APPLICABILITY OF THE CHEVRON-NOTCH TECHNIQUE FOR FRACTURE TOUGHNESS DETERMINATION IN GLASS

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ABSTRACT: *The applicability of the chevron-notch (CN) three-point bend test for determining the fracture toughness (K_{Ic}) of borosilicate glass was studied. As-received specimens with straight sharp notch were also tested in three-point bending for comparison purposes. It was found that both chevron and straight notched specimen geometries were equally suitable for the fracture toughness determination in terms of average values and data variability according to Weibull and Student's t statistics. Testing with CN geometry showed that annealing of specimens at a temperature (550^{o}C) higher than the glass transformation temperature led to a lower value of K_{Ic}. The difference in mean fracture toughness values (0.75 and 0.64 MPam$^{1/2}$ before and after annealing, respectively), was ascribed to the different level of compressive residual stresses in the samples.*

INTRODUCTION

For several load-bearing and other critical applications, it is very important to be able to quantify the fracture behaviour of glass. In particular, the exact determination of the fracture toughness (K_{Ic}) of glass products is required in order to discern subtle effects of processing or in-service conditions, including thermal and environmental factors, on their fracture resistance. For this purpose, an accurate and sensitive testing method is necessary due to the low fracture toughness of glass. Several methods have been developed to determine fracture toughness of brittle materials and their relative advantages and disadvantages have been discussed.

The chevron notched (CN) specimen technique is a well-established method used to determine the fracture toughness and the work of fracture of brittle materials, including polycrystalline ceramics [1-7], monolithic glass [2,3,8-10], glass-ceramics [3] and particle and fibre-reinforced glass and ceramic matrix composites [12]. Reported advantages of the CN bend test are: simplicity of loading under difficult conditions, for example at elevated temperatures or in reactive environments, reproducibility of results and small amount of material required per specimen [9]. In chevron notches, the crack front increases in width from zero to the full thickness of the specimen as the crack length increases in the notch plane. It is assumed that stable crack growth occurs from the apex of the chevron during loading. There is, therefore, no need to precrack the specimen, which is a time consuming and tedious task; this is a further advantage of the CN geometry.

Despite these advantages, several authors have shown that the presence of a chevron notch in a bend specimen does not guarantee *per se* the formation of a stable growing crack during loading. In particular, unstable crack propagation in CN vitreous silica [9] and soda-borosilicate glass [3] specimens has been observed.

Considering the contradicting results reported in the literature the purpose of the contribution is twofold. Firstly, to analyse relative suitability of the chevron notch technique and the more frequently used straight notch method for the fracture toughness determination of glass. Secondly, to use the results of CN bend test for assessing any fracture toughness changes that occur due to annealing and ageing of borosilicate glass specimens.

EXPERIMENTAL METHODS

The material investigated was borosilicate (DURAN®) glass. Nominal properties of the glass are given in Table 1. The samples, fabricated by Schott Glaswerke (Mainz, Germany), were in the form of rectangular test bars of dimensions 4.5x3.8x100 mm^3. Selected samples were subjected to annealing heat-treatment at 550 ºC for 20 hours. Another group of samples was tested after having been stored for three years at room temperature in a desiccator. These samples will be referred to as “aged” samples.

TABLE 1: Properties of the borosilicate glass investigated [11]

Glass type	Density [g/cm^3]	Young’s modulus [GPa]	Poisson’s ratio	Thermal exp. coeff. [$°C^{-1}$]	Flexural strength [MPa]
DURAN®	2.23	63	0.22	$3.25 \cdot 10^{-6}$	~ 60

Chevron notches were cut using a thin diamond wheel. The specimens were loaded in three-point bending (span of 16 mm) at a constant cross-head speed of 0.1 mm/min at room temperature. Load - deflection traces were recorded and the fracture toughness was calculated from the maximum load (F_{max}) and the corresponding minimum value of geometrical compliance function (Y^*_{min}) using the equation [17]:

$$K_{Ic} = \frac{F_{\max}}{BW^{1/2}} Y^*_{\min} \tag{1}$$

where B and W are the width and thickness of the specimens, respectively. The calculation procedure used for the purposes of this investigation has been described in detail elsewhere [13]. The CN depth a_0 was measured from SEM micrographs of fractured specimens.

Additionally, straight-notched (SN) specimens, which are usually recommended as a standard geometry for brittle materials [14], were tested for comparison purposes. Straight notches having width of about 150 μm and depth of about 1.5 mm were cut using a thin diamond wheel. The tip was sharpened by drawing by hand a conventional razor blade coated with diamond paste of size of 0.1 μm through the notch. Other testing conditions were the same as in case of CN specimen technique.

RESULTS AND DISCUSSION

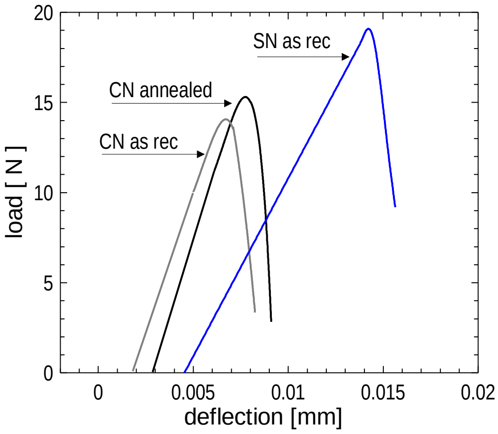
Comparison of Chevron and Straight Notched Specimen Techniques

The load deflection traces for both CN and SN specimen techniques were found to be similar in general form but, as will be discussed, differed in detail. A summary of the primary data obtained is given in Table 2.

Typical examples of load - deflection records are shown in Figure 1. Evidence for the presence of a crack growing in a stable manner during loading is the appearance on the loading curve of a smooth change in specimen compliance. On the other hand, unstable crack growth is characterised by a load-displacement curve that is linear up to the peak load followed by a sudden load drop. This is the case for specimens with straight notches, for which the load increases monotonically to a maximum, and then the crack initiates at the notch root and runs in unstable manner. In contrast, for CN specimens the crack initiates at about 1/2 to 2/3 of maximum applied load, and then runs in stable manner (i.e. the crack driving force is controlled by increasing length of crack tip) until the maximum load is reached. The onset of the stable growth stage is marked by the arrow on the detail of the load - deflection trace in Figure 1b.

TABLE 2: Primary data obtained with both specimen geometries

STRAIGHT NOTCH				CHEVRON NOTCH			
Sample	Fracture load F_{max} [N]	Notch depth [mm]	Fracture toughness K_{Ic} [$MPam^{1/2}$]	Sample	Fracture load F_{max} [N]	Notch depth [mm]	Fracture toughness K_{Ic} [$MPam^{1/2}$]
1	16,50	1,58	0,69	1	9,34	1,39	0,79
2	13,06	1,91	0,67	2	9,85	1,40	0,82
3	13,24	2,08	0,69	3	9,38	0,98	0,59
4	19,03	1,66	0,83	4	13,47	0,93	0,81
5	17,92	1,67	0,79	5	9,99	0,99	0,64
6	17,19	1,62	0,75	6	11,33	0,99	0,73
7	16,86	1,53	0,70	7	12,51	1,02	0,84



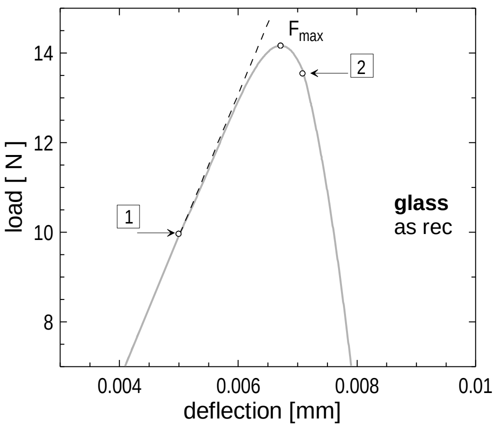
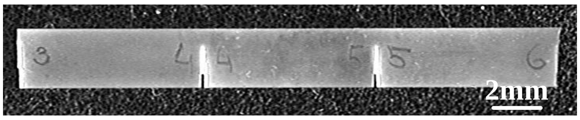


Figure 1: (a) Load-deflection traces obtained using 3-point bending for both chevron notched (CN) and straight notched (SN) specimen geometries; (b) detail of departure from linearity and maximum load point in CN test.

It should be pointed out that fully catastrophic failure in chevron notch specimens did occur in a few cases, leading to specimen destruction. This behaviour was characterised by a discontinuity in the load-deflection curve, i.e. an uncontrolled drop of load, as shown by arrow 2 in Figure 1b. In the present experiments, however, almost all CN specimens were in one piece after the test, as shown in Figure 2.

Fully equivalent fracture behaviour was also found when quantitatively comparing the results of both methods (Table 2). For example, very close mean K_{Ic} values were obtained: 0.75 ± 0.01 $MPam^{1/2}$ for CN specimens and 0.73 ± 0.01 $MPam^{1/2}$ for SN specimens. The Student's - *t* statistical test (*t* = 0.38, n = 7, with n: number of samples tested) demonstrated that the small difference in K_{Ic} values was not significant. Furthermore the standard deviations obtained by both techniques are low, which indicates that the measurements are highly reproducible.



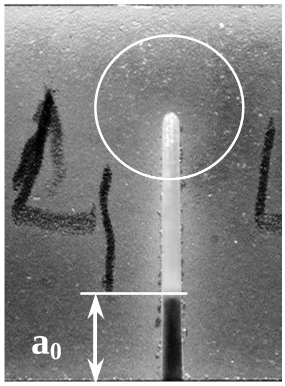


Figure 2: Chevron-notched specimen after the testing showing unbroken specimens 4 and 5 (above) and detail of the chevron notch tip (right).

As shown in Figure 3a, the scatter characteristics, as determined using Weibull statistics, are slightly different; the Weibull modulus being m = 6.0 and m = 9.0 for CN and SN specimens respectively. This difference can be explained by the different shape and size of the highly stressed regions around the notch in both configurations. The larger the volume of specimen that is stressed, the lower the scatter as greater is the likelihood of the presence of a critical flaw leading fracture initiation. The SN geometry stresses a larger volume and hence there is a higher probability for the presence of a fracture initiation site in this volume, compared to the chevron notched specimen; this results in a higher Weibull modulus with SN geometry.

Effect of annealing and ageing on K_{Ic}

Figure 3b shows the statistical variation of the data in the form of Weibull plots. Data for aged samples, which were tested three years after the initial (as-received) tests, are also shown. Th e measured fracture toughness values for the different batches are: (0.75 ± 0.01) $MPam^{1/2}$, (0.74 ± 0.01) $MPam^{1/2}$ and (0.64 ± 0.01) $MPam^{1/2}$ for the as-received, aged and annealed samples, respectively. Thus, the initial average value of K_{Ic} has decreased by about 15% after annealing and by about 1.4% after room temperature aging.

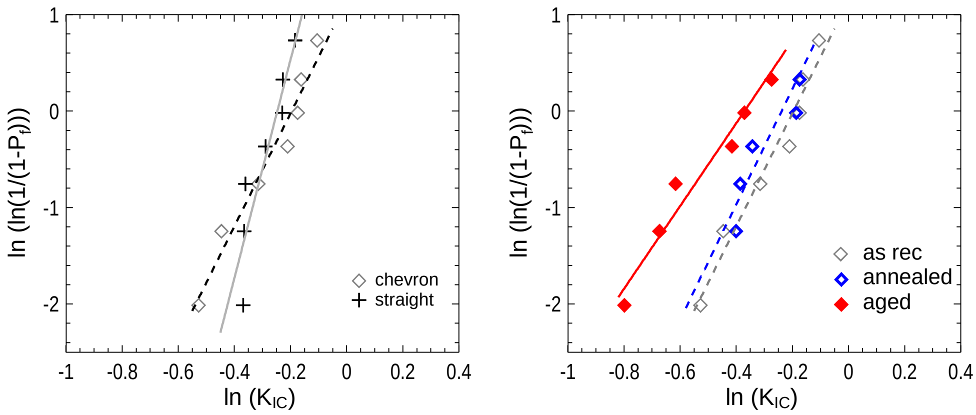


Figure 3: Weibull plot of fracture toughness data obtained from CN and SN specimen techniques (a). Data for as-received, annealed (550 °C, 20 h) and aged (3 years, RT) samples are shown (b).

Applying the Student's *t* statistical method, it was possible to confirm that the difference in measured K_{Ic} values was "highly significant" in statistical terms for the annealed samples. A value $t = 3.53$ was calculated, which, for a population of seven data, is related to a very low probability ($P < 0.01$) of the data belonging to the same population.

In glass with a surface compressive stress field profile, fracture toughness in the non-annealed condition should be higher than after annealing, as found by Reddy et al. [3]. It is thus suggested that the subtle but real (statistical significant) decrease of K_{Ic}, as determined accurately by the chevron notch technique and confirmed by the Student's *t* statistical method, is due to the relief of surface compressive stresses in the annealed borosilicate glass samples. This should have occurred during annealing at 550 °C, which is 25 °C above the glass transition temperature of DURAN® glass [15]. With lower compressive stresses remaining in the sample, lower external loads are required to reach the tensile stress level for crack nucleation, which leads to lower resistance against crack initiation and thus to a decrease of fracture toughness.

Analysis of Figure 3a reveals that the mean fracture toughness for the aged material (0.74 $MPam^{1/2}$) is also lower than that of the samples tested in the as-received state (0.75 $MPam^{1/2}$) although higher than the value for the annealed material. The Student's - *t* statistical test applied on these data demonstrated however that the small difference in average K_{Ic} values was not significant. A true small reduction in K_{Ic} in aged samples may be expected due to aging related stress relief effects. However the statistical consideration of the available data impedes confirmation of these effects in the present experiments. Nevertheless the result of this part of the study indicates that there is a limit for the applicability of the chevron notch technique to detect subtle differences in K_{Ic} of glass: for the relative low number of measurements conducted in this study (7), the test cannot significantly differentiate K_{Ic} differences of about 1.5 %.

It is finally worthwhile to point out that the Weibull plots in Figure 3a seems to support the discussed mechanism for K_{Ic} change after annealing. Measurements made on specimens in the non-annealed condition are characterized by nearly the same value of Weibull modulus (m = 6.0 and m = 5.5 for as-received and aged samples, respectively) reflecting the relatively small change in the stress situation as indicated by the fracture toughness values. The modulus however has a lower (m = 4.2) value for the thermally annealed specimens. It is suggested that this behaviour is due to changes in the fracture initiation condition, which should be different for specimens with and without residual surface compressive stresses. A further investigation on the quantitative effect of the level of residual compressive stresses on K_{Ic} of borosilicate glass, as determined by the chevron-notch technique, is the focus of on-going research.

CONCLUSION

This work has demonstrated that the chevron-notch three-point bending test is a reliable method for accurate determination of the fracture toughness of borosilicate glass. This is manifested by the low standard deviation, and thus reproducibility, of measured values and by their good agreement with data obtained using other techniques. It was also shown that subtle changes in fracture toughness values of glass samples, for example as the result of an annealing heat-treatment above the glass transition temperature, can be assessed accurately by the chevron notched specimen technique. Thus, the advantages of the chevron notch technique, in particular the savings in notch cutting time, easy preparation of the notch (no precracking needed) as well as its suitability for testing under specialized conditions should make the chevron notch test the technique of choice to determine K_{Ic} of glass.

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