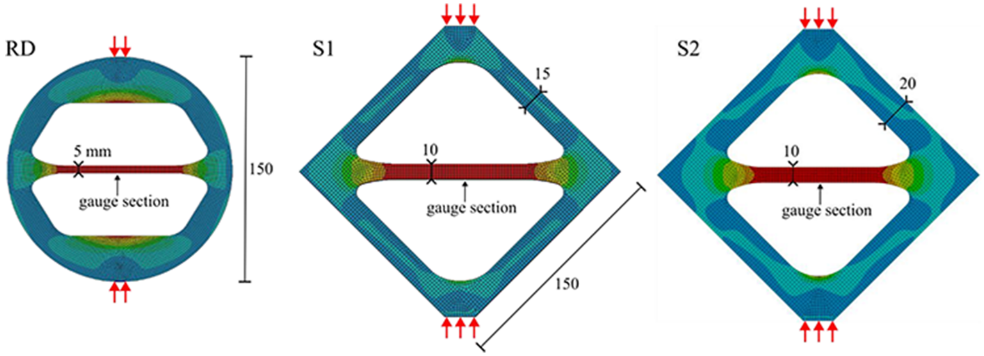

Figure 2. Geometries explored in this research, with basic dimensions and numerically-derived maximum principles stress intensities.

S1 and S2) (Figure 2). The loaded volume in the gauge section is 3750 mm^2 for RD, 8300 mm^2 for S1 and 7270 mm^2 for S2.

All 3 designs are numerically analyzed in Ansys workbench 2024. Geometry RD is modelled with loads and supports over a width of 10 mm, representing a soft interlayer placed between the samples and testing machine. Geometries S1 and S2 are tested with a uniformly distributed load at the top and bottom flat section (Figure 2).

Mechanical tests are performed on soda-lime float-glass samples, cut via a CNC-controlled waterjet cutter. For each geometry, 5 samples are cut from 10 mm thick glass; some of these were rejected due to excessive cutting damage, resulting in 13 usable samples (4 or 5 per geometry). Due to the coarse finish of the waterjet cutter, the glass surface in contact with the testing machine is manually grinded to ensure a good contact. Four additional samples of design S2 are cut from 12 mm glass and are tested first to validate the safety of the setup, and check for out of plane buckling.

All specimens are tested in a 10kN Zwick Z10 displacement-controlled universal testing machine, at a displacement rate of 1.0 mm/min. The specimens are wrapped on both flat sides with a thin self-adhesive foil to retain their cohesion after failure, preventing secondary fracture due to impact with the testing machine and simplifying post-fracture examination. Transient internal stresses are visualized through the photo-elastic effect by using a white LCD screen and a polarizing camera filter . All tests are recorded in a 240 fps video.

After failure, fractographic analysis is performed using a Keyence VHX 7000 digital microscope to identify the fracture origins and the radius of the fracture mirrors. The mirror radius is used to estimate the stress at the moment of fracture, using Orr's formula (Orr, 1972): $\sigma\sqrt{R} = A$, where R is the mirror radius, and A, a material-specific mirror constant. The inner mirror radius R_i is used, with a mirror-mist constant of 1.92 for soda-lime (Wachtman *et al.*, 2009).

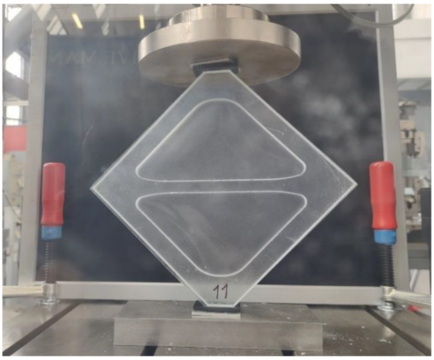

Figure 3. Overview of test setup.

4 RESULTS

4.1 *Numerical analysis*

For geometry RD, the stress in the gauge section was found to be uniform and scaling linearly. An R_s of 1.030 was found, in agreement with (Fuller *et al.*, 2007). S1 was found to have an R_s of 0.61, while for S2, an R_s of 0.75 was found, a reduction of 41% and 27% respectively.

The stress and strain found using FEA for geometry RD were compared with values obtained from formulas (1) and (2). The strain derived using FEA was found to be 3.0% lower than calculated analytically using the vertical deformation. For the horizontal deformation, the deviation was less than 0.1%. The stress value within the gauge section found by FEA was found to be 14% higher than the one derived from formula (1), resulting in K=15.7. This could indicate an imprecision within the fringe counting method employed by Durelli.

4.2 *Mechanical testing on waterjet-cut specimens*

Four preliminary tests were conducted using square samples cut from 12 mm glass. Aluminium strips were placed as an interlayer between the samples and the testing machine to prevent peak stresses due to the direct contact of the two hard materials. Upon loading, these samples displayed significant eccentric loading effects, with a strong diagonal stress distribution in the gauge section (Figure 4, top). This is a sign of the top and bottom loading surfaces not being parallel, or the samples being tilted in relation to the testing machine, resulting in asymmetric loading. This was reproduced in the numerical calculation by limiting the load and support to half of the contact surface (Figure 4, bottom). For subsequent tests, a soft neoprene interlayer of 2 mm was used between the specimens and the testing machine. This significantly reduced the visible asymmetry, though for some samples some asymmetry was still visible.

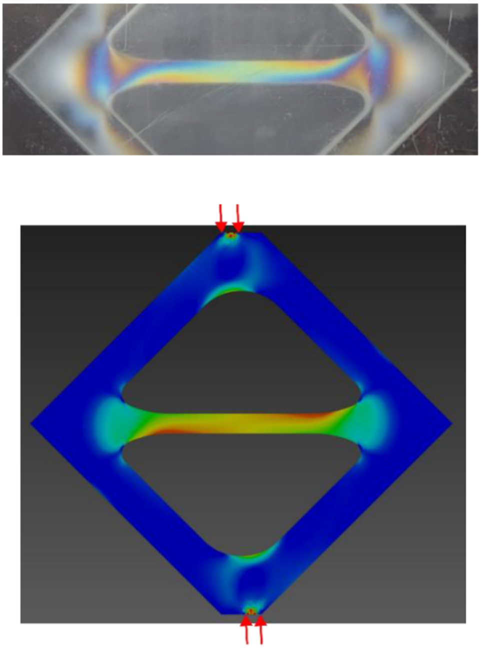

Figure 4. Stress asymmetry visible through photoelasticity (top) and recreated in FEA (bottom).

Upon fracture, all samples broke into multiple fragments. The video recordings failed to directly capture the origin of failure. Between frames, cracks appeared both in the gauge section, and at least in one of the outer edges. Fractographic analysis revealed that the largest fracture mirrors, and consequently the lowest failure stresses, were consistently located within the gauge section. The only exception is one of the round samples, where the lowest failure stress was found in the top section of the outer ring (Figure 5).

In the majority of samples, failure originated at the waterjet-cut surfaces, while 2 samples broke due to edge failure. No cases of surface failure from the air or tin sides of the glass were found.

The failure stress in the gauge section for each sample was determined through FEA, based on the measured failure load. The results are given in Table 1.

Table 1. Overview of test results.

Geometry		Thickness	F_{break}	$\sigma_{gauge,FEA}$	
#	name	mm	N	MPa	
1-4	S2 (calibration)	12	-	-	
5	RD	10	3144	32.9	Mean: 34.1 MPa
6	RD	10	3269	34.2	CV: 0.04
7	RD	10	3192	33.4	
8	RD	10	3320	36.0	
9	S1	10	3069	26.5	Mean: 29.8 MPa
10	S1	10	3471	30.0	CV: 0.065
11	S1	10	3460	29.9	
12	S1	10	3549	30.7	
13	S1	10	3659	31.7	
14	S2	10	4116	30.0	Mean: 29.6 MPa
15	S2	10	4238	30.9	CV: 0.036
16	S2	10	3913	28.6	
17	S2	10	3969	29.0	

5 DISCUSSION

5.1 *Reliability of tests*

In principle, the location and direction of failure in the outer edges of the specimens coincide with the maximum tensile stresses due to bending in the sample without the central strip (**Error! Reference source not found.**). This is supported by the failure stresses estimated through fracture mirror measurements, which show that the lowest stresses consistently occurred within the gauge section of the samples. This suggests that initial failure occurs in the central strip. Fractures in the outer edges are secondary: the sudden redistribution of forces leads to large bending moments and high associated tensile stresses.

The location of failure within the gauge section varies. Out of the 13 samples with rectangular geometry, 7 broke at the very end of the gauge section, at the point where the edge transitions from straight to curved. This is a sign of the presence of unintended stress concentrations, caused by an excessively sharp curve, asymmetric loading, or a combination of the two. The first four tests, performed without a soft interlayer, displayed strong asymmetric strains and consistently failed near or at the end of the gauge section. However, sample 14 also displayed strong asymmetric loading, yet failed some distance away from the end of the gauge section. Failure at the end of the gauge section is therefore not always a valid indicator of asymmetric loading.

Only in one round sample was the largest fracture mirror, and consequently, the lowest failure stress, found outside of the central gauge section (Figure 4, right). This sample shows a crack in the middle of the top section of the outer edge, with the crack running outwards. This matches the location of the highest tensile stress outside of the gauge section identified by the numerical modelling for this geometry. This indicates an invalid failure, originating outside of the gauge section.

5.2 *Comparison of geometries*

Little difference was found between designs S1 and S2. Both designs displayed similar failure behavior with the smallest fracture mirrors occurring within the gauge section. FEA shows lower secondary stresses for SD1 ($R_s = 0.61$, compared to 0.75 for SD2), while both performed better than the original geometry RD ($R_s = 1.03$). Failure stresses in the gauge section were comparable for both SD1 and SD2, with SD2 showing a lower spread. These measurements cannot be considered statistically significant due to the small sample size; further testing is required for verifying this.

5.3 *Recommendations*

To improve the reliability of the theta specimen, the main priority would be to reduce the risk of eccentric loading. This can be achieved through:

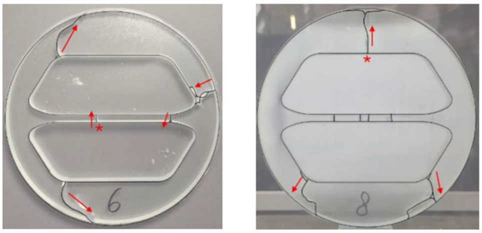

Figure 5. Failure of RD samples, with direction of crack propagation indicated with arrows, and the location of the largest fracture mirror indicated with an asterisk (*). Left: fracture pattern after initial failure in gauge section. Right: Likely invalid initial failure in upper edge of sample.

- more precise post-processing of the contact surfaces to ensure these are fully parallel to each other and the gauge section;
- replacing the flat contact surfaces with curved surfaces, to avoid the need for fully parallel contact surfaces. Alignment of the sample can instead be achieved using a 45° angle ruler.

If regular failure at the ends of the gauge section persists when eccentricity is minimized, the blending curve towards the gauge section should be revised to be more gradual.

The goal of this research was to explore the theta specimen as a testing method for volumetric cast glass. Transferring this testing method from waterjet-cut float glass to volumetric cast glass has several consequences. Glass casting offers a large freedom of shape, which means that theta-specimens could be cast directly. Representative results could be obtained by using a mold similar to the one used for the final component, and casting the specimens simultaneously to the final component.

When used for glass cast from waste material, more severe flaws can be expected, originating from contaminations within the cullet (Bristogianni and Oikonomopoulou, 2023). When using a theta specimen, this brings with it a risk of invalid failure caused by a severe flaw located outside of the gauge section.

Several approaches can be considered to further adapt the theta geometry for the testing of cast glass with notable bulk flaws:

- optimization of the sample geometry to further reduce stresses outside the gauge section;
- a methodology for verifying the initial failure location that is reliable and simple to perform;
- reducing the presence of flaws outside the gauge section, for example by using kiln-casting to place the glass to be tested in the gauge section only, while using glass of higher purity in the remainder of the sample.

Further investigation is needed for exploring these approaches.

6 CONCLUSION

The new square theta design is promising for performing uniaxial tensile tests on brittle solids. Compared to the original geometry proposed by Durelli *et al.*, the geometries SD1 and SD2 subject a larger volume to tension, while reducing the maximum principal stresses outside of the gauge section by 41% and 27% respectively. Though the video recording employed failed to capture the initial failure location, post-fracture analysis suggests consistent failure within the intended gauge section. The current setup is revealed to being sensitive to eccentric loading, resulting in unintended, skewed stress gradients within the gauge section. This is likely caused by the two flat loading surfaces not being fully parallel due to fabrication tolerances, the samples being placed within the testing device at a slight angle, or a combination of these two factors.

Further improvements to the square theta design need to be made to reducing the risk of eccentric loading, for example by replacing the flat contact surfaces with rounded surfaces, and taking additional precautions to ensure proper alignment to the testing machine.

Translating the theta specimen from homogeneous float glass to glass cast from waste material is expected to increase the risk of invalid failure, due to the presence of severe flaws that might occur outside the gauge section. Several approaches to adress this are proposed; further research is required to explore these.

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