

1 **Thermal healing of realistic flaws in glass**

2 Marco Zaccaria¹ and Mauro Overend²

3 ¹BE, MSc, Department of Engineering, University of Cambridge, UK, corresponding
4 author, email mz287@cam.ac.uk

5 ²BE&A, MSc, PhD, CEng MStructE MICE, Senior Lecturer in Building Engineering Design,
6 University of Cambridge, UK, email: mo318@cam.ac.uk

7 **Abstract**

8 For any given environmental conditions the tensile strength of glass is a function of the
9 geometry of the critical flaw and the residual stresses in the vicinity of the flaw. The
10 strength of heat treated glass is conventionally considered to be equal to the sum total
11 of the residual stress and the extrinsic strength of annealed glass. Recent experiments
12 suggest that there is an additional contribution to strength due to crack healing. In
13 order to quantify it, uniaxial and equibiaxial strength tests on both as-received and
14 carefully annealed glass specimens were performed for different edge geometries and
15 edge finishes. The results show that strength recovery due to healing is significant and
16 this strength gain appears to correlate with the quality of the edge finish. Possible
17 explanations of this phenomenon are provided. Independently of healing effects, it was
18 also found that the edge quality has a marginal effect on the mean strength, but has a
19 significant positive effect at low fractile values often used in design applications.

20 **Keywords:** edge strength, surface strength, crack healing, residual stress.

21

22

23

24

25

26 **Introduction**

27 Over the past century glass has been used in increasing volumes in buildings. Its role
28 has diversified: from simply supported panels for windows, to glass façades with ever
29 increasing sizes of glass panels and smaller supported areas. The trend of using glass in
30 a more structural manner extends to other applications such as staircases and roofs.
31 Post-production processes such as tempering and lamination have brought about
32 significant improvements in the performance of glass. However, the fundamental
33 reasons for some of the strength and failure phenomena in glass are not fully
34 understood.

35 Theoretically glass is a very strong material, with an intrinsic (i.e. flawless) tensile
36 strength based on intermolecular forces as high as 32 GPa (Shelby 1997), but this is
37 significantly reduced by stress concentrations at the tip of surface flaws. These flaws,
38 also known as Griffith flaws, are unavoidable consequences of handling, transportation
39 or in-service weathering and are generally found in large numbers on the surface of
40 glass and can be classified as scratches or digs (Fig. 1). When the flaws are subjected to
41 crack opening stresses (aka mode I loading), the stress concentration at the crack tip is
42 described by the stress intensity factor K_I , which is a function of the shape and depth of
43 the flaw. Irwin (1957) defined mode I loading as:

$$44 \quad K_I = Y\sigma \sqrt{\pi a} \quad (1)$$

45 where,

46 Y is the geometry factor accounting for the shape of the crack,

47 σ is the tensile stress normal to the crack,

48 a is the crack depth.

49

50 It is particularly difficult to measure the flaw geometry and size prior to fracture. In fact,
51 the flaw tip tends to be too small or between surfaces in close optical contact that is
52 impossible to identify it from a top view with an optical microscope. Other instruments
53 such as surface profilometers are equally unsuitable as they are unable to penetrate to
54 the depth of the flaw tip. This difficulty is compounded further by the presence of
55 median and lateral cracks (Fig. 2) that extend from the tip of the surface flaw. These
56 cracks are formed when the glass is chipped or scratched, even when this is done by
57 carefully controlled indentation or cutting (Schula and Schneider, 2013).

58 A common way of increasing extrinsic tensile strength of glass is tempering (thermal or
59 chemical). These processes induce a residual stress state of compression in the surface
60 regions of the glass and tension in the core of the glass. The compression on the surface
61 enables the glass to resist tensile stresses at least as high as the residual stress,
62 providing that there are no flaws deeper than the pre-compression layer. The processes
63 of thermal and chemical tempering are not described here for brevity, but can be found
64 in more specific literature (Haldimann et al. 2008, Zijlstra and Burggraaf 1968).

65 Commercially annealed glass is not entirely stress-free, in fact a small degree of residual
66 stress, ranging from 4-11 MPa, has also been reported on as-received (commercially
67 annealed) soda-lime-silica glass from float plants. This residual stress in commercially
68 annealed glass is attributed to the cooling step in the annealing lehr of the float process
69 which is not sufficiently slow to prevent residual stress from forming altogether
70 (Zaccaria and Overend 2012). In this paper the term “annealed glass” is used to describe
71 soda-lime-silica glass that is free of residual stress. The laboratory process performed to
72 achieve this is described in subsequent parts of this paper.

73 Recently it was observed (Nielsen et al. 2010) that the extrinsic strength (f_{FT}) of fully
74 tempered glass (FTG) is not simply the sum total of the extrinsic strength of annealed
75 glass (f_{AN}) and the surface residual stress (σ_{RES}):

$$76 \quad f_{FT} \neq f_{AN} - \sigma_{RES} \quad (2)$$

77 But an additional strength is also recorded, leading to:

$$78 \quad f_{FT} = f_{AN} - \sigma_{RES} + f_{HEAL} \quad (3)$$

79 where f_{HEAL} is a strength gain due to crack healing.

80 A similar additional strength has been recorded (Zaccaria and Overend 2014) for
81 chemically tempered glass (CTG) suggesting that equation (3) could be extended to all
82 glasses that are subjected to a temperature profile of the type used in post-production
83 processes. Equation (3) indicates that the extrinsic strength is governed by the critical
84 flaw, residual stress and healing and is usually obtained from destructive tests, but the
85 contribution from healing is not fully characterised.

86 Crack healing can be defined as a spontaneous process consisting of crack closure
87 associated with a strength recovery. Griffith (1920) postulated that cracking could be a
88 reversible process only in the case of very narrow cracks, i.e. when the two cracked
89 surfaces correspond to one another and there is no debris between them.

90 Several researchers have studied the underlying causes of crack healing. The main
91 parameters investigated are humidity and temperature profile. Healing was measured
92 in terms of the energy required to re-open an artificial crack and in some studies was
93 also observed visually.

94 Crack healing was investigated in humid and inert conditions, noticing that humidity
95 prevents re-bonding by triggering chemical reactions at the flaw tip (Wiederhorn and
96 Townsend 1970). Michalske and Fuller (1985) focused on the effect of controlled levels
97 of humidity ranging from 0.01% to 100% and they also proposed a chemical model of

98 crack healing. The effect of temperature on crack healing was studied by Hrma et al.
99 (1988), who investigated various temperature profiles and concluded that temperature
100 favours healing, but that prolonged heat treatments lead to weakening. In a study by
101 Inagaki et al. (1985) healing was observed visually on notched glass samples under
102 cyclic loading and crack closure was ascribed to a mechanism similar to hysteresis.
103 Crack closure was also visually observed by Girard et al. (2011), who took humidity and
104 heat treatment into account and described healing as a step-by-step process involving
105 relaxation of the stress immediately below the crack tip caused by indentation, crack
106 blunting, followed by crack closure.

107 The existing body of research identifies the main factors that appear to affect crack
108 healing in glass. All of the studies were performed by creating an artificial crack in glass
109 and subsequently measuring the energy required to re-open it. These studies provide
110 very useful information, but the phenomenon merits further investigation, in particular,
111 to quantify:

- 112 1- The extent to which realistic (rather than indented) flaws are affected by crack
113 healing. Realistic (Griffith) flaws would be expected to be more susceptible to
114 healing, due to their size and optical contact, but this has yet to be ascertained;
- 115 2- The true strength gain resulting from thermal crack healing. In fact, any thermal
116 treatment typically produces not only a residual stress (σ_{RES}), which enhances its
117 performance, but also an additional strength due to healing (f_{HEAL});
- 118 3- The effect of thermal crack healing on a realistic flaw population rather than on a
119 single flaw, and the strength increase at lower fractile values (rather than simply
120 on the mean strength) as these values are important in real-world applications.

121 The aim of this paper is to quantify the strength gain of glass due to crack healing
122 (f_{HEAL}) as a result of temperatures encountered during an annealing cycle. In doing so it

123 addresses the three principal gaps in knowledge listed above. This is done by carefully
124 annealing as-received soda-lime-silica float glass. Annealing has the benefit of removing
125 any remaining residual stress in the glass while providing a heating cycle below its
126 transition temperature, thereby leaving the atomic structure unaffected. The annealed
127 glass is subsequently tested to destruction and these results are compared to strength
128 data obtained from as-received glass. To account for different realistic flaw populations,
129 one type of untreated glass surface and three types of industry standard edge finish are
130 tested on a coaxial double ring (CDR) and a 4-point bending (4PB) set-up, respectively.
131 The strength contribution of residual stress (σ_{RES}) is determined by photoelastic
132 measurements with a scattered light polariscope (SCALP) (Anton and Aben 2003).

133

134 **Method**

135 Standard soda-lime-silica glass (SLSG) has been used in this study. Its expected chemical
136 composition and properties are shown in table 1 and table 2, respectively.

137 Four series were investigated (Table 3), each consisting of:

- 138 - 16 as-received float glass;
- 139 - 16 as-received float glass subsequently annealed in the laboratory.

140 The series were tested as follows:

- 141 - Series I coaxial double ring (CDR), size of the specimens 150 x 150 x 6mm;
- 142 - Series II, III, IV four point bending (4PB), size of the specimens 150 x 20 x 6 mm.

143 The three series tested in 4PB differ from one another in terms of edge finish: as cut,
144 chamfered grinded, chamfered polished (Figure 3).

145 Surface pre-compression was measured with a calibrated scattered light polariscope
146 (SCALP 5.0).

147 Specimen edges were investigated before and after annealing by means of an optical
148 microscope to identify any changes in flaw morphology.

149

150 *Coaxial double ring tests*

151 A CDR setup was used to test the surface strength of as-received and annealed glass
152 (Fig. 3). The glass specimens were tested using a universal testing machine with a 30 kN
153 load cell. The diameters of the loading and support rings were 51 mm and 126 mm,
154 respectively. A double hinged connection was placed between the cross-head and the
155 loading ring to ensure uniform contact between the loading ring and the glass. Before
156 testing, a UV-light detector was used to identify the tin side and all CDR specimens were
157 tested with the tin side in tension. A self-adhesive film was applied to the compression
158 side (air side) in order to hold the glass fragments together after fracture. The
159 specimens, jig sizes and the cross head speed comply with ASTM C 1499 (2003). The
160 crosshead speed of 0.02 mm/s was selected in order to fracture the specimens within 2
161 minutes, thereby limiting the effect of slow crack growth (Wiederhorn 1967 and Munz
162 and Fett 1999). The CDR setup induces an equibiaxial stress state on the surface of the
163 glass within the loading ring, therefore fracture is expected to originate at the largest
164 flaw within the loading ring, where the tensile stress is at its peak. Load at failure and
165 test duration were recorded.

166

167 *Four point bending*

168 A four point bending setup was used to test the strength of three different edge finishes
169 of as-received and annealed glass. The edge finish was as follows (Fig. 4):

- 170 - As-cut: the edge is sharp and might not be perfectly straight; density of flaws is
171 not controlled (DIN 1986);

- 172 - Grinded: the edge is chamfered and grinded; chips and flaws are allowed; the
173 finish is opaque (DIN 1986);
- 174 - Polished edge: the edge is chamfered and polished; flaws and chips do not occur;
175 slight polishing marks are allowed; the surface finish is shiny (DIN 1986).

176 All chamfers are 1.5 mm long at an angle of 45°. The different edge finish affects the
177 morphology and the density of the flaws, which will directly affect the stress at failure.
178 However, the effect of the flaws cannot be quantified non-destructively, but can be
179 determined by comparing the stress at failure.

180 The universal testing machine used is the same as for the CDR tests, fitted with a 4PB jig
181 that loads the 150 mm long glass specimens at third points (i.e. 50mm shear span and
182 50 mm load span). A double hinged connection between the crosshead and the loading
183 arm allows the load to be applied uniformly. The sizes of the specimens and the jigs
184 comply with ASTM C 1161 (2008). A crosshead speed of 0.02 mm/s was used in these
185 tests, to induce fracture within 2 minutes.

186 The 4PB setup induces bending about the major axis of the specimens, thereby resulting
187 in a uniaxial tensile stress state which is constant along the 50 mm load span length of
188 the bottom edge (as-cut/grinded/polished). Fracture is therefore expected to originate
189 at the largest flaw within the load span. A transparent self-adhesive tape was applied on
190 both sides of the beam in order to retain glass fragments together after fracture.

191

192 *Annealing and surface microscopy*

193 Annealing was performed in the laboratory to remove the residual stress from the as-
194 received glass. The annealing cycle was identical to that used by El-Sayed and Hand
195 (2011) i.e. heating the glass at a rate of 2°C/min up to 560°C, holding for 2 hours and
196 then cooling it at 2°C/min to room temperature.

197 Edges of series II, III and IV were also examined with an optical microscope before and
198 after the annealing process (Fig. 5). Flaws were recorded and measured. The
199 investigation was carried out to establish whether the annealing process had caused
200 any visible morphological changes in the flaws.

201

202 *Photoelastic stress measurements*

203 Residual stresses were measured for all the specimens with a SCALP. One reading per
204 side per specimen was performed. For the series I the reading was made in the middle
205 of the plate where failure was expected to originate. For the remaining series II, III, IV,
206 although failure was expected to originate at the edges, it was not possible to measure
207 the residual stress at this location due to restrictions of the device (Glasstress Ltd,
208 Scattered Light Polariscope SCALP instruction manual ver 5.5, unpublished).

209 A measurement representative of the residual stress of the specimen was therefore
210 made on the 20 mm side, parallel to the length of the specimen (Fig. 5). Typical SCALP
211 measurements are shown in Fig. 7 and 8 for as-received and annealed specimens,
212 respectively. The arithmetic mean of the residual stresses obtained for the respective
213 series are shown in table 5. Edge working in the form of cutting, grinding and polishing
214 is also expected to produce residual stresses in the vicinity of the edge, but it was not
215 possible to measure this and these residual stresses are assumed to be relatively
216 constant within each series thereby having a negligible effect on the comparisons made
217 in this paper.

218

219 **Results**

220 Stress at failure was calculated using Kirchhoff-Love plate theory for CDR tests and
221 Euler-Bernoulli beam theory for the 4PB tests.

222 For CDR tests, in the particular case of annular loading and support stress at failure
 223 equals (ASTM C 1499-2003 and Young et al. 2002):

$$224 \quad \sigma_f = \frac{3L}{2\pi h^2} \left[(1 - \nu) \frac{\phi_S^2 - \phi_L^2}{2\phi^2} + (1 + \nu) \ln \frac{\phi_S}{\phi_L} \right] \quad (3)$$

225 where,

226 L is the load at failure in N,

227 h is the glass thickness in mm,

228 ϕ_S is the diameter of the reaction ring in mm,

229 ϕ_L is the diameter of the loading ring in mm,

230 ν is the Poisson ratio

231 ϕ is the diameter of a circle that expresses the characteristic size of the plate and for a

232 squared plate can be expressed as follows:

$$233 \quad \phi = \frac{l}{0.90961 + 0.12652 \frac{h}{\phi_S} + 0.00168 \ln \frac{l - \phi_S}{h}} \quad (4)$$

234 where l is the length of the side of the square glass specimen in mm.

235 For the 4PB tests, the reduction in second moment of area due to the chamfers was

236 taken into account, as failing to do so would lead to an error of 11.6% in tensile stress.

237 The relatively simple equations are not shown here for brevity.

238 In order to compare data independent of stress history, failure stresses were converted

239 to a 60 s equivalent stress (Haldimann et al. 2008 and Overend and Zammit 2012). This

240 represents the constant tensile stress to which the given specimen should be subjected

241 in order to induce failure after 60 seconds. In the general case this can be expressed as

242 follows:

$$243 \quad \sigma_{t60} = \left[\frac{1}{t_0} \int_0^{t_f} \sigma^n(t) dt \right]^{1/n} \quad (5)$$

244 where,

245 σ_{t60} is the 60 s equivalent stress,

246 t_0 is the equivalent time period, (60s),

247 $\sigma(t)$ is the stress history,

248 t_f is the time at failure of the test,

249 n is the slow crack growth parameter, 16 for float soda-lime-silica glass (Haldimann et
250 al. 2008).

251 For the case of constant stress rate used in this study, Eq. (5) can be re-written as
252 follows:

$$253 \quad \sigma_{t60} = \sigma_f \left[\frac{t_f}{t_{60}(n+1)} \right]^{1/n} \quad (6)$$

254 Mean 60 s equivalent failure stresses are shown in table 4.

255 Two-parameter Weibull statistical analysis was performed on the 60 s equivalent
256 failure stresses. The method of moments (EN 12603-2002) was used to find the best
257 fitting 2-parameter Weibull curve to the given test data. Table 6 shows: the resulting
258 Weibull parameters θ and β , representing the scale parameter and the shape
259 parameter, respectively; the Anderson-Darling goodness-of-fit statistic ρ_{AD} ; and the
260 0.001 and 0.5 fractile strengths, $f_{f;0.001}$ and $f_{f;0.5}$, respectively. The corresponding
261 cumulative Weibull plots are shown in Figs. 9-12.

262 The mean strength increase due to healing, \bar{f}_{Heal} for each series can be determined by
263 re-arranging Eq. (3) and accounting for any residual stress that is present after the
264 laboratory annealing (σ_{ResAN}), giving:

$$265 \quad \bar{f}_{Heal} = \frac{1}{16} \sum_{i=1}^{16} [(f_{AN,t60} + \sigma_{ResAN}) - (f_{AR,t60} + \sigma_{ResAR})]_i \quad (7)$$

266 where,

267 $f_{AN,t60}$ is the 60 s equivalent strength of the i-th annealed glass specimen,

268 σ_{ResAN} is the surface residual stress of the i-th annealed specimen,

269 $f_{AR,t60}$ is the 60 s equivalent strength of the i-th as-received glass specimen,

270 σ_{ResAR} is the surface residual stress of the i-th as-received specimen,

271 16 is the number of specimens for each batch.

272 The expressions in the first and second parenthesis of Eq. (7) are a measure of the
273 extrinsic strengths of annealed and as-received glass, respectively. The difference
274 between the extrinsic strengths of annealed and as-received glass is a measure of the
275 strength gain due to healing. Equation (7) is in fact equivalent to:

$$276 f_{Heal,Px} = [(f_{AN,Px} + \bar{\sigma}_{ResAN}) - (f_{AR,Px} + \bar{\sigma}_{ResAR})] \quad (8)$$

277 where, for each series,

278 $f_{Heal,Px}$ is the strength gain corresponding to the chosen fractile Px ;

279 $f_{AN,Px}$ is the annealed glass strength corresponding to the chosen fractile Px ;

280 $f_{AR,Px}$ is the as-received glass strength corresponding to the chosen fractile Px .

281 In this paper Eq. (8) has been used to calculate the extrinsic strength gain due to healing
282 in each series at the 0.5 and 0.001 fractiles (table 7).

283 Comparison of flaws performed with an optical microscope before and after annealing
284 did not reveal any morphological changes in the density of the flaw. Typical
285 micrographs from this study are shown in fig. 6 a-d for as-cut and polished edge finish
286 (series II, IV). It was difficult to ascertain any differences in the depth of the flaws
287 perpendicular to the plane of view, but there was no apparent change to the length
288 along the plane of view and other visible morphological features.

289

290 **Discussion**

291 *Thermal healing*

292 The strength gain due to crack healing can be assessed by comparing the extrinsic
293 strength of annealed glass with the extrinsic strength of as-received glass for each test

294 series. By considering mean values and 0.5 fractile (best-fit) values in table 7 it is
295 evident that strength gain occurs for all series. There are however significant
296 differences in the lower fractile (0.001) values. More specifically, the as-cut and the
297 grinded series show an extrinsic strength loss, whilst the CDR and the polished series
298 exhibit a gain in extrinsic strength. This suggests that healing can have a significant
299 influence on the low fractile values typically used in real-world applications, but that
300 this phenomenon is sensitive to the edge or surface quality. A further illustration of this
301 can be seen in the Weibull plots in figs. 9-12, which show that heat treatment was
302 successful in reducing the scatter of failure stress values (i.e. the gradient of the best-fit
303 line) for smaller flaws (CDR and polished series) whilst it increased the scatter for
304 specimens with larger flaws (as-cut and grinded series). Furthermore, the best-fit lines
305 of the polished series are almost parallel, indicating that the strength gain is fairly
306 consistent for all flaw sizes present on the polished edges.

307 The sensitivity of healing to edge/surface quality is confirmed further by considering
308 the 4PB series alone. Here the strength gain appears to be correlated with the quality of
309 the edge finish. More precisely, not only does the edge quality correlate with higher
310 strength (as expected), but the extrinsic strength gain from thermal healing is also more
311 significant. This trend is confirmed by the 0.001 fractile although in this case the
312 improvement is from a significant strength loss for the as-cut series (-28.8%) to a
313 moderate strength loss (-4.1%) to a substantial strength gain (+35.0%).

314 In comparison, series I showed healing for all the fractiles, but the healing had a much
315 larger beneficial effect on the lower fractile strength. This suggests that of all the flaw
316 populations considered in this study, healing was most effective for the smaller flaws
317 encountered on the glass surface (series I).

318 Microscopical investigation of the edges before and after annealing did not show any
319 change in flaw morphology (Fig. 6). However this does not rule out that a morphological
320 change occurs at a smaller scale or on areas which are impossible to investigate with an
321 optical microscope. A comparison of the flaw size and morphology before and after
322 annealing would help to explain the nature of the healing mechanism. Currently it is
323 possible to measure it only after failure (Fig. 13), but not before, thereby ruling out the
324 possibility to know the flaw size before annealing. However, a possible explanation of
325 the thermal healing mechanism can be drawn by merging the findings of this
326 experimental investigation with the existing literature. In fact, it is likely that in the
327 vicinity of the flaw tip a combination of applied stress, morphology of the flaw and
328 humidity affect the strength before and after heat treatment. Namely, in as-received
329 float glass (before heat treatment) (Fig. 14a):

- 330 - A residual stress profile with compression on the surface and tension in the core
331 exists. This is typical of as-received float glass;
- 332 - There is humidity at the flaw tip.
- 333 - The crack is formed and its geometry is characterised by a sharp tip;
- 334 - This is immediately followed by the formation of radial/median/lateral cracks
335 just below the flaw tip (Schula and Schneider 2013);
- 336 - Crack formation also causes local stresses at the flaw tip, similarly to those
337 generated during an indentation (Anunmana et al. 2009);

338 After heat treatment (annealing) (Fig. 14b):

- 339 - The residual stress profile is relaxed as confirmed by the photoelastic stress
340 measurements performed in this study;

- 341 - The crack retains its overall morphology as confirmed by the visual inspection
342 (Fig. 6), but an optically invisible blunting at the crack tip may occur. This
343 increases glass strength by reducing stress concentrations (Watson et al. 2013);
344 - And/or sub-critical cracks tend to close (re-bonding) as they match the
345 definition of reversible cracks (Griffith 1920);
346 - Local stresses in the vicinity of the flaw tip undergo relaxation (Girard 2011);
347 - If the crack surfaces are in close optical contact humidity levels at the tip would
348 not rise instantaneously, thereby, leading to an apparent gain in strength
349 (Wiederhorn and Townsend 1970, Michalske and Fuller 1985), but on its own it
350 cannot explain the increase in strength observed in this study.

351

352 *Edge strength*

353 Another important finding independent of thermal healing, is that the quality of
354 surface/edges (i.e. flaw density and morphology) investigated in this study (which are
355 typical of those found in real-world applications) has a relatively small influence on the
356 mean and 0.5 fractile strengths, but has a very significant effect at the low fractile
357 strengths commonly used in design applications. For example, polished edges in the as-
358 received glass are on average 4.7 MPa (3.5%) stronger than as-cut edges (table 4), but
359 the strength of polished edges at the 0.001 fractile value is 39.4 MPa (114.2%) higher
360 than that of as-cut edges (table 6). This influence of edge finish at low fractile values is
361 even more pronounced after thermal treatment (annealing). The reason for this
362 sensitivity at low fractile values is that although the mean (and 0.5 fractile) values are
363 only marginally affected by edge finish, the scatter of failure strengths (and implicitly
364 the flaw sizes) are significantly reduced by grinding and more so by polishing. This is
365 also evident in the magnitude of the shape parameter β in table 4 and manifests itself in

366 the increasing slope in the best-fit lines when comparing across fig. 10, fig. 11 and fig.
367 12.

368

369 **Conclusions**

370 This study showed that thermal healing of realistic flaws can induce a significant
371 strength gain in soda lime silica glass. This was quantified by testing as-received glass
372 specimens and glass specimens carefully annealed in the laboratory and comparing
373 their strength at failure. The effect on glass surface strength and on the edge strength of
374 three different edge finishes was considered. The results showed that the mean strength
375 increase for the glass surface, as-cut edges, and grinded edges was in the order of 1.9%
376 to 4.8%, but that this increase was 18.9% for polished edges. The effect of thermal
377 healing at low fractile values used in design applications (e.g. 0.001) was even more
378 pronounced for the polished edges with an increase as high as 35%, whilst as-cut and
379 grinded edges showed a decrease of 28.8% and 4.1%, respectively. The overall trend
380 was that a better quality edge finish resulted in a higher strength gain or healing.

381 This study also showed that for the low strength fractiles commonly used in design
382 applications, a good quality edge finish results in significantly higher edge strength.
383 Namely as-received polished edges proved to be 114.2% stronger than as-received as-
384 cut specimens. The same figure for average values is as low as 3.5% instead. The
385 benefits of a good quality edge finish at low fractile values are even more substantial
386 when glass undergoes thermal healing.

387 More work is required to better understand crack healing, in particular there is a need
388 to:

- 389 - Investigate different heating cycles. In fact cycles at a temperature higher than
390 the transition temperature may trigger increased morphological modifications

391 and changes at the atomic structure level. Also, thermal heating cycles typical of
392 thermal tempering and chemical tempering could be of crucial importance for
393 the application of these products;

394 - Investigate the morphological change of both natural flaws and artificially
395 induced cracks, with the help of more powerful instruments, such as an atomic
396 force microscope;

397 - Investigate crack healing for different surface flaws population (i.e. as-received
398 glass vs naturally weathered glass) to determine whether healing has a similar
399 effect on different surface flaw populations.

400

401 **Acknowledgements**

402 The contribution of Chris Cavanagh to part of the experimental testing, Trend Marine
403 Ltd and EPSRC is gratefully acknowledged.

404

405 **Notation list**

406 The following symbols are used in this paper:

407 a = crack depth;

408 f_{AN} = extrinsic strength of annealed glass;

409 $f_{AN,Px}$ = annealed glass strength for a given fractile P_x ;

410 $f_{AN,t60}$ = 60 s equivalent strength of the i -th annealed specimen;

411 $f_{AR,Px}$ = as-received glass strength for a given fractile P_x ;

412 $f_{AR,t60}$ = 60 s equivalent strength of the i -th as-received specimen;

413 $f_{f;0.001}$ = 0.001 fractile strength;

414 $f_{f;0.5}$ = 0.5 fractile strength;

415 f_{FT} = extrinsic strength of fully tempered glass;

416 f_{HEAL} = strength gain due to healing;

417 \bar{f}_{Heal} = average strength gain due to healing;

418 $f_{Heal,Px}$ = strength gain due to healing for a given fractile P_x ;

419 h = glass specimen thickness;

420 K_I = stress intensity factor for mode I loading;

421 L = load at failure;

422 l = length of the side of the square glass specimen;

423 n = slow crack growth parameter;

424 t_0 = reference time period;

425 t_f = time to failure;

426 Y = flaw geometry factor;

427 β = surface strength shape parameter describing Weibull distribution;

428 θ = surface strength scale parameter describing Weibull distribution;

429 ν = Poisson's ration;

430 $\pi = 3.14159265359$;

431 ρ_{AD} = Anderson-Darling Weibull goodness of fit index;

432 σ = nominal tensile stress normal to the crack plane;

433 σ_f = stress at failure;

434 $\sigma^n(t)$ = stress history;

435 σ_{RES} = surface residual stress;

436 σ_{ResAN} = surface residual stress of the i-th annealed glass specimen;

437 $\bar{\sigma}_{ResAN}$ = average surface residual stress of annealed glass;

438 σ_{ResAR} = surface residual stress of the i-th as-received glass specimen;

439 $\bar{\sigma}_{ResAR}$ = average surface residual stress of as-received glass;

440 σ_{t60} = 60 s equivalent stress;

441 \emptyset = diameter of a circle that express the characteristic size of the glass plate;

442 \emptyset_L = diameter of the loading ring;

443 \emptyset_S = diameter of the reaction ring.

444

445 **References**

446 Anton, J., Aben, H. (2003). "A compact scattered light polariscope for residual stress
447 measurement in glass plates." *Proc. Glass processing days*, Tampere, Finland.

448 Anunmana, C., Anusavice, K. J., Mecholsky, J.J., (2009). "Residual stress in glass:
449 indentation crack and fractography approaches." *Dent.Mater.*, 40(11), 1453-1458.

450 ASTM (2003). "Standard method for monotonic equibiaxial flexural strength of advanced
451 ceramics at ambient temperature." *C1499-03*, West Conshohocken, PA.

452 ASTM (2008). "Standard test method for flexural strength of advanced ceramics at
453 ambient temperature." *C1161-02c*, West Conshohocken, PA.

454 DIN (1986). "Glass in building; glass edges; concept, characteristics of edges types and
455 finishes." *1249-11:1986-09*, Berlin, Germany.

456 EN (2002). "Glass in building. Procedures for goodness of fit and confidence intervals
457 for Weibull distributed glass strength data." *12603:2002*, European committee for
458 standardization, Brussels, Belgium.

459 EN (2004). "Glass in building – Basic soda lime silicate glass products – Part 1:
460 Definitions and general physical and mechanical properties". *572-1:2004*, European
461 committee for standardization, Brussels, Belgium.

462 El-Sayed, T., Hand, R. J. (2011). "Modelling the strengthening of glass using epoxy based
463 coatings." *J. Eur. Cer. Soc.*, 31, 2783-2791.

464 Girard, R., Faivre, A., Despetis, F. (2011). "Influence of water on crack self-healing in
465 soda-lime silicate glass." *J. Am.Cer. Soc.*, 94, 2402-2407.

466 Griffith, A. A. (1920). "The phenomena of rupture and flow in solids." *Phil.Trans. R. Soc.,*
467 *Series A*, 221, 163-198.

468 Haldimann, M., Luible, M., Overend, M. (2008).*Structural use of glass*.Struct. Eng.Doc. no.
469 10, International Association of Bridge and Structural Engineers.

470 Hrma, P., Han, W. T., Cooper, A. R. (1988). "Thermal healing of cracks in glass." *J. non-*
471 *cryst.solids*, 102, 88-94.

472 Inagaki, M., Urashima, K., Toyomasu, S., Goto, Y., Sakai, M. (1985). "Work of fracture and
473 crack healing in glass." *J. Am.Cer. Soc.*, 68, 704-706.

474 Irwin, G. (1957). "Analysis of stresses and strains near the end of a crack transversing a
475 plate." *J. Appl. Mech.*, 24, 361-364.

476 Michalske, T. A., Fuller, E. R. (1985). "Closure and repropagation of healed cracks in
477 silicate glass." *J. Am.Cer. Soc.*, 68(11), 586-590.

478 Munz, D., Fett, T. (1999).*Ceramics: mechanical properties, failure behaviour, materials*
479 *selection*.Springer, Berlin and Heidelberg, Germany.

480 Nielsen, J. H., Olesen, J. F., Stang, H., (2010). "Characterization of the residual stress state
481 in commercially fully toughened glass". *J. Mat. Civ. Eng.*, 22(2), 179-185.

482 Nielsen, J. H., Olesen, J. F., Poulsen, P. N., Stang, H. (2010). "Finite element
483 implementation of a glass tempering model in three dimensions." *Comp. and Struct.*, 88,
484 963-972.

485 Overend, M., Zammit, K. (2012). "A computer algorithm for determining the tensile
486 strength of float glass" *Eng. Struct.*, 45, 68-77.

487 Shelby, J. E., (1997). *Introduction to glass science and technology*. Roy. Soc. Chem.
488 Cambridge, UK.

489 Schula, S., Schneider J. (2013). "Characterization of the scratch visibility of annealed and
490 tempered soda-lime-silicate glass." *Proc., COST Action TU0905, Mid-term Conference on*
491 *Structural Glass*, Porec, Croatia.

492 Sullivan, J. D., Lauzon, P. H. (1986). "Experimental probability estimators for Weibull
493 plots." *J. Mat. Sc.*, 5, 1245-1247.

494 Varshneya, A. K. (2012). *Fundamentals of Inorganic Glasses*, The society of Glass
495 Technology, Sheffield, UK.

496 Watson, J., Nielsen, J. H., Overend, M. (2013). "A critical flaw size approach for predicting
497 the strength of bolted glass connections" *Eng. Struct.*, 57, 87-99.

498 Wiederhorn, S. M. (1967). "Influence of water vapour on crack propagation in soda-lime
499 glass". *J. Am. Cer. Soc.*, 50, 407-414.

500 Wiederhorn, S. M., Townsend, P. R. (1970). "Crack healing in glass." *J. Am. Cer. Soc.*, 68,
501 486-489.

502 Young, W., Budynas, R., Sadegh, A. (2002). *Roark's formulas for stress and strain, 7thed*,
503 McGraw-Hill, New York, NY.

504 Zaccaria, M., Overend, M. (2012). "Validation of a simple relationship between the
505 fracture pattern and the fracture stress of glass." *Proc., Engineered Transparency*,
506 Dusseldorf, Germany.

507 Zaccaria, M., Overend, M. (2014). "The mechanical performance of bi-treated
508 glass." *Proc., Challenging Glass 4 & COST Action TU0905 Final Conference*, Lausanne,
509 Switzerland.

510 Zijlstra, A. L., Burggraaf, A. J. (1968). "Fracture phenomena and strength properties of
511 chemically and physically strengthened glass." *J. Non-Cryst. Solids*, 1, 49-68.

512

513

514 **List of tables**

515 **Table 1.** SLSG composition (% mass) according to EN 572-1:2004.

516 **Table 2.** Relevant SLSG properties.

517 **Table 3.** Summary of test specimens.

518 **Table 4.** Test data and 60 s equivalent failure stresses.

519 **Table 5.** Measured residual stresses.

520 **Table 6.** Weibull analysis of 60 s equivalent failure stresses.

521 **Table 7.** Summary of extrinsic strengths and strength gains.

522

523 **Figure captions**

524 **Fig. 1.** Flaws on a glass pane surface, top view from an optical microscope.

525 **Fig. 2.** Vicker's indented glass beam cross-section after breakage.

526 **Fig. 3.** CDR test setup.

527 **Fig. 4.** From left to right as-cut, chamfered ground and chamfered polished edges.

528 **Fig. 5.** Schematic view of a 4PB specimen showing the edges examined under the
529 microscope and the position of the SCALP measurement.

530 **Fig. 6.** Micrograph of glass flaws: series II before (a) and after (b) annealing; series IV
531 before (c) and after (d) annealing.

532 **Fig. 7.** SCALP measurement on as-received glass.

533 **Fig. 8.** SCALP measurement on annealed glass.

534 **Fig. 9.** Cumulative Weibull plot for Series I.

535 **Fig. 10.** Cumulative Weibull plot for Series II.

536 **Fig. 11.** Cumulative Weibull plot for Series III.

537 **Fig. 12.** Cumulative Weibull for Series IV.

538 **Fig. 13.** Micrograph of polished edge after failure.

539 **Fig. 14.** Schematic explanation of crack healing: (a) flaw, as-received float glass, (b) in-
540 lab annealed glass.

541

542 **Tables**

543 **Table 1.** SLSG composition (% mass) according to EN (2004).

SiO₂	CaO	Na₂O	MgO	Al₂O₃	Others
69-74%	5-14%	10-16%	0-6%	0-3%	0-5%

544

545

546 **Table 2.** Relevant SLSG properties.

Property	Symbol	Value	Source
Density (kg/m ³)	ρ	2500	Haldimann et al. (2008)
Young's modulus (MPa)	E	70000	Haldimann et al. (2008)
Poisson ratio (-)	ν	0.23	Haldimann et al. (2008)
Stress intensity factor (MPa m ^{1/2})	K_{IC}	0.75	Overend and Zammit (2012)
Slow crack growth parameter	n	16	Overend and Zammit (2012)
Coefficient of thermal expansion (10 ⁻⁶ K ⁻¹)	α_T	9	Haldimann et al. (2008)
Glass transition temperature (°C)	T_g	575	Shelby (1997)
Annealing point (°C)	T_a	550	Shelby (1997)
Photoelastic constant (TPa)	C	3.01	Nielsen (2010)

547

548

549 **Table 3.** Summary of test specimens.

Series	Dimensions (mm)	Edge Finish	Test	# of specimens
I	150x150x6	N/A	CDR	16 as-received 16 annealed
II	150x20x6	As-cut	4PB	16 as-received 16 annealed
III	150x20x6	Grinded	4PB	16 as-received 16 annealed
IV	150x20x6	Polished	4PB	16 as-received 16 annealed

550

551

552 **Table 4.** Test data and 60 s equivalent failure stresses.

Series	Failure stress (MPa)		60 s equivalent stress (MPa)		Standard Deviation (MPa)	
	As- received	Annealed	As- received	Annealed	As- received	Annealed
I	179.3	181.0	147.3	148.9	74	51
II	173.1	172.6	134.5	135.2	28	38
III	170.2	175.0	135.0	139.4	21	23
IV	181.3	211.9	139.2	163.6	18	16

553

554

555 **Table 5.** Measured residual stresses.

Series	Mean Residual stresses (MPa)		Standard Deviation (MPa)	
	As-received	Annealed	As-received	Annealed
I	-4.6	-2.2	0.37	0.71
II	-4.2	-2.4	0.52	0.67
III	-4.4	-2.5	0.53	0.59
IV	-3.5	-2.2	0.61	0.56

556

557

558 **Table 6.** Weibull analysis of 60 s equivalent failure stresses.

	Series	Weibull Parameters		Goodness-of-fit	Fractile strengths	Fractile strengths
		θ	β	ρ_{AD}	$f_{f;0.5}$	$f_{f;0.001}$
I	As-received	175.6	2.02	0.071	146.4	5.7
	Annealed	167.0	3.27	0.11	149.7	20.3
II	As-received	148.9	4.72	0.11	137.8	34.5
	Annealed	<i>151.8</i>	<i>3.75</i>	<i>0.01</i>	<i>137.7</i>	<i>24.1</i>
III	As-received	145.0	6.80	0.32	137.5	52.5
	Annealed	151.3	6.08	0.29	142.5	48.6
IV	As-received	146.1	10.13	0.42	140.9	73.8
	Annealed	170.6	12.25	0.55	165.6	97.1

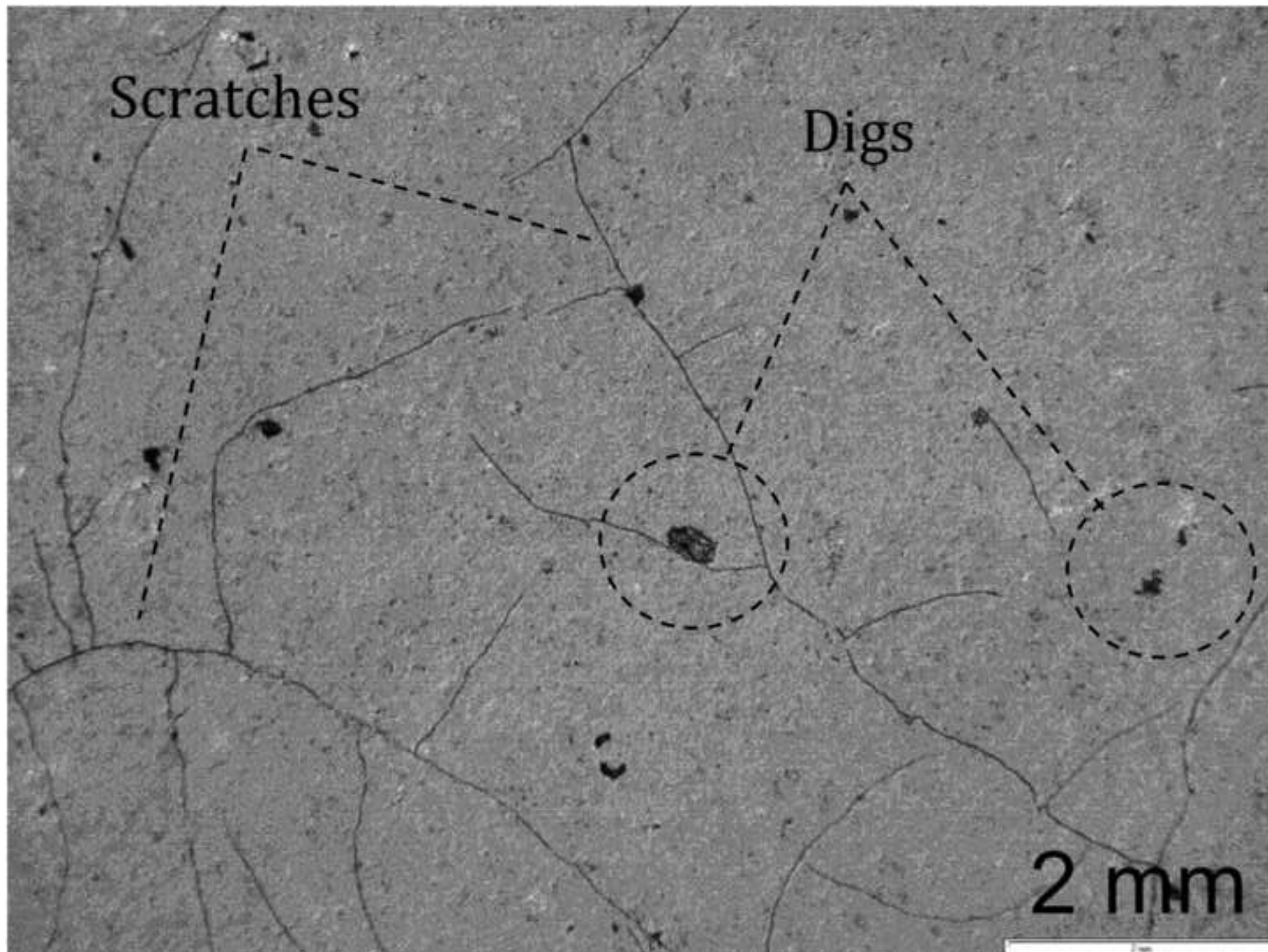
559 Note: values in italics indicate a poor Weibull fit.

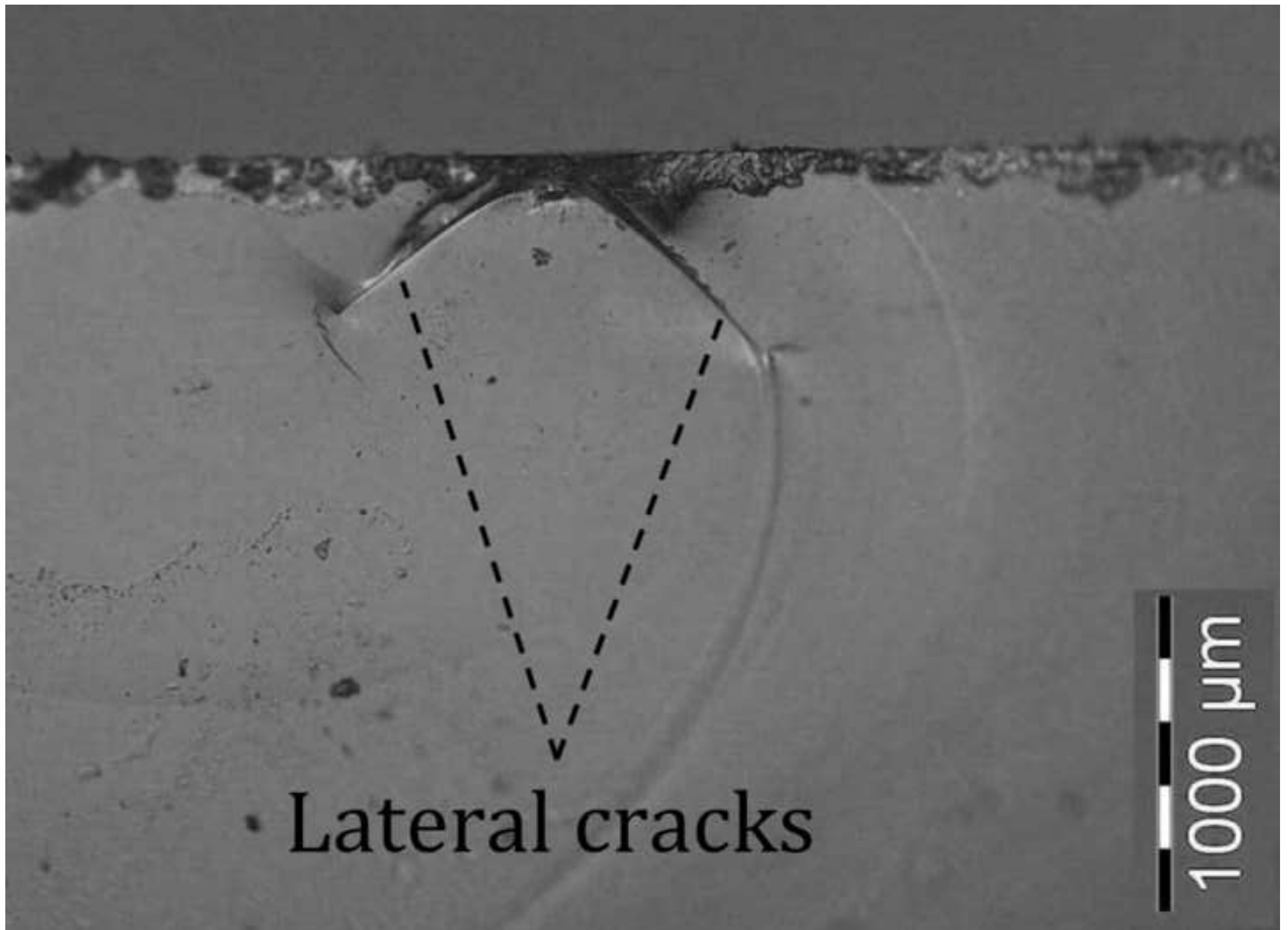
560

561 **Table 7.** Summary of extrinsic strengths and strength gains.

Series	Extrinsic strength of Annealed glass (MPa)			Extrinsic strength of As-received glass (MPa)			Extrinsic Strength gain (MPa)		
	$f_{AN,Px} + \bar{\sigma}_{ResAN}$			$f_{AR,Px} + \bar{\sigma}_{ResAR}$			f_{Heal}		
	Mean	0.5	0.001	Mean	0.5	0.001	Mean	0.5	0.001
I	146.7	147.5	18.1	142.7	141.8	1.1	4.0 (2.8%)	5.7 (4.0%)	17.0 (offlimits)
II	132.8	135.3	21.7	130.3	133.6	30.5	2.5 (1.9%)	1.7 (1.3%)	-8.8 (-28.8%)
III	136.9	140.0	46.1	130.6	133.1	48.1	6.3 (4.8%)	6.9 (5.2%)	-2.0 (-4.1%)
IV	161.4	163.4	94.9	135.7	137.4	70.3	25.7 (18.9%)	26 (18.9%)	24.6 (35.0%)

562





[Click here to download Figure: Figure 3.tif](#)



[Click here to download Figure: Figure 4.tif](#)

