Fracture Toughness of Adhesive Joints. I. Relationship between Strain Energy Release Rates in Three Different Fracture Modes and Adhesive Strengths

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SYNOPSIS

The three strain energy release rates, $G_{\rm IC}$, $G_{\rm IIC}$, and $G_{\rm IIIC}$, of adhesive joints can be attributed to their ability to resist crack propagation of solids in the adhesive layer. The dependencies of $G_{\rm IC}$, $G_{\rm IIC}$, and $G_{\rm IIIC}$ on crack lengths for various adhesive joints were determined using the double-cantilever beam specimen by a compliance method. The two types of adhesive strengths, i.e., adhesive tensile strength and adhesive shear strength, corresponding to $G_{\rm IC}$ and $G_{\rm IIC}$, respectively, were carried out at room temperature and 65% RH with a crosshead speed of 10 mm/min. The $G_{\rm IC}$, $G_{\rm IIC}$, and $G_{\rm IIIC}$ were dependent upon crack length and had constant values irrespective of geometric parameters of the specimen over the crack length of five times adherend thickness, 0.65 (= crack length over half a length of span) and eight times adherend thickness, respectively. In the region of the crack length, we determined the following increasing order of fracture toughness: $G_{\rm IC} < G_{\rm IIC}$. A positive correlation was found between adhesive tensile strength and $G_{\rm IC}$. A significant relation between adhesive shear strength and $G_{\rm IIC}$ was not found in this work. Further studies are needed to clarify the relation between adhesive shear strength and $G_{\rm IIC}$ with general adhesives. © 1994 John Wiley & Sons, Inc.

INTRODUCTION

When an external force is applied to adhesive joints, usually stress is extremely concentrated at the crack tip of the adhesive joints, and failure of the adhesive joints occurs as the force comes to a critical value. To understand adhesive fracture behavior, one needs to recognize that an adhesive layer contains flaws, surface roughness, and microvoids, which are introduced during the gluing process employed. Adhesive strength is typically defined as a simple value calculated by dividing a critical external force at failure of an adhesive joint by its gluing area. It is not, however, apparent what adhesive strength is not well defined because adhesive joints generally contain a flaw, usually considered as an elliptical crack, and

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they propagate under stress. Hence, it is necessary to apply fracture mechanics to adhesive joint failure.

In fracture mechanics, two parameters, the stress intensity factor (K) and the strain energy release rate (G), are used to explain the initiation and propagation process of the crack inherently present in joints. When K and G exceed some critical values, K_C and G_C , respectively, the crack in an adhesive joint propagates. K_C , often referred to as the fracture toughness, and G_c , the strain energy release rate, provide a measure of the energy required to extend a crack over a unit area by concentrating stress at the crack tip. K_C and G_C are the material constants. The energy criteria for adhesive fracture will be adopted. We conducted fracture testing of adhesive joints with various adhesives for wood. Fracture mechanical approaches for failure of adhesive joints have been done; the effects of geometry of adhesive joints by Takatani et al.,¹ the effects of moduli and bonding thickness on adhesive fracture energy by Ouezdou and Chudnovsky,² Chai,^{3,4} Kinloch and

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Shaw,⁵ Mostovoy and Ripling,^{6,7} and R. Ebewele et al.¹³; the effects of time and temperature on Mode I fracture by Kobayashi et al.,^{8,9} Bascom et al.,¹⁰ and Binter et al.¹¹; and the effects of environmental attack on adhesive joints by Cognard.¹²

A crack tip in an adhesive joint may be stressed in three different models: I, II, and III. In many cases, fracture of an adhesive joint by mode I has been considered, but there are few studies on other fracture modes. In practice, it is necessary to consider a comprehensive evaluation of all three different fracture modes for adhesive joints.

For this article, dependencies of strain energy release rates on crack length were studied, and an optimum range of crack length, in which the strain energy release rates are almost independent of the geometry of adhesive joints, was determined. Finally, the relationship between the strain energy release rates and their corresponding adhesive strengths in wood-adhesive joints are considered.

THEORY

Since a crack-tip front in an adhesive joint is stressed by an external force from various directions, there is a need to consider fracture behavior in the three different fracture modes, i.e., opening mode (mode I), plane-shear mode (mode II), and tearingshear mode (mode III).

The fracture resistance of adhesive joints was represented by the strain energy release rate (G_C) , which is the critical value required to extend a crack over a unit area along a bond line by stress concentration. Based on linear elastic fracture mechanics, the displacement, δ , is given by

$$\delta = C \cdot P \tag{1}$$

where C and P are the compliance of a specimen and the load applied. In a fixed-loading condition, variation of displacement, $\Delta \delta$ is related to that of the variation of a crack length, ΔA , as follows:

$$\Delta \delta = P(\partial C/\partial A) \Delta A \tag{2}$$

The strain energy, U, at failure is $U = (1/2)P_C\Delta\delta$, which defines the strain energy release rate, G_C , as follows:

$$U = \frac{1}{2} P \Delta \delta = G_C b \cdot \Delta A \tag{3}$$

where b and P_C represent the width of the specimen

and failure load. From eqs. (2) and (3), G_C is given by

$$G_C = \frac{P_C^2}{2b} \cdot \frac{\partial C}{\partial A} \tag{4}$$

In the case of adhesive joints, early work done by Mostovoy and Ripling^{6,7} using a tapered double cantilever beam for structural adhesives. From the formulas of the strength of materials for two cantilever beams with uniform thickness, h, Young's modulus, E, and a span that is equal to the crack length, A, dC/dA is given by the following reliable relation:

$$\frac{\partial C}{\partial A} = \frac{8}{Eb} \left[\frac{3A^2}{h^3} + \frac{1}{h} \right]$$
(5)

The strain energy release rate, G_C , for adhesive joints, thus, can be represented as follows:

$$G_C = \frac{4P_C^2}{b^2 E} \left[\frac{3A^2}{h^3} + \frac{1}{h} \right]$$
(6)

where the term in parentheses is related to the geometry of the specimen.

In this article, the strain energy release rate of adhesive joints will be determined for various crack lengths by eq. (6). G_C was almost constant irrespective of geometry of the specimen within experimental conditions, and G_C , thus determined, was taken as a reasonable fracture energy for adhesive fracture joints. Finally, the relation between the strain energy release rates in three different fracture modes and their corresponding adhesive strengths was considered.

EXPERIMENTAL

Materials

Various commercially available adhesives were used as glues and are shown in Table I. Kaba, Japanese Birch (*Betula maximowicziana* Regel), was used as the adherend for which physical properties are summarized in Table II.

Measurement of Dynamic Mechanical Properties of Films

The films used were prepared by casting the polymer on a Teflon sheet and cured, if necessary, at room

Adhesives	Composition	<i>T</i> (<i>E</i> " max) (°C, Cured)	<i>E'</i> at 21°C (dyne/cm²)	<i>E</i> " at 21°C (dyne/cm²)	Tan δ at 21°C
AVUT	Water-based vinyl polymer– isocyanates H-3ª	5	$1.0 imes10^{10}$	$7.5 imes10^{8}$	0.075
EP001	Epoxy polyamines ^a	-54.7	$1.54 imes10^8$	$4.34 imes10^7$	0.26
EP 007	Epoxy polyamides ^a	64.6	$1.0 imes10^{10}$	$1.00 imes10^7$	0.0382
EC34569	Epoxy polyamines ^a	84	$1.12 imes10^{10}$	$3.42 imes10^{8}$	0.0305
\mathbf{EsetR}	Epoxy polyamides ^a	55.5	$1.16 imes10^{10}$	$3.03 imes10^8$	0.026
PM200	Epoxy silicon ^a	-51.3	$4.65 imes10^8$	$5.81 imes10^7$	0.125
KU224	Polyurethanes	21	$1.04 imes10^{10}$	$6.23 imes10^{8}$	0.06
KU661/2	Polyester (polyol) polyisocyanates ^a	46.3	$1.01 imes10^{10}$	$2.99 imes10^8$	0.027
CH18	Polyacetates	27.3	$1.08 imes10^{10}$	$2.64 imes10^9$	0.245
Y400	Polyacrylates polyacrylates ^a	72.2	$1.18 imes10^{10}$	$1.13 imes10^9$	0.096
SGA	Polyacrylates polyamines ^a	-11.3	$6.06 imes10^9$	$7.58 imes10^8$	0.125
Α-α	$Poly(\alpha$ -cyano acrylates) ^b				
3000DXH	$Poly(\alpha$ -cyano acrylates) ^b				

Table I Dynamic Viscoelastic Properties of Various Adhesives Used

^a Curing agents.

^b Not succeeded in measuring dynamic viscoelastic properties.

temperature and 65% RH for 7 days. Dynamic mechanical properties of the films were measured using a Rheovibron DDV-II (Toyo Baldwin Co.) at 110 Hz with average heating rate of 1°C/min.

Adhesive Fracture Test

The adhesion procedure was carried out with a woodadherend with a grain angle of 5°, which is the angle necessary to prevent wood-failure along the grain prior to fracture. Dimensions of the specimens are shown in Figures 1–3. The amount of adhesive employed was $250-300 \text{ g/cm}^2$. The specimens were pressed under about 10 kg/cm² in all cases and were kept at 20°C, 65% RH, for about 4 days for curing.

Adhesive fracture tests were carried out at 21°C with a crosshead speed of 2 mm/min using a Tensilon (Orientec Co.) for the three types of fracture specimens. Each testing was carried out with three specimens. The strain energy release rates were calculated by eq. (6) using the failure load (P_C) shown in Figure 4.

Adhesive Strength Test

The measurements of adhesive tensile strength and adhesive shear strength were carried out at room temperature with a crosshead speed of 10 mm/min using the Tensilon. Seven specimens were tested for each condition. The geometry of the specimens is shown in Figure 5.

RESULTS AND DISCUSSION

Generally, adhesive joint strengths are dependent to a great extent on the mechanical properties of adhesive polymers. Because adhesive polymers are

Table II	Characteristics of Adherends
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	Specific Gravity		Moisture	Young's Modulus (10 ⁵ kgf/cm ²)	
Adherends	Air	Dry	(%)	$E_{\mathrm{I}}, E_{\mathrm{II}}$	E_{III}
Kaba1 Kaba2	0.68 0.88	0.64 0.78	14.8–16.5 14.9	1.38 1.16	$\begin{array}{c} 1.34 \\ 1.12 \end{array}$

Kaba1 was used for AVUT, EP001 and EP007. Kaba2 was used for the other adhesives. E_1 and E_{II} were Young's moduli of adherends in mode I and mode II, respectively. E_{III} was Young's modulus of adherend in mode III.



Figure 1 Adhesive joint specimen for mode I fracture test.

viscoelastic materials, the mechanical properties of polymer then change as a function of temperature, frequency, or loading rate. It is necessary to know the dynamic mechanical properties of the polymers in order to recognize the viscoelastic fracture behavior of adhesive joints. Dynamic mechanical properties of various adhesive films provide information on molecular movements of polymer chains over a wide range of temperatures.

In Table I, the temperature of E''_{\max} , the tensile storage modulus (E'), the tensile loss modulus (E''), and loss tangent $(\tan \delta)$ at 21°C of films are summarized. The adhesives for which the temperature of E''_{\max} is over 21°C were in a glassy state, and those for which the temperature of E''_{\max} is below 21°C were in a rubbery state under the testing conditions. Deformation of adhesive polymer is closely related to micro-Brownian motions of the segments. The adhesives in rubbery and fluid states can be deformed easily by external force. On the other hand, adhesives in the glassy state are hardly deformed by external force.

The crack length dependencies of G_{IC} , G_{IIC} , and G_{IIIC} in three different fracture modes are shown in Figures 6–8, respectively. Figure 6 shows that the value of the G_{IC} of adhesive joints with the epoxy resins gradually increased as a function of crack length. In the region where A is larger than 6 cm,



Figure 2 Adhesive joint specimen for mode II fracture test.



Figure 3 Adhesive joint specimen for mode III fracture test.

i.e., where A/h is larger than 6, G_{IC} became constant and was not affected by geometric parameters of the specimen. Similar behavior of the crack length de-



Figure 4 Load-deflection curves for three fracture modes.



Figure 5 Dimensions of specimens for adhesive tests.

pendence of G_{IC} could be found in other adhesives joints.

The $G_{\rm IIC}$ of epoxy-wood joints decreased with increasing crack length and had constant values over A/L of 0.6, where L is half the length of the span supported by the grids. The change of $G_{\rm IIC}$ in the glassy state was considerable, but $G_{\rm IIC}$ in the rubbery state did not change much, as shown in Figure 7. For other adhesive joints, the same crack-length dependence of $G_{\rm IIC}$ could be found.

In mode III, G_{IIIC} increased as crack length increased up to 8 cm, i.e., eight times adherend thickness, and reached a plateau over this crack length,



Figure 7 Strain energy release rate, G_{IIC} , as a function of crack length, A/L, for constant crosshead speed and temperature.

where the value of $G_{\rm IIIC}$ was not affected by geometric parameters of substrates, as shown in Figure 8. We found that the crack-length dependencies of $G_{\rm IIIC}$ were similar to other adhesive joints.

The crack-length dependence of G_{IC} , G_{IIC} , and G_{IIIC} described above could be clarified from the failure load (P_C) vs. crack length (A) curves as shown in Figure 9. P_C decreased considerably as A increased from 0 to about 6 cm, i.e., six times adherend thickness, in mode I and from 0 to about 7 cm, i.e., seven



Figure 6 Strain energy release rate, G_{IC} , as a function of crack length, A, for constant crosshead speed and temperature.



Figure 8 Strain energy release rate, G_{IIIC} , as a function of crack length, A, for constant crosshead speed and temperature.



Figure 9 Failure load, P_c , as a function of crack length for three fracture modes.

times adherend thickness, in mode III. In mode II, P_C decreased with an increment of A/L in the range of 0 and 0.6. Over these crack-lengths, P_C did not change much with increasing crack length. Within the specimen size range in this study, eq. (6) showed that the strain energy release rate was strongly dependent on the failure load. The strain energy release rate was considered to be a constant value over the region of crack lengths described above, because

Table IIICritical Strain Energy Release Ratesfor Modes I, II, and III

	G_{IC}	G_{11C}	G_{IIIC}			
Adhesives	(kgf cm/cm ²)			G_{IIC}/G_{IC}	$G_{\rm IIIC}/G_{\rm IC}$	
AVUT	0.34	2.05	0.93	6.0	2.7	
EP007	0.28	2.77	0.72	9.9	2.6	
EP001	0.18	2.15	0.52	11.9	2.9	
PM200	0.34	2.80	0.51	8.1	1.5	
\mathbf{EsetR}	0.19	2.57	0.69	13.3	3.6	
EC3569	0.39	5.55	2.53	14.2	6.5	
KU224	0.10	1.72	0.47	16.6	4.5	
KU661/2	0.20	3.71	0.80	19.0	4.1	
CH18	0.24	2.93	0.82	12.3	3.4	
Y400	0.07	2.11	0.76	30.4	10.9	
SGA	0.24	2.54	0.50	10.8	2.1	
Α-α	0.10	9.47	0.83	54.8	8.6	
3000DHX	0.11	4.06	1.36	38.3	12.9	

the P_C vs. A curve has a plateau in the same region. Similar results were reported by Takatani et al.¹ They reported that the principal stress at the crack tip was not changed in the range of $A \ge 5$ h by finite element analysis of stress for mode I joints.

In the plateau region, where the values are not affected by geometric parameters of specimens, $G_{\rm IC}$, $G_{\rm IIC}$, and $G_{\rm IIIC}$ may be regarded as fracture toughness measures for each adhesive joint. In Table III, $G_{\rm IC}$, $G_{\rm IIC}$, and $G_{\rm IIIC}$ average values, estimated from the



Figure 10 Relationship between adhesive tensile strength and strain energy release rate, G_{IC} , for various adhesives.

plateau region, are summarized. Since, in practice, adhesive joints are often exposed to various environmental conditions and usually subjected to external forces from various directions, it is necessary to obtain information on overall fracture toughness for each adhesive joint in order to design a stable structure.

We determined the following increasing order of fracture toughness measures: $G_{IC} < G_{IIIC} < G_{IIC}$, G_{IIC} / G_{IC} is generally between 10 and 50 and G_{IIIC} / G_{IC} is between 2 and 10. Similar results were found by Chai⁴ and Liechti and Freda.¹⁴

In fracture behavior, testing of adhesive tensile strength is similar to that of the tensile-opening mode (mode I). If parameters except for failure load, P_{C} , are constant under the experimental conditions, $G_{\rm IC}$ is represented as a function of failure load. As it is expected that P_c has a positive correlation against adhesive tensile strength, adhesive tensile strength was plotted vs. G_{IC} in Figure 10. It is evident that there is a positive correlation between adhesive tensile strength and the square root of G_{IC} , but the correlation coefficient, r, was not very high. Testing of adhesive shear strength is similar to that of the plane-shear fracture mode (mode II). It was difficult to find a good correlation between adhesive shear strength and the square root of G_{IIC} in this work (Fig. 11). Lower values of r may be due to the following two facts: One is that various adhesives consisting of different components and materials with different molecular structure have different me-



Figure 11 Relationship between adhesive shear strength and strain energy release rate, G_{IIC} , for various adhesives.

chanical properties, and the other is that there could be a difference in strain rate at the crack-tip between adhesive tests and adhesive fracture tests.

CONCLUSIONS

- 1. G_{IC} , G_{IIC} , and G_{IIIC} had constant values irrespective of geometric parameters of the specimen over the crack length of five times adherend thickness, 0.65 (= crack length over half a length of the span), and eight times adherend thickness, respectively. The strain energy release rates in these regions must be regarded as a reasonable measure of fracture toughness for the three different fracture modes.
- 2. For all adhesive joints, strain energy release rates showed the following increasing order of fracture toughness: $G_{IC} < G_{IIIC} < G_{IIC}$. Also, G_{IIC}/G_{IC} and G_{IIIC}/G_{IC} were between 10 and 50 and between 2 and 10, respectively.
- 3. A positive correlation was found between adhesive tensile strength and the square root of $G_{\rm IC}$ with a correlation coefficient of 0.6.
- 4. We could not find a significant correlation between adhesive shear strength and the square root of G_{HC} .

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