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A simplified method for determining K_{IC} in glass with liquid solutions impregnation

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In order to determine K_{IC} in a simplified form, rectangular pieces of glass were scratched with a glazier's diamond, considering this scratch as the crack tip. The sides of the crack were made by sticking two rectangular pieces of proper thickness with epoxy resin as was made in a previous work. The K_{IC} was determined in glass in which the cracks are impregnated with liquid solutions of kerosene, sodium hydroxide and sodium silicate. For the samples impregnated with kerosene the mean value of K_{IC} remains constant, whereas for the other two solutions a decrease in the mean value of K_{IC} is observed, a 10% in samples impregnated with sodium hydroxide and 16% in samples impregnated with sodium silicate, when comparing them with their respective groups of samples of glass without impregnating. The corresponding Weibull diagrams were made and Weibull functions of three parameters were obtained. The respective parameters of the probability distribution functions of K_{IC} were estimated by means of the graphic method of nomograms.

1. Introduction

The traditional method for obtaining the critical stress intensity factor in mode I of fracture, K_{IC}, requires the generation, by fatigue, of an initial crack. This procedure, owing to its laborious application, implies a limited number of samples for determining K_{IC}. When dealing with brittle materials the problem even becomes more complex. For these reasons, a simplified method to determine K_{IC} in brittle materials has been developed and sucessfully applied to glass, cement mortar and compacted neat cement paste [1, 2]. The said method consists in making a pseudocrack in samples of rectangular cross-sectional area which are later subjected to the three-point bending test. Also in [1, 2] a statistical analysis of the results was made, finding thereby Weibull specific risk functions of two and three parameters and the Kies–Kittl specific risk function. Many authors, following the traditional experimental procedure for obtaining K_{IC}, obtained Weibull specific risk functions of two parameters [3–9].

The objective of the present work is to study the modifications of K_{IC} in glass, obtained through the three-point bending test, impregnating the pseudocrack with liquid solutions of kerosene, sodium hydroxide and sodium silicate. In this way the presence of the material in diverse environments, aggressive or not, is simulated. Also, for each group of samples the mean value of K_{IC} and the fracture mean strength were determined and the respective Weibull diagrams of probability [9, 10] were made.

2. Experimental procedure

Three groups of 40, 40 and 30 samples of glass in the form of beams of rectangular cross-sectional area of length L, width b, height W and crack size a were made from three pieces of glass sheet extracted from a given commercial glass sheet. These three pieces of glass sheet were taken from three different zones of that commercial glass sheet. For all samples, the crack was made in the same manner. From the pieces of glass sheet, rectangular bits of length L, width b and height $W/2$ were cut. Then, with a glazier's diamond a scratch was made at a distance $L/2$ from the bit ends and extending all its width. Also, from the same piece of glass sheet, rectangular bits of length $L/2$, width b and height $W/2$ were cut. Afterwards, the transversal edges of these last rectangular bits were polished by using a mechanical polishing disk and the same ones were employed as the profile of the crack induced. Finally, two of these bits of length $L/2$ were sticked with epoxy resin to every bit of length L in order to obtain samples having a pseudocrack formed by a scratch (crack tip) and the polished transversal edges (profile of the crack), as can be seen in Fig. 1. The scratch was situated on the side opposite to the one of application of load. Three saturated liquid solutions for impregnating the pseudocrack were employed, namely, kerosene, sodium hydroxide and sodium silicate. For each group, half of samples were impregnated and the other half remained unimpregnated, for the respective three solutions. In view of the fabrication of the samples, it is clear that each group of 40, 40

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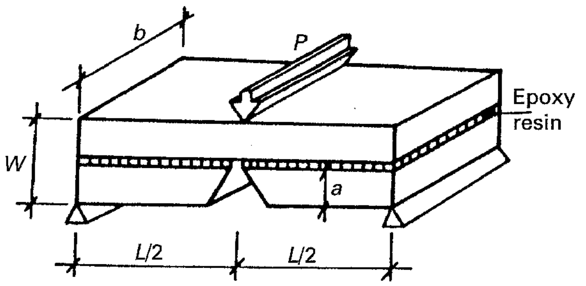

Figure 1 Schematic of the construction of the glass samples to be subjected to the three-point bending test. The pieces of length $L/2$ are stuck on the side opposite to the loading one.

and 30 samples came from a same piece of glass sheet; in other words, each one of the three pieces of glass sheet was employed for each group of samples. After impregnating every sample, and waiting for 2 min to have a good impregnation, the three-point bending test was performed immediately. For all samples the testing span was 40 mm.

3. Distribution of K_{IC}

According to Probabilistic Strength of Materials [9, 10], if the critical stress intensity factor K_{IC} follows a Weibull distribution, this can be pointed out as follows:

$$F(K_{IC}) = 1 - \exp\left\{-\frac{B}{B_0}\phi(K_{IC})\right\} \qquad (1)$$

where F is the cumulative probability, ϕ is the Weibull specific risk function, B_0 is the unit length and B is the crack width which, particularly in this work, is equal to beamwidth b, as can be seen in Fig. 1. The Weibull specific risk function, when assuming some analytical expression known *a priori* for it, can be expressed as follows:

$$\phi(K_{IC}) = \begin{cases} \left(\dfrac{K_{IC} - K_{ICL}}{K_{ICO}}\right)^m, & K_{ICL} \leqslant K_{IC} < \infty \\ 0, & 0 \leqslant K_{IC} \leqslant K_{ICL} \end{cases} \qquad (2)$$

where m and K_{ICO} are the Weibull parameters depending on the process of generation of the crack and on the material manufacturing and K_{ICL} is the lower limit value for the critical stress intensity factor under which no crack propagation occurs. When $K_{ICL} = 0$ the Weibull specific risk function of two parameters, m and K_{ICO}, is obtained. There is also another expression for the function $\phi(K_{IC})$, termed Kies–Kittl function [11], and given by

$$\phi(K_{IC}) = \begin{cases} K\left(\dfrac{K_{IC} - K_{ICL}}{K_{ICS} - K_{IC}}\right)^m, & K_{ICL} < K_{IC} < K_{ICS} \\ 0, & 0 < K_{IC} < K_{ICL} \\ \infty, & K_{ICS} < K_{IC} \end{cases} \qquad (3)$$

where K_{ICS} is the upper limit value for the critical stress intensity factor over which the crack propagation always occurs and K is the Kittl constant. Depending on the best fitting of the experimental data to one of the expressions pointed out in Equations 2 and 3 will be the statistical distribution followed by a given set of material samples.

The critical stress intensity factor was determined employing the following equation:

$$K_{IC} = \sigma Y \sqrt{a} \qquad (4)$$

where a is the crack length and Y is a function depending on the geometry of the samples which, after [12], is defined by

$$Y = Y\left(\frac{a}{W}, \frac{L}{W}\right) \qquad (5)$$

and can be obtained from the tables. In Equation 4, as σ is the maximal stress of fracture obtained in the three-point bending test, the expression for K_{IC} is transformed into

$$K_{IC} = \frac{3}{2}\frac{PL}{bW^2} Y\sqrt{a} \qquad (6)$$

where P is the fracture load, and L, b and W are the length, width and height of the beam, respectively.

In order to make the respective Weibull diagrams, $\ln[\ln 1/(1 - F(K_{IC})]$ against $\ln K_{IC}$, the following expression was employed as the cumulative probability estimator:

$$F(K_{IC}) = \frac{n - 0.5}{N} \qquad (7)$$

where the experimental values of K_{IC} have been ascendantly ranked, n is the number of samples having a critical stress intensity factor less than or equal to K_{IC} and N is the total number of samples tested.

4. Analysis of the results

In Figs 2–4 are shown the Weibull diagrams of K_{IC} of glass samples impregnated with the different liquid solutions of kerosene, sodium hydroxide and sodium silicate, respectively. As can also be observed, the distribution functions of K_{IC} for all groups of samples are in accord with Weibull distribution function of three parameters with the lower limit value for the critical stress intensity factor $K_{ICL} \neq 0$. In every figure

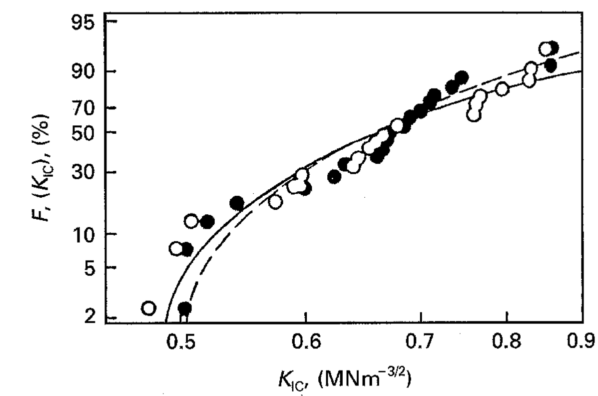

Figure 2 Weibull diagram for glass samples for (●) with and (○) without kerosene impregnation. The dashed and the continuous lines are the fitted curves to the experimental data without and with impregnation, respectively.

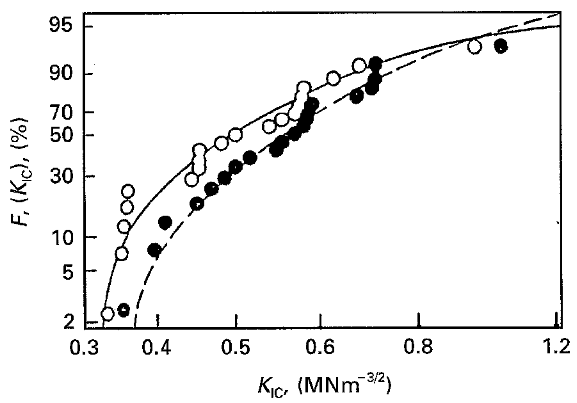

Figure 3 Weibull diagram for glass samples; (●) with and (○) without sodium hydroxide impregnation. The dashed and the continuous lines are the fitted curves to the experimental data without and with impregnation, respectively.

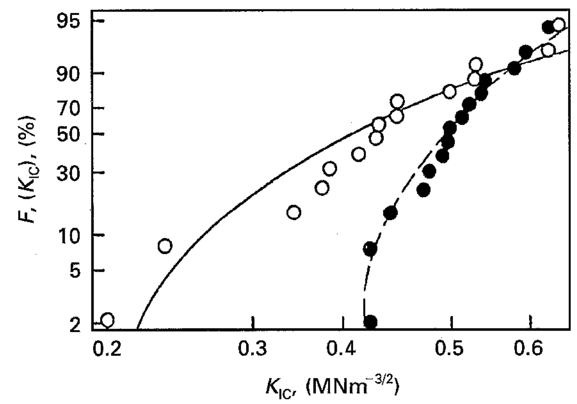

Figure 4 Weibull diagram for glass samples; (●) with and (○) without sodium silicate impregnation. The dashed and the continuous lines are the fitted curves to the experimental data without and with impregnation, respectively.

the diagrams for the glass with and without impregnation are shown, in order to make its interpretation easier. In Fig. 2 the Weibull diagrams for both groups of samples tend to superpose, that is to say, the impregnation with kerosene did not modify appreciably the distribution of K_{IC} and the value if K_{ICL} is practically the same one for samples with and without impregnation. In Figs 3 and 4, related to samples impregnated with sodium hydroxide and sodium silicate, a different behaviour is clearly appreciated with respect to their similar samples without impregnation. Here, the impregnation decreased the critical stress intensity factor, making easier the crack propagation because of the decrease in the value of K_{ICL}.

The number of glass samples, their mean dimensions, the mean size of the pseudocrack length, the mean values of the critical stress intensity factor and the mean strength of the samples with and without impregnation with each one of the liquid solutions are shown in Table I.

As in Figs 2–4, also from Table I, the noninfluence of the kerosene on the determination of K_{IC} when testing samples with and without impregnation was deduced. For the other two solutions, sodium hydroxide and sodium silicate, a decrease in the mean value of the critical stress intensity factor of 10 and 16%, respectively was determined. An important result to be emphasized is the one related to the mean values of K_{IC} obtained in glass samples without impregnating. Three mean values, quite different, were obtained: 0.67, 0.56 and 0.50 MN m$^{-3/2}$, with a difference up to 25%. This reaffirms the fact of dealing with independent populations, that is to say, three different pieces of glass sheet, each one for each group of samples tested with and without impregnation. As all of them came from the same commercial glass sheet, the anisotropy of it is evident.

From the physical viewpoint, the results shown in Table I may be explained as follows: the solutions of sodium hydroxide and sodium silicate are dissociated in their positive and negative ions, which is not the case for the kerosene. This dissociation allows that the sodium, as electrolyte and with atomic radius of order 1 Å, goes into the crack and contributes to its propagation when the material is loaded.

The Weibull parameters were estimated by a graphical method which consists in obtaining them by comparison using a nondimensional nomogram [10]. The values of these parameters for all groups of samples are shown in Table II.

From Table II, it is evident that the effect of both sodium solutions is not only to decrease the value of K_{IC} but also to decrease the value of parameter K_{ICL} and consequently to activate smaller cracks. Decreasing the critical stress intensity factor K_{IC} means decreasing the surface energy γ_s, the energy required to create a new fracture surface, as can be seen taking into account that $K_{IC} = \sigma(\pi a)^{1/2}$ is equal to $(2E\gamma_s)^{1/2}$, where σ is the stress activating the crack, a is the crack size and E is the Young modulus. From the energetic viewpoint, the decrease in surface energy

TABLE I Mean values of $\bar{K}_{IC}$ and mean strength $\bar{\sigma}$ of glass samples subjected to the three-point bending test and with pseudocrack induced. W is the beam height, and a is the crack length. Beam width $b = 11.3$ (mm) and testing span $L = 40$ (mm). WO means without impregnation and WI means with impregnation

Solution	WO/WI	Number of samples N	W (mm)	a (mm)	$\bar{K}_{IC}$ (MN m$^{-3/2}$)	$\bar{\sigma}$ (MPa)
Kerosene	WO	20	9.4	4.7	0.67	3.9
	WI	20			0.68	3.9
Sodium hydroxide	WO	20	7.7	3.9	0.56	3.6
	WI	20			0.50	3.2
Sodium silicate	WO	15	7.8	4.0	0.50	3.1
	WI	15			0.42	2.6

TABLE II Weibull parameters m, K_{ICL} and K_{ICO} of glass subjected to the three-point bending test with different liquid solutions impregnation. WO means without impregnation and WI means with impregnation

Solution	WO/WI	m	K_{ICL} ($MNm^{-3/2}$)	K_{ICO} ($MNm^{-3/2}$)
Kerosene	WO	2.0	0.47	0.047
	WI	2.0	0.45	0.045
Sodium hydroxide	WO	2.0	0.31	0.032
	WI	1.3	0.20	0.006
Sodium silicate	WO	1.5	0.39	0.017
	WI	1.5	0.21	0.011

may be explained as follows: as the sodium solutions are dissociated into positive and negative ions, the latter go to the fracture surface and remain in their most convenient positions, that is to say, positive ions of the solution are located in negatively charged vacancies of the fracture surface of glass and inversely for the negative ions, which makes that surface to become neutral. In this way, when propagating the crack the new surface becomes electrically uncharged and therefore the surface energy and also the values of K_{IC} and of parameter K_{ICL} are decreased with respect to a similar sample without impregnating.

5. Conclusions

The influence of three liquid solutions of impregnation, namely, kerosene, sodium hydroxide and sodium silicate, on the determination of the critical stress intensity factor was studied, also making a statistical analysis of experimental data with the Weibull distribution. All groups of samples followed a distribution function in accord with a Weibull function of three parameters. The kerosene did not have any influence, as was observed in the respective Weibull diagrams and in the mean values of K_{IC} of samples with kerosene impregnation with respect to those ones without impregnation. On the other hand, the solutions of sodium hydroxide and sodium silicate had influence on the critical stress intensity factor, decreasing the mean value of K_{IC} and causing a shifting of the corresponding curve in the Weibull diagrams with respect to the curve of samples without impregnating. The impregnation of the crack by means of the latter solutions also decreased the value of parameter K_{ICL} and consequently activated the smaller cracks—because of the smaller atomic radius of the sodium ion which may go into the cracks contributing then to their propagation and producing a decrease in the value of the surface energy γ_s of the glass by neutralizing the charged vacancies of the new broken surface. The environment in which the cracks are propagated is very important because some of them may change the propagation mechanism of cracks, making it easier as in the present research.

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