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### **Use of the Blister Test to Study the Adhesion of Brittle Materials. Part I. Test Modification and Validation**

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# Use of the Blister Test to Study the Adhesion of Brittle Materials. Part I. Test Modification and Validation

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In the traditional configuration of the blister test, where the adhesive sheet is mounted on top of the substrate of interest, a brittle or fragile adhesive exhibits early cohesive failure during pressurization, preventing the measure of the interfacial fracture energy  $F$ . To permit the determination of this important materials system parameter in ice-substrate systems, we modified the blister test to an inverted configuration, where the substrate is the continuous top sheet, and the brittle adhesive (ice) is in the form of a thin interlayer under the substrate, but mounted on a massive, inert base. Several aspects of the modified test were examined, and the test was found to be valid within the range evaluated.

**KEY WORDS** Blister test; interfacial fracture energy; system parameter; adhesive interlayer; brittle adhesives.

## I INTRODUCTION

There are two key requirements that must be met when one is using mechanical tests to study the adhesion between two dissimilar materials. The first is to use a mechanical test that yields a system parameter, independent of geometry. The second is to achieve failure at the interface between the two materials so that conclusions can be drawn about adhesion rather than cohesion.

The first requirement can usually be met by choosing the appropriate test; that is, one especially designed for determination of a system parameter. For many of the long-standing joint strength tests (butt tensile, lap shear, etc.) a system parameter is not readily extractable from test results. The test results are expressed as failure loads normalized by specimen cross-sectional area or specimen width, and these results are not independent of the other geometric details of the specimen or of the elastic properties of the adhering materials. Thus, many of the long-standing joint strength tests are limited to quality control applications or to comparison of surface treatments for a single adhesive-substrate system.

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Tests that are interpreted by fracture mechanics analysis do not have the above shortcomings. In fracture mechanics tests the details of the specimen geometry, the stress distribution in the specimen, and the elastic properties of the two adhering materials are taken into account explicitly. This allows the desired materials system parameter, characteristic of the interface alone, to be extracted from the test results. The system parameter is designated interfacial fracture energy, and is defined as the energy required to separate unit area of interface. Tests developed for the determination of interfacial fracture energy values in bimaterials systems include the double cantilever beam test<sup>1</sup> and the blister test.<sup>2,3,4</sup>

The second requirement in studying adhesion, that of achieving failure strictly at the interface, can be extremely difficult to meet with some adhesive systems. This difficulty arises when a substance that adheres well to the surface of another has low bulk strength. A good example of such a substance is ice, which adheres well to most surfaces but which is brittle and fragile as usually prepared. Attempts to separate ice from a substrate result in cohesive fracture of the ice itself, leaving a well adhered layer of ice on the substrate. This behavior precludes a meaningful study of ice adhesion.

The means to achieve interfacial failure when one material is brittle or fragile is not always clearcut. Attempts can be made to increase the bulk strength of the brittle material by eliminating flaws or by reinforcing it with another material in a way that does not interfere with the interface. Sometimes a change in the details of the joint geometry can produce the desired failure mode.

Considerable numbers of unsuccessful attempts to achieve interfacial failure between ice and various substrates led us to modify the blister test to a configuration that was inverted relative to our original trials. This was found to work well, and shows promise as a general technique for studying the adhesion of brittle or fragile materials.

Part I of this paper describes the modifications made to the traditional blister test and also presents the results of experiments conducted to confirm the validity of the modified version.

## II BACKGROUND

The traditional configuration of the blister test is shown in Figure 1. In this configuration, a circular nonbonded area (penny-shaped crack) is present at the interface between the adhesive sheet and the underlying substrate. As the specimen is pressurized from the inside, the nonbonded portion of the adhesive sheet deflects vertically like a blister. (The deflection is exaggerated for emphasis in the Figure). At some critical value of the pressure, designated by  $P_c$ , the pre-existing penny-shaped crack at the interface propagates radially. In a blister test, unstable, rather than stable crack propagation occurs, so that failure is catastrophic and only one  $P_c$  measurement can be made per specimen.

The interfacial fracture energy  $F$  is computed from an energy balance equation containing the measured  $P_c$ , details of specimen geometry, and the elastic

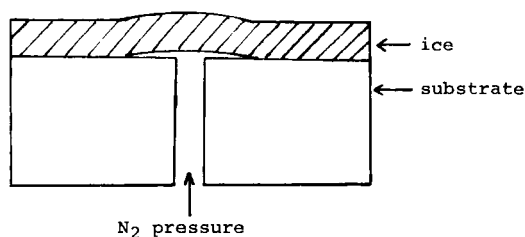


FIGURE 1 Traditional blister test configuration. A continuous sheet of the adhesive material surmounts the massive block of substrate. Circular debonded area, placed at the interface between the substrate and the adhesive sheet during specimen preparation, serves as initial crack. Pressure is applied through the central hole, deflecting the adhesive sheet upward (exaggerated in figure). At a critical pressure,  $P_c$ , the initial crack propagates, debonding the adhesive sheet from the substrate.

properties of the parts of the specimen that deform. It is usually assumed that the high thickness of the substrate prevents its deforming during the pressurization process. Therefore, the substrate is not part of the energy storage and release process and its elastic properties do not affect the values obtained for  $P_c$ .

Andrews and Stevenson have presented an elastic analysis of the blister test that concludes in an expression for fracture energy in terms of critical pressure  $P_c$ , specimen geometry, and adhesive sheet elastic properties.<sup>5</sup> They stated that the elastic strain energy stored in the system during pressurization is comprised of both near field (localized deformations in the adhesive near the crack tip) and far field energy (gross deformation of the adhesive sheet). The expression they use for near field stored strain energy is:

$$U_n = \frac{4(1-\nu^2)}{3E} P^2 c^3 \quad (1)$$

where  $c$  = initial crack radius,  $E$  = Young's modulus of the adhesive sheet, and  $\nu$  = Poisson's ratio of the adhesive sheet.

To obtain the far field contribution to the strain energy, Andrews and Stevenson computed the vertical deflection of the nonbonded portion of the adhesive sheet as a function of pressure by using Timoshenko and Woinowsky-Krieger's deflected thick circular plate model.<sup>6</sup> They then computed the elastic energy stored by the applied pressure acting through the equilibrium vertical deflection of the adhesive sheet. The resultant expression for the far field stored energy is:

$$U_f = \frac{P^2 \pi (1-\nu^2)}{32 E h^3} \left\{ c^6 + \frac{6h^2}{1-\nu} c^4 \right\} \quad (2)$$

where  $h$  = thickness of the adhesive sheet and all other symbols are as above.

At the critical pressure,  $P_c$ , the strain energy released by the system to form a unit area of crack is equivalent to  $F$ , the interfacial fracture energy. The released energy is found by taking the derivative of the total stored energy ( $U_n + U_f$ ) with

respect to interfacial area fractured at constant pressure. This result is:

$$F = \frac{dU}{dA} = \frac{P_c^2 c(1 - \nu^2)}{E} \left\{ \frac{3}{32} \left[ \left( \frac{c}{h} \right)^3 + \left( \frac{c}{h} \right) \frac{4}{1 - \nu} \right] + \frac{2}{\pi} \right\} \tag{3}$$

Because it is a system parameter,  $F$  should have a constant value no matter what the values of the geometric quantities  $h$  and  $c$ . The constancy of  $F$  can be verified by testing specimens with different values of  $h$  and  $c$ . A practical way to do this is shown by rearranging the above equation to:

$$P_c = \sqrt{EF} \sqrt{f(h/c)/c} \tag{4}$$

where

$$f(h/c) = \frac{1}{1 - \nu^2} \left\{ \frac{3}{32} \left[ \left( \frac{c}{h} \right)^3 + \left( \frac{c}{h} \right) \frac{4}{1 - \nu} \right] + \frac{2}{\pi} \right\}^{-1}$$

If  $F$  is truly a system parameter, independent of geometry, a plot of  $P_c$  versus  $\sqrt{f(h/c)/c}$  will produce a good straight line whose slope is equal to  $\sqrt{EF}$ . The value of  $F$  can be computed directly from this slope.

For situations where the top sheet, *i.e.*, the adhesive, is a fragile material, premature failure often occurs within the adhesive sheet itself in preference to failure at the interface. Figure 2 shows diagrams of these undesirable failures. Such cohesive failures nearly always occurred when ice served as the adhesive sheet on top of a thick block of the substrate of interest.

In an attempt to overcome the problem of cohesive failure in a brittle adhesive, we rearranged the bimetals system to put the tougher “substrate” as the continuous sheet on top of the ice, which now must have a central hole. This arrangement is shown in Figure 3, where the blistering of the “substrate” is exaggerated for emphasis. Figure 3 also reveals that the ice is now present as a thin layer, called an interlayer, attached to and supported by a block of inert material with a central hole. Ice as a thin, supported layer is more manageable than a large block of ice.

The “inverted” configuration in Figure 3, which is inverted only from our point of view of having placed the ice on the top earlier, gave reproducible data with the fracture locus at the “substrate”-ice interface. The analysis presented by Andrews and Stevenson<sup>5</sup> for the traditional configuration can be applied to the

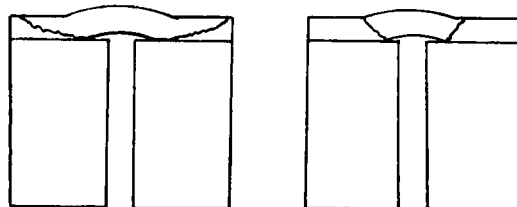


FIGURE 2 Undesirable failures occurring in the traditional blister test. When the adhesive sheet on top is a brittle material, the initial crack may propagate into the adhesive sheet itself rather than along the interface. When this happens, the measured critical pressure does not pertain to interfacial adhesion, but pertains to cohesive failure in the adhesive.

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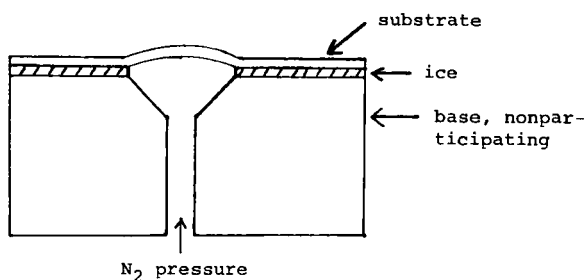


FIGURE 3 The inverted configuration of the blister test. The substrate, in the form of a thin continuous sheet, is on top. The brittle adhesive is present as a thin interlayer, between the substrate and a massive base. The central hole at the top serves as the initial debonded area; the circular junction line between the ice and the substrate forms a uniform crack tip. The vertical deformation of the substrate is exaggerated for emphasis.

inverted configuration as well. A refinement, if necessary, can be included to account for the possible participation of the thin ice layer in the energy storage and release process.<sup>7,8,9</sup> This will be discussed later.

### III EXPERIMENTAL

#### A Traditional blister test specimen preparation

The specimen shown in Figure 1 was begun by preparing a cylinder about 130 mm in diameter and 50 mm high of the substrate of interest (metal or polymer). Commercially available rod stock of large diameter was convenient to use. A 6.3-mm ( $\frac{1}{4}$ -inch) diameter hole was drilled along the cylinder axes, clear through from top to bottom. At the bottom, threads were machined on the inside of the hole to accept a threaded brass fitting to which stainless steel tubing for gas delivery was attached. The top surface of the cylinder was polished to smoothness by 600-grit silicon carbide paper.

The cylinder of substrate material was prepared for the ice layer formation in the following way. Duct tape was wrapped around the outside of the cylinder so it extended above the top surface, forming a containing wall for water. The central hole was temporarily stoppered with a silicone-coated rod that was situated flush with top surface of the cylinder and protruded from the bottom surface for later removal. Finally, an initial crack, or debonded area, was formed on the top surface of the cylinder by spraying mold release with the aid of a circular stencil. An alternative way of forming the debonded area was to cut a circle of thin film, such as Teflon<sup>®</sup>, and center it and leave it on the cylinder's surface.

To form the ice layer itself, water near 0°C poured onto the cylinder surface (previously cooled to 0°C). Water depths ranging from 2.5 mm to 6.5 mm were tried. This assembly was then cooled to the test temperature. Prior to test, the central stopper was removed carefully by drawing it out through the bottom of the substrate cylinder.

A second method was also used to prepare the ice layer for the traditional blister test. In this method, a cylindrical disc of ice with a thickness in the range of 2.5 mm to 6.5 mm was prepared separately in a mold. This disc was placed onto the top surface of the substrate cylinder while the latter was at a temperature just above 0°C. Contact with the warmer substrate surface caused the prepared ice disc to melt slightly and fuse to the substrate cylinder. This assembly was then immediately chilled to the desired test temperature.

For the second method of ice layer formation, the debond radius was established in a different way from that for the first method. The central hole of the substrate cylinder was remachined with a tapering enlargement to increase its radius at the top to that desired for the debonded area. That is, instead of providing a debonded ice-substrate interface by means of mold release or a release film, we removed the substrate material itself from beneath the ice sheet. An assortment of debond radii were created this way in different substrate cylinders. Keeping the same radius at the bottom of all the holes allowed the convenience of attaching the same tubing for the pressurizing gas to each specimen before test.

A necessary feature of the machined central hole used as the debonded area itself was that a small step, about a millimeter deep, was machined into the top edge. This, based on water's inability to round sharp corners easily, was intended to prevent any melted ice from flowing into the hole and forming an irregular crack tip on freezing.

### **B Inverted blister test specimen preparation**

The specimen shown in Figure 3 was begun by forming a cylinder of polymer or metal to be used as a base for the bimetals system of interest. Commercially available rod stock of 100 to 150 mm in diameter was convenient to use. A central hole about 6.3 mm in diameter was machined along the cylinder axis, from top to bottom, with a fitting attached at the bottom of the hole for the gas delivery line (see above). The central hole was widened at the top in a gradual taper so that its radius at the horizontal surface corresponded to the radius desired for the debonded area. A small step, about a millimeter deep, was machined into the top edge of the central hole. The step served as a barrier to water flow, making the ice boundary controllable and resulting in a reproducible crack tip. Several cylinders were prepared like this to provide an array of debonded areas.

To prepare the ice interlayer, the top surface of the cylindrical base, at a temperature just above 0°C, was covered with water. Typically, the water formed a layer a few millimeters thick, retaining itself in places at the edges of the horizontal surface by its own surface tension. A circular sheet of the substrate of interest (thickness in the range of 0.3 to 2 mm) was placed gently on top of the water layer and was pushed to within 0.20 mm of the surface of the base. This, of course, expelled some water into the central hole and out the exterior perimeter.

The water which had been expelled onto the machined step of the central hole was sucked up with very small diameter rubber tubing attached to the barrel of a

syringe. The presence of water to the brink of the step, but not down onto it, resulted in a uniform crack tip all around the radius of the debonded area.

The assembly of base, water, and substrate was immediately cooled to freeze the water, and was conditioned at the desired test temperature for at least 2 hours. The final result was a continuous sheet of the substrate of interest adhered on its underside to an ice layer 0.20 mm thick with a central hole, the ice layer in turn adhered to and supported by a thick base with a central hole.

### C Testing procedure

A schematic diagram of the test set-up is shown in Figure 4. All testing was conducted in the cold room at  $-20^{\circ}\text{C}$ . Nitrogen gas was used to pressurize the specimen, the flow being controlled by a manually operated valve. Three pressure gages with different ranges were mounted on line, so that the appropriate gage for each specimen could be activated as needed. The gages were calibrated so that the high and low ends of their ranges overlapped. Pressurization rate, controlled manually, was about 14 kPa/sec (2 psi/sec). The critical pressure, as well as the initial debond radius, the ice thickness, and substrate sheet thickness for the inverted blister test, were recorded for each specimen. After failure, which was always catastrophic, both fracture surfaces were examined to determine locus of failure.

### D Modulus determinations

For the inverted blister test, moduli of the substrate materials at the test temperature,  $-20^{\circ}\text{C}$ , had to be obtained for use in the analysis of results. To do this, strips 12.7 mm wide and 127 mm long were cut from the same sheet material used for the blister specimen. These specimens were mounted in an Instron universal test machine with toothed wedge grips, leaving a gage length of 76 mm between the grips. The test machine was fitted with a temperature chamber controlled to  $-20 \pm 1^{\circ}\text{C}$  with liquid nitrogen as coolant. Tensile loading was conducted at a crosshead displacement rate of 6 mm/min. Moduli were computed from the load *versus* displacement trace at low displacements.

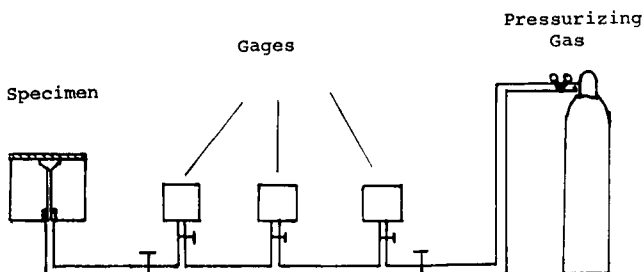


FIGURE 4 Schematic diagram of the test set-up. The compressed pressurizing gas is connected by metal tubing to the specimen. The pressurization of the specimen is controlled by a manually operated valve, and pressure is monitored by a gage selected from those mounted in the line near the specimen.



### VI RESULTS AND DISCUSSION

#### A. Comparison of traditional with inverted blister test results

As already stated, we first used the traditionally configured blister test, with the ice as a continuous sheet surmounting the substrate of interest. No interfacial failures whatever were obtained for ice on an aluminum substrate. For ice on a polymethyl methacrylate (PMMA) substrate, about 60% of the specimens gave interfacial failure. By making and testing excessive numbers of ice-PMMA specimens, we could accumulate a sufficient number of interfacial failures to construct a plot of  $P_c$  versus  $\sqrt{(f(h/c)/c)}$  for the traditional blister test. The plot is shown in Figure 5 (top). In the inverted blister configuration, with the

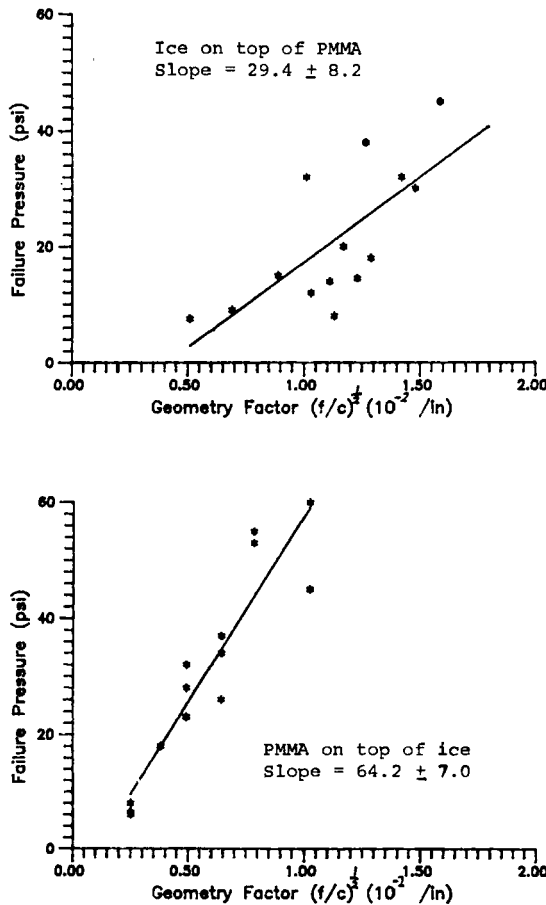


FIGURE 5 Comparison of plots of critical pressure versus geometry factor for traditional and for inverted blister test configurations. Data from specimens with ice as the continuous sheet on top of PMMA substrate show high scatter (top). Data from specimens with PMMA substrate as continuous sheet on top of ice interlayer form a good straight line with low scatter (bottom). Slopes of best fit lines are stated  $\pm 1$  std. dev.

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substrate as a continuous sheet on top of the ice, all of the specimens tested gave complete interfacial failure. The plot of this data is also shown in Figure 5 (bottom).

For both plots in Figure 5, the straight lines through the data can be extrapolated to zero within experimental error. The data are expected to pass through zero, since approaching zero on the  $x$ -axis corresponds to larger and larger initial crack size, which in turn results in lower and lower critical pressure. The important comparison is between the scatter in the two plots. (The slopes are not expected to be the same.) The scatter in the traditional blister test data is so large that the straight line relation cannot be determined with high confidence. It is not likely that this scatter can be reduced since it originates in the inherent nonuniformity in thickness of ice formed by freezing ponded water. The scatter in the inverted blister test data is lower, allowing the value for  $F$  to be computed from the slope with greater precision.

### **B. Influence of support material on the blister test result**

The energy balance analysis presented in the BACKGROUND section contained the assumption that only the continuous top sheet deforms and is, therefore, the only material in the specimen whose elastic properties influence the test result. We wished to make sure that this assumption could be trusted, and that the properties of the large block of support material underlying the ice in the inverted blister test configuration did not influence the test result. To do this, we carried out the blister test using two different substances—PMMA and aluminum—as the support material for a single ice-substrate system. The results are shown in Figure 6. The slopes of the straight lines in the two plots are within 7% of each other, indicating negligible influence of support material on the test result. When the value of  $F$  is computed from the slope (see Eq. 4), the difference almost vanishes because the square root is taken.

### **C. Role of ice as an interlayer instead of as a thick slab**

Normally, in a blister test for a bimaterials system, the material regarded as the substrate is in the form of a thick block with a central hole and the material regarded as the adhesive is in the form of a thin continuous sheet on top. In the inverted configuration just described, the “substrate” on top deforms in response to pressurization while the ice layer underneath is presumed not to deform because it is adhered to the large inert support block. Perhaps the possibility should be examined that the thin interlayer of ice does deform slightly near the crack tip as the continuous top sheet of “substrate” deforms in response to pressurization. Significant ice layer deformation would influence the test result, and ice's elastic properties would have to be used in the analysis in order to extract the correct value for  $F$ .

Fortunately, Williams<sup>7,8</sup> and Burton *et al.*<sup>9</sup> have already analysed the configuration of a one-dimensional strip blister, where a thin interlayer of material

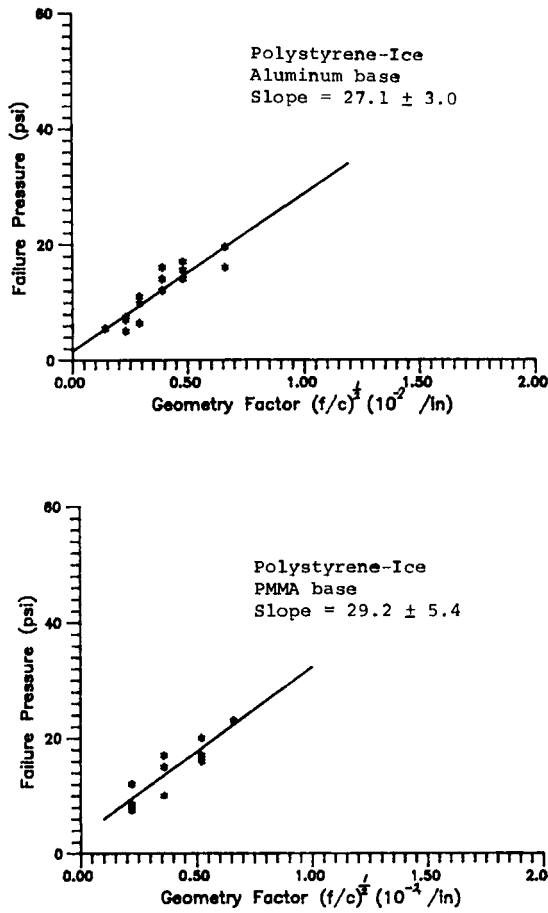


FIGURE 6 Comparison of plots of critical pressure versus geometry factor for inverted blister test with two different base materials. For the ice-polystyrene system, both aluminum (top) and PMMA (bottom) bases give the same results. Slopes of best fit lines are stated ±1 std. dev.

joins a continuous top sheet to an underlying block of material. We can use their analysis to evaluate whether or not we need to be concerned with the effect of the ice interlayer properties on our test result. Their analysis pertained to crack propagation at the interface between the top sheet and the interlayer, and incorporated the elastic properties of both of those substances. Their final equation is:

$$P_c^2 = \frac{3}{2} \left(\frac{h}{c}\right)^3 \frac{EF}{c(1 + \nu^2)} \left[ 1 - 4 \left\{ \frac{1}{3} \left(\frac{h}{c}\right)^3 \frac{E}{kc} \right\}^{1/4} \right] \tag{5}$$

where

$$k = \frac{1 - \nu'}{(1 - 2\nu')(1 + \nu')} \frac{E'}{h'}$$

The primed quantities are for the ice interlayer and the unprimed quantities are for the top sheet. The properties of the ice interlayer are contained within  $k$ , which in turn is contained within the second term in brackets, denoted  $Q$  for convenience. It is the effect of ice properties on  $Q$  that is of real interest, since  $Q$  is subtracted from 1 in Eq. (5). For the effects of the interlayer's bulk properties to be negligible,  $Q$  must be small compared with 1, a situation realized when the interlayer thickness goes to zero or when the ratio of moduli  $E/E'$  becomes very small.

The size of  $Q$  with respect to 1 can be easily estimated. Table I shows computed  $Q$  values for two different ice interlayer thicknesses, bonded to both high modulus (steel) and low modulus (Teflon<sup>®</sup>) substrates. The Table shows that even for the higher modulus substrate and thicker interlayer,  $Q$  remains much less than 1. Thus, ice used as a thin interlayer influences of  $P_c^2$  less than 10%. When the square root is taken in the process of computing  $F$ , the influence is even smaller. As an additional note of caution, we point out that, according to Eq. (5), large values of  $h$  combined with very small values of  $c$  could cause  $Q$  to become large enough to be of concern. We recommend that  $h/c$  be kept sufficiently small to avoid this.

#### D. Influence of substrate thickness

According to theory,  $P_c$  is a function of the ratio  $h/c$ , not of the absolute value of the thickness  $h$  of the continuous top sheet. To make sure that the test system behavior was consistent with theory, we conducted two test series, each with a different setting of  $h$ . The selected settings for  $h$  were 0.30 mm and 0.38 mm as per available sheet thicknesses. Variation of  $\sqrt{(f(h/c)/c)}$  was achieved at constant  $h$  by changing  $c$ . The various combinations of  $h$  and  $c$  provided geometry factor values that ranged over nearly two orders of magnitude. The results for the two series are plotted in Figure 7.

TABLE I  
Estimated values of  $Q$  for different substrates and ice interlayer thicknesses

Substrate	Steel		Teflon <sup>®</sup>	
Substrate modulus, GPa, measured at $-20^\circ\text{C}$	33.4		1.32	
Substrate Poisson's ratio	0.30		0.33	
Substrate thickness, mm	0.30		0.30	
Initial debond radius, mm	12.7		12.7	
Ice modulus, GPa, value at $-20^\circ\text{C}$ *	9.5		9.5	
Ice Poisson's ratio*	0.35		0.35	
Ice thickness, mm:	0.10	0.30	0.10	0.30
$Q$	0.050	0.066	0.078	0.103

\*Values from Ref. 10.

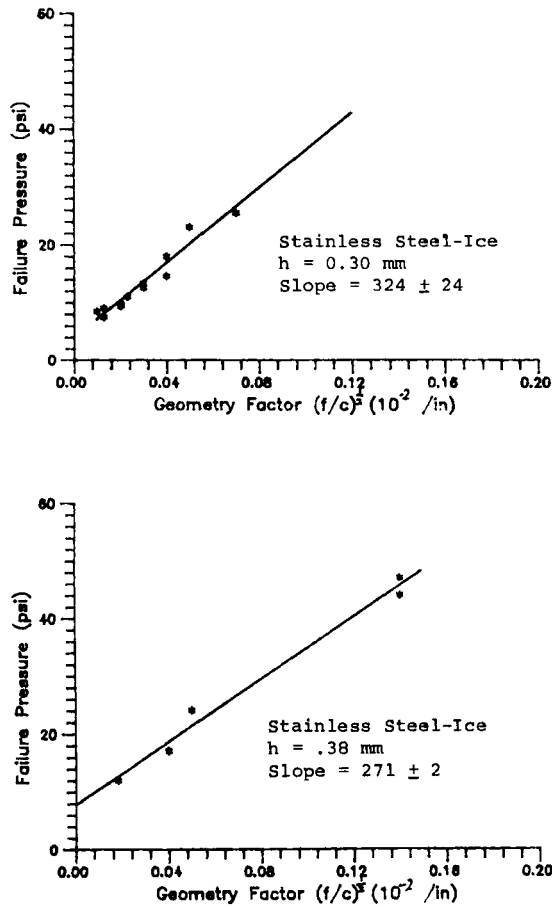


FIGURE 7 Comparison of plots of critical pressure versus geometry factor for inverted blister test with two different thicknesses of substrate. Slopes for stainless steel substrates 0.30 mm and 0.38 mm thick are within 15% of each other. When the square root is taken to compute  $F$ , the difference becomes even smaller, indicating no undesirable effect of thickness. Slopes of best fit lines are stated  $\pm 1$  std. dev.

The plots for the two substrate thicknesses are linear and have slopes that are within 15% of each other. This modest difference in slope becomes almost negligible once the square root is taken and  $F$  is computed. The fact that the data points for both series fit essentially the same line indicates that the test system conforms to theory over the dimensional values considered.

Thin membrane-like films that stretch rather than bend in response to pressurization were not of interest here, and for such cases another analysis is appropriate.<sup>11,12</sup> Presumably, if the  $h/c$  value became small enough, the test results would conform to a stretching membrane analysis, rather than to the analysis summarized by Eq. (4). At the other extreme, if  $h/c$  became large enough, the test results would conform to an analysis based solely on the near

field contribution to strain energy (Eq. (1)). The analysis for this case is also described in Ref. 11.

The values of  $h/c$  at which a specimen's behavior is dominated by near field contributions, bending deformation, or membrane stretching, respectively, would be expected to change slightly, depending on the elastic properties of the substrate sheet and the adhesive interlayer. Proper use of the blister test requires merely that one establish which analysis pertains to the specimen configuration at hand.

## V CONCLUSION

Up to now, evaluation of interfacial adhesion in ice-substrate systems has been a problem because of the fragility of the ice and its tendency to exhibit cohesive failure. A modified form of the blister test has been found very useful for evaluating interfacial failure in ice-substrate systems. To ensure success, the test must be arranged in the following way:

- 1) The nonfragile material, which can sustain deformation, must be used as the continuous sheet on the top.
- 2) The fragile material (ice) must be present as a thin interlayer, having a central hole, underneath the continuous sheet.
- 3) The fragile material must be supported underneath by an inert block of material, also having a central hole.

This arrangement, which can be thought of as inverted, gives reproducible results that conform to theory within the range of values considered. Failures are interfacial, and a value for a materials system parameter, the interfacial fracture energy, can be extracted from the results. The results are not influenced by extraneous factors such as the nature of the support material underlying the fragile material. While the usefulness of the test was demonstrated on ice systems, we feel it could be used successfully on any system where one of the constituents is very brittle.

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