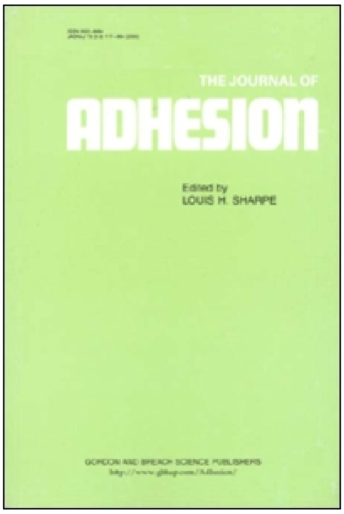

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Quantitative Measurement of the Energy of Fracture of an Adhesive Joint Using the Wedge-Test†

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The cleavage of adhesive joints upon the introduction of a wedge allows a measurement of the energy of fracture. Careful attention to the design of the joint leads to quantitative results. We show the relationship between the length of fracture and the geometrical parameters of the joint, the variation of the resistance of fracture with humidity; we also show that the fracture length follows a Weibull statistics, allowing for quality control.

KEY WORDS Cleavage of adhesive joints; durability; energy of fracture; resistance to fracture; wedge test; Weibull statistics.

INTRODUCTION

The cleavage of an adhesive bonded joint by a wedge is not usually used for quantitative determination of the adhesive joint properties due to the possible deformation of the adherends.

However, careful attention to the experimental conditions permits the determination of the adhesive resistance to rupture, R, and other related properties. If E is the Young modulus of the adherend, e its thickness, h the wedge height and l_0 the fracture length:

$$R = \frac{3Eh^2e^3}{16l_0^4} \tag{1}$$

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† Presented at the Tenth Annual Meeting of The Adhesion Society, Inc., Williamsburg, Virginia, U.S.A., February 22–27, 1987.

provided that there is no plastic deformation in the substrate and no energy dissipated or yielding in the adhesive layer.

Thus the measure of the fracture length gives the resistance to fracture through Eq. (1) and its relation to surface parameters or environmental conditions. Experimental verification was already given for the case of two long strips of glass joined together with silicone oil. In these conditions R was equal to twice the oil surface tension as would be expected.[1]

1 EXPERIMENTAL CONDITIONS

Two rectangular plates made from flat (±5 μm over 5 cm) metal strips are assembled with a liquid adhesive deposited as a central line using a syringe. The plates are filed at one end in order to form a mouth where the wedge can easily be introduced (Figure 1) and

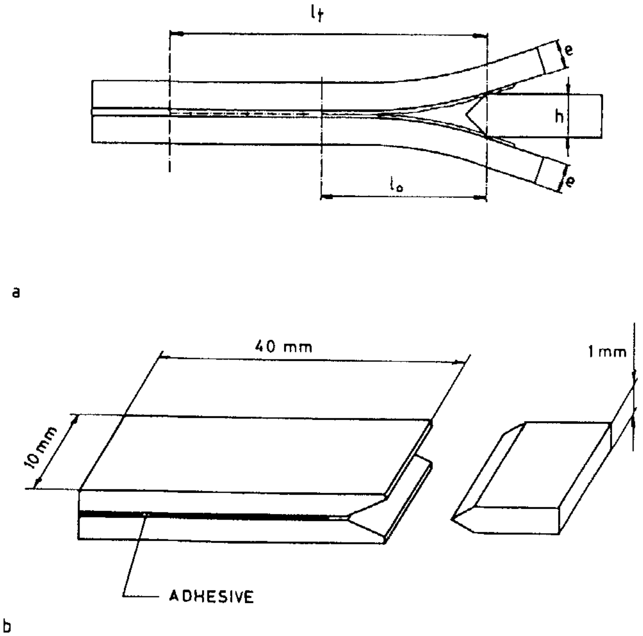

FIGURE 1 Schematic drawing of the wedge-test geometry.

then positioned and maintained with two clips. Polymerisation is carried out following the procedure recommended by the manufacturer. The resulting adhesive layer is very thin, of the order of 20 μm and it appears that thickness variations of the order of plus or minus 10 μm have little effect.

The wedge is introduced by means of a micrometric device (Figure 2) so that its edge protrudes some 2 mm from the mouth. This creates a crack, the length of which is measured by the distance separating the line where the wedge touches the beam and the crack tip. In order to locate the crack tip a micrometer is opened to the height of the assembly +0.01 mm; the crack tip is the point where the micrometer touches the assembly (Figure 3).

In general the metal used is hardened stainless steel AISI 304 of spring quality which avoids plastic deformation of the adherends. The adhesives used are formulations based on Shell, Ciba-Geigy and EMS-Chemie resins. The simplest formulation gives higher experimental scatter whereas filled adhesives give more reproducible results and also an initial cohesive failure. They will be designed by reference to the caption of Figure 6. The commercial adhesive AV 118 is used for purposes of comparison.

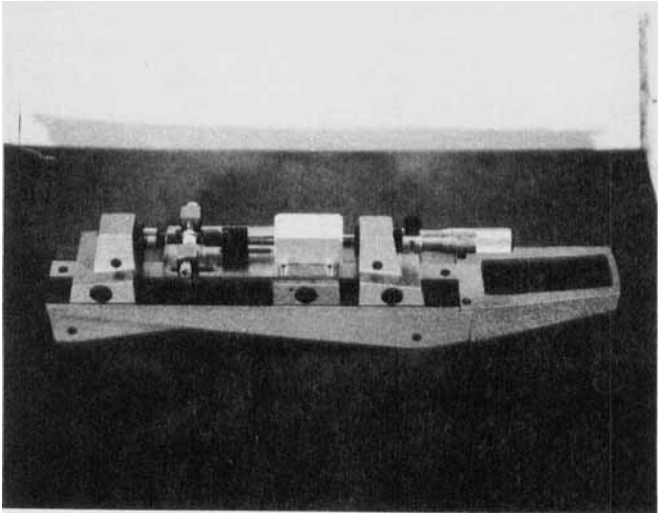

FIGURE 2 Micrometric device used to introduce the wedge.

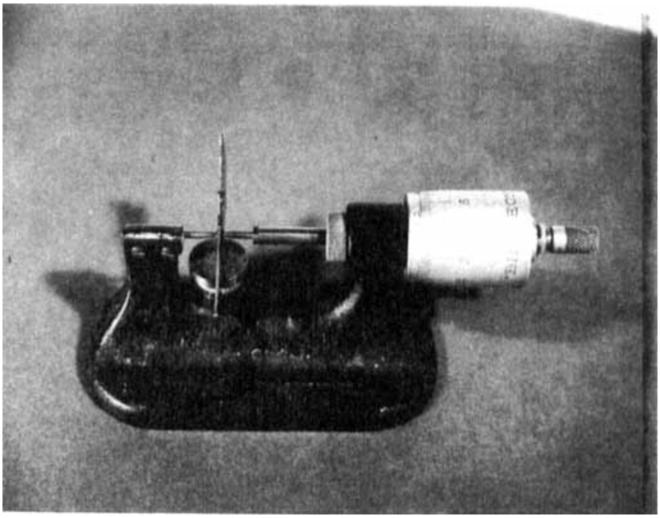

FIGURE 3 Determination of the position of the tip with a micrometer.

2 EQUILIBRIUM CONDITIONS

The kinetics of the fracture propagation has not been studied yet, thus the time necessary to attain equilibrium is not known precisely. Since driving potential for fracture propagation is the difference between the elastic strain energy release rate, G, and the energy of fracture, W_S,† crack growth becomes very slow when G approaches W_S. Nevertheless our latest results, obtained over one year, confirm our previous estimations[3,4] that in many cases the crack length is stable after one day in a dessicator. In Figure 4 the energies of fracture deduced from the crack length of five different epoxy adhesives are given. Only one rubber-modified epoxy adhesive shows slight crack progression which may be attributed to yielding. A joint made of the same adhesive, but filled, did not show any progression of fracture; neither did various epoxy-nylon adhesives. The wedge test is in fact, a double cantilever beam submitted to a constant displacement and thus the conclusions of S. S. Wang[5] *et al.* should be valid.

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† At equilibrium R and W_S are identical.

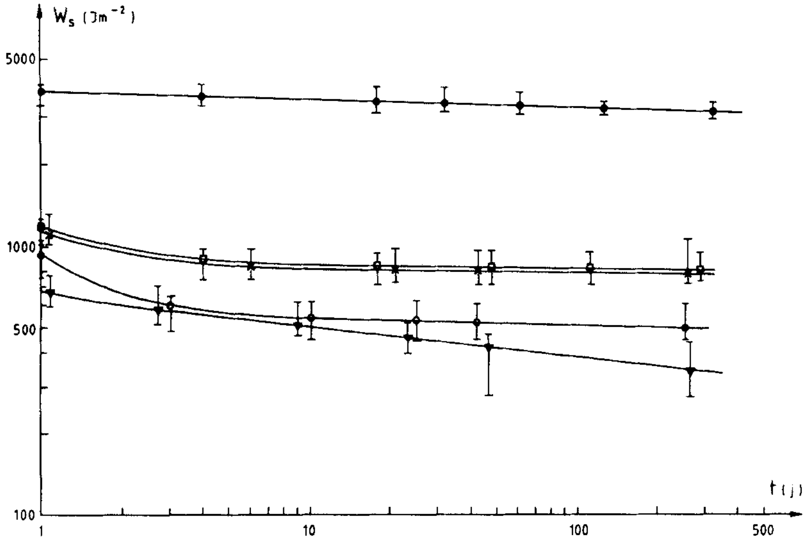

FIGURE 4 Variation of the energy of fracture with time for 5 adhesives. Only the unfilled, rubber-modified epoxy shows some variation probably due to yielding. ● epoxy nylon, □ epoxy-rubber modified, × filled epoxy-rubber modified, ○ epoxy nylon, ▼ epoxy-rubber modified (unfilled).

These authors used finite elements analysis and found, in the case of purely elastic behaviour, that the stress at the crack tip is similar to that in a monolithic system and may be described by the stress intensity factor K_I and the strain energy release rate G_I which are related to each other by $G_I = K_I^2/E_I$. Furthermore, they found that G_I is independent of the adhesive thickness as our experimental results[4] suggest.

3 INFLUENCE OF THE EXPERIMENTAL GEOMETRY

3.1 Influence of the wedge-height

In our configuration the substrate beam should have a limited length, l, and the crack length should be maintained below $l/2$. Since R varies as l_0^4 the accuracy is very low for small cracks in strong adhesives; therefore, l_0 values of some 10 mm are preferred.

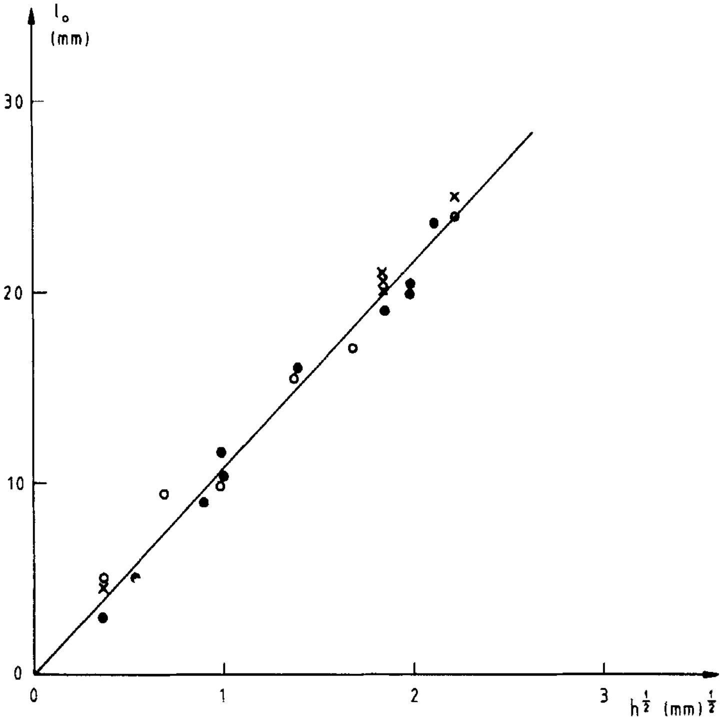

FIGURE 5 Variation of the fracture length with the square root of the wedge height.

The choice of the wedge height permits the variation of the crack length. Equation (1) applies for the joint [stainless steel-adhesive C (20 μ) – stainless steel] cleaved by wedges of height varying from 0.15 to 5 mm as we have shown. From the experimental parameters, Eq. (1) becomes

$$W_S^{0.25} = \frac{1.47}{l_0} h$$

and the slope of Figure 5 gives $W_S = 340\ \mathrm{Jm}^{-2}$ where h is 1 mm.

3.2 Application of the wedge-test to various substrates: influence of Young's modulus

Much of our work has been carried out on stainless steel of spring quality which is sometimes coated with another material.

In some cases it would be useful to study directly the adhesion to another metal. From Eq. (1) it can be seen that the simplest way to do this is to adapt the thickness of the substrate plates taking into account their elasticity. For example, titanium (BSTA 6) is easily deformed and has a modulus of 103 MPa. In order to cleave an assembly between two titanium beams 2 mm thick plates are used. In these conditions a 1 mm thick wedge provokes a cohesive fracture of 20 ± 2 mm in the joint using adhesive C18 which was polymerised at 140°C. Following Eq. (1):

$$W_S = \frac{1.54 \times 10^{-4}}{l_0^4\,(\mathrm{m})}\,\mathrm{Jm}^{-2}$$

The above values correspond to an energy of fracture of $1200 \pm 300\,\mathrm{Jm}^{-2}$. In contrast, adhesive fracture on stainless steel or gold gives 500 and $100\,\mathrm{Jm}^{-2}$ respectively.

Kennedy *et al.*[6] studied the cleavage of Redux 312/5 adhesive between titatium beams ($e = 2.10 \times 10^{-3}$) cleaved by a 3.17 mm wedge and found 53 mm of fracture length, corresponding to a resistance to fracture of $1515\,\mathrm{Jm}^{-2}$. This was very close to that which we observed with our formulation C.

4 INFLUENCE OF HUMIDITY

4.1 The debonding of adhesive joints

The debonding of adhesive joints in outdoor environments is highly undesirable and its origin is still not completely understood.

The cleaved adhesive joint placed in humid conditions clearly illustrate this phenomenom. When a cleaved assembly, which has its fracture in equilibrium with the laboratory atmosphere, is placed in a humid environment the fracture length increases. The kinetics of the variation depends upon the type of the adhesive for a given wedge-test configuration. As shown in Figure 6 some adhesives will debond very fast at 40°C and 90% RH (for example the plasticised resin D) while others (such as adhesive A) show a very slow evolution. The debonding process in humid conditions, in common with all environmental delamination, is always interfacial. When the initial fracture is cohesive it progresses to the interface and proceeds along it (Figure 7) whilst it is maintained in a humid atmosphere.

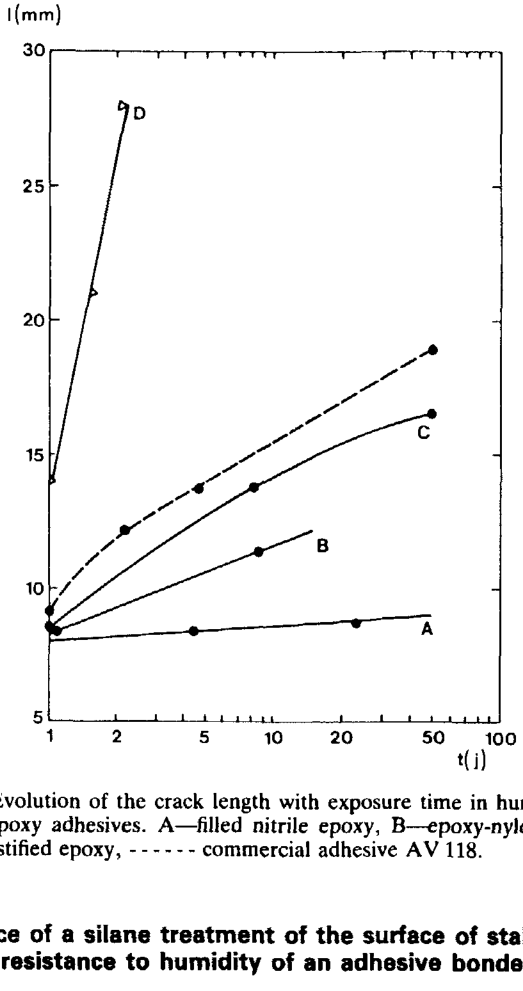

FIGURE 6 Evolution of the crack length with exposure time in humid conditions for different epoxy adhesives. A—filled nitrile epoxy, B—epoxy-nylon, C—epoxy-nitrile, D—plastified epoxy, ------ commercial adhesive AV 118.

4.2 Influence of a silane treatment of the surface of stainless steel on the resistance to humidity of an adhesive bonded joint

A study has been made with the plasticised adhesive D which, as mentioned above, is very sensitive to humidity and delamination. Two series of joints cleaved by the same process differ only in that one is silane treated and the other not. Upon cleavage the initial fracture is cohesive in both cases and runs over 14 mm, corresponding to a resistance to fracture $R = 75 \pm 25\ \mathrm{Jm}^{-2}$. After 24 hours in hot humid conditions (40°C; 90% RH) the crack length reaches 28 mm for the silane treated substrate while it exceeds 30 mm for the untreated samples (see Table I). After 4 days of exposure all the

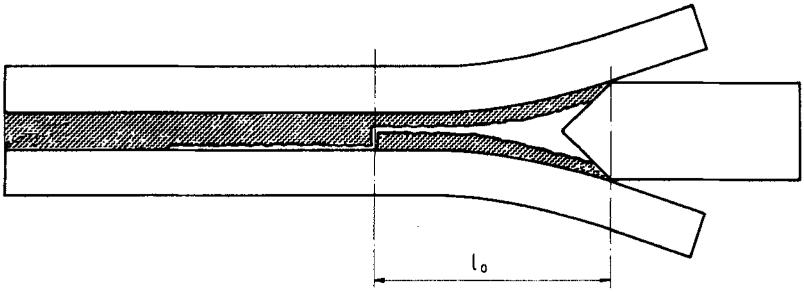

FIGURE 7 Crack always proceeds along interface in humid conditions. An initially cohesive fracture progresses to the interface and proceeds along it when maintained in the humid atmosphere.

TABLE I
Values of the crack length of joints in mm using adhesive *D*, between two stainless steel beams untreated or silane treated. l_0 is the initial fracture length $l(t)$ its value after time t of exposure to 40°C 90% RH

Stainless steel Silane treated	l_0	(24*h*)	$l(4j)$
	16	29	33
	16.8	25	31
	16.8	28	32
	16.2	28	32
	14.8	26	32
	16.2	26	33
Without treatment	17.8	34	debond
	13.8	35	debond
	16.3	26	debond
	17.8	36	debond
	14.8	35	debond
	17.5	36	debond

untreated samples are debonded while the silane-treated ones still have a resistance to fracture of some $5\,\mathrm{Jm}^{-2}$ ($\pm 0.5\,\mathrm{Jm}^{-2}$) showing the benefit of the silanisation.

5 THE DISTRIBUTION OF THE FRACTURE LENGTH FOR VARIOUS SAMPLES FOLLOWS WEIBULL STATISTICS

The mechanical properties of adhesive joints show a more pronounced scatter in the case of simple formulations and unfilled resins, as compared to commercial filled adhesives. Similar scatter is found for ceramics. For these materials it has been shown that the reliability of a given property, for example resistance to rupture, follows a Weibull statistics, that is $\ln \ln P_i^{-1} = m \ln \sigma - m \ln \sigma_0$ where P_i is the probability that σ is given value σ_i.[7] The observation that Weibull statistics can also be applied to cured bulk epoxy resins has recently been shown.[8] This led us to consider its possible applicability to the resistance to fracture in the wedge-test.

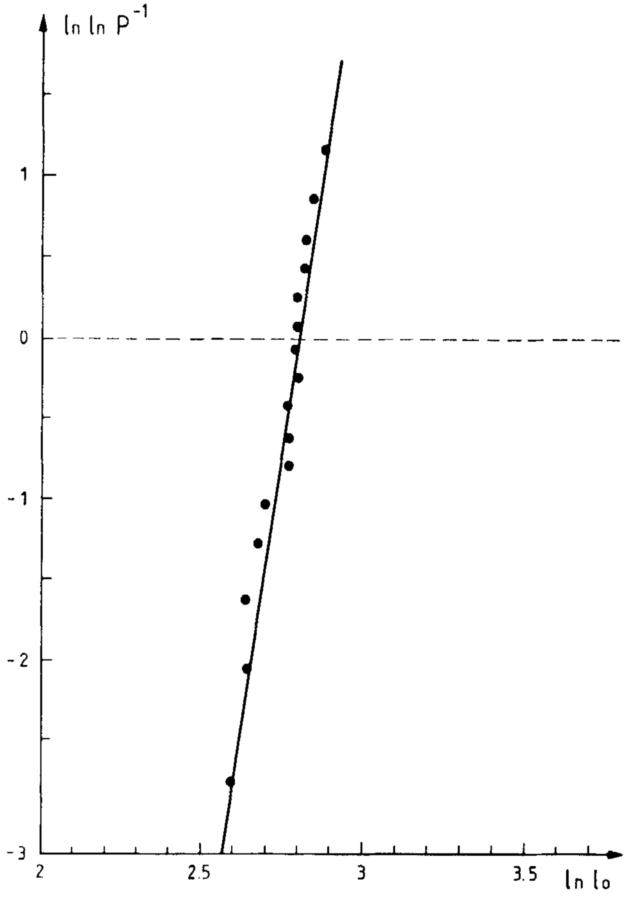

FIGURE 8 Weibull plot of the initial fracture length.

TABLE II

Values of the initial crack length for wedge-tests using adhesive D between stainless steel, in order of increasing value. P_i is the probability that l will be higher than l_i

No	l_0 (mm)	$P = 1 - \frac{1}{17}$	$\ln \ln P^{-1}$	$\ln l_0$
1	13.5	0.94	−2.78	2.60
2	14	0.88	−2.05	2.64
3	14	0.82	−1.61	2.64
4	14.5	0.76	−1.29	2.67
5	15	0.70	−1.03	2.70
6	16	0.64	−0.80	2.77
7	16	0.58	−0.61	2.77
8	16	0.52	−0.42	2.77
9	16.5	0.46	−0.25	2.80
10	16.5	0.40	−0.08	2.80
11	16.5	0.34	+0.07	2.80
12	16.5	0.28	−0.24	2.80
13	17	0.22	+0.41	2.83
14	17	0.16	+0.60	2.83
15	17.5	0.10	+0.83	2.86
16	18	0.04	+1.17	2.89

We have considered many adhesives and found that, in each case, the logarithm of the initial value of the length of fracture was a linear function of $\ln \ln P$. An example is given in Figure 8 for adhesive D, which has the highest scatter among the adhesives considered in this work. The law of probability P is obtained from the rank i of the N values of l_0, put in order of increasing length as shown in reference 7b.† From the values given in Table II, one finds the Weibull modulus for adhesives $D : m = 13.7$. Values of P give the probability that the fracture length reaches a given value and allows the quality control of the adhesive. In the case of adhesive D the probability that the fracture length exceeds 18 mm is 4% and that it exceeds 19 mm is 0.0009. In other words, there can only be 1/1000 joints which have a resistance to fracture below 30 $\mathrm{J m^{-2}}$ (corresponding to $l_0 = 19$ mm).

Although Weibull statistics apply well to the initial fracture this is not so as far as the progression in humid atmosphere is concerned, which prohibits any prediction of the behaviour in such conditions.

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† $P = 1 - \frac{i}{N+1}$.

What is actually observed is that those adhesive joints which are initially weaker (that is having the greater length of fracture) are also those that are more sensitive to humidity. For these joints, the progression of the fracture in a humid environment is much faster that it is for stronger joints.

CONCLUSION

The tests currently available to measure the properties of adhesive joints do not provide the data required for mechanical engineering. Quantitative use of the wedge-test can give the resistance to fracture of an adhesive joint and its variation with the parameters defining the adhesion problem. More work is needed to check the influence of thickness, internal stresses and general applicability of the results. The main drawback in the method is that the locus of failure is only known at the end of the test by destruction of the assembly. Nevertheless, we have shown that sound results (comparable to other more time consuming and expensive methods) may be obtained which allow a much better characterisation of the joints than was available up to the present time. In addition, knowledge of the fracture behaviour permits the prediction of the lifetime in service at least for indoor applications.

Acknowledgements

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