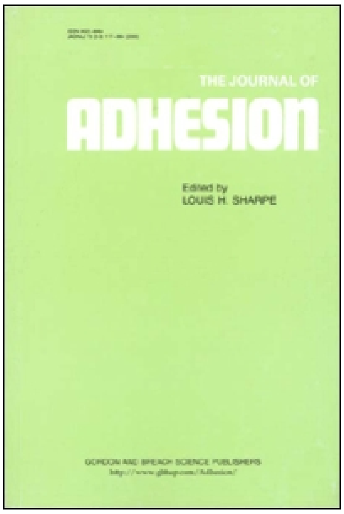
This article was downloaded by:
On: *22 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



The Journal of Adhesion

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

Fracture Evaluation of a Thermoplastic Polyimidesulfone as Adhesive

S. Mall[a]
[a] Department of Engineering Mechanics, University of Missouri, Rolla, MO, U.S.A.

To cite this Article Mall, S.(1987) 'Fracture Evaluation of a Thermoplastic Polyimidesulfone as Adhesive', The Journal of Adhesion, 20: 4, 251 — 260

To link to this Article: DOI: 10.1080/00218468708074945

URL: http://dx.doi.org/10.1080/00218468708074945

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

J. Adhesion, 1987, Vol. 20, pp. 251–260

Printed in the United Kingdom

Fracture Evaluation of a Thermoplastic Polyimidesulfone as Adhesive

S. MALL

*Department of Engineering Mechanics, University of Missouri,
Rolla, MO 65401, U.S.A.*

(*Received December 23, 1985; in final form March 24, 1986*)

An experimental study was undertaken to evaluate the fracture strength of a thermoplastic polyimidesulfone as adhesive. This study involved the measurement of critical fracture energy, G_{IC}, as a function of its molecular weight and temperature. The molecular weight was varied by endcapping with phthalic anhydride. The double cantilever beam specimens consisting of two bonded titanium adherends (Ti-6Al-4V) were tested. There was a considerable loss of fracture energy as molecular weight decreased beyond a certain value. Furthermore, the fracture energy decreased with increasing temperature.

KEY WORDS Adhesive; fracture; molecular weight; temperature; thermoplastic polyimidesulfone; titanium adherends.

INTRODUCTION

The trend in the aircraft industry has been towards stronger, lighter and more efficient structures. Adhesive joints have been proven useful and have helped toward achieving this goal. In fact, the high strength-to-weight ratio of adhesively bonded joints has led to their application in primary fuselage structures.[1] However, to design adhesive bonded structures with reliability, there is a need to develop the suitable failure criteria for evaluating their strength, integrity and durability. Their failure mode is characterized by flaw growth and progressive crack propagation. The flaws that may be anticipated include internal cracks, surface cuts, and microvoids inherently present as a result of the processing methods employed. Further, stress singularity may exist at a bond termination in the absence of flaws. Thus, an appropriate failure criterion must be based on fracture mechanics principles.[2,3] This is simply an extension of Griffith's hypothesis.[4] It supposes that fracture occurs when sufficient energy is released from the stress field to generate new fracture surfaces at the instant of crack propagation. This strain energy release rate provides a measure of the energy required to extend a crack over a unit area and is termed the fracture energy.

This paper presents the details and results of a study to evaluate the fracture energy in the opening mode for a thermoplastic polyimidesulfone as adhesive since this property has been widely recognized as the appropriate criterion for bonded joint failure. This study involved the experimental investigation of effects of temperature and endcapping with varying concentrations of phthalic anhydride on its fracture energy.

EXPERIMENTAL

Polyimidesulfone

Polyimidesulfone ($PISO_2$) is a very promising, high temperature polymer, developed at NASA Langley Research Center, Virginia, which has the excellent thermoplastic properties generally associated with polysulfone, and the solvent resistance and thermal stability of aromatic polyimides.[5] It is a candidate for several aerospace applications. It was evaluated as an adhesive in the present study. $PISO_2$ is a linear aromatic polymer which may contain free amine and anhydride or acid end groups. These end groups are possible moisture pickup sites. The molecular weight of the polymer may also be modified by the introduction of appropriate end caps on to the $PISO_2$ chain. The use of endcapping to modify the molecular weight of a linear polymer is a well known technique. The effects of various types of end caps on its thermomechanical and related properties have been investigated.[6] In the present study, the effect of endcapping the $PISO_2$ with the varying

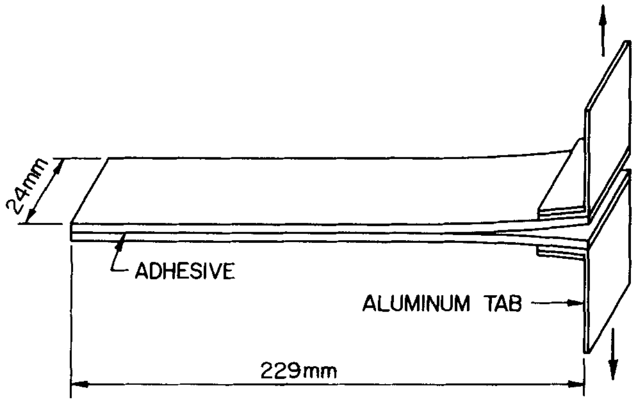


FIGURE 1 Double cantilever beam specimen.

concentration of phthalic anhydride on its fracture strength in a bonded joint was investigated.

Specimen preparation

The double cantilever beam (DCB) specimen, shown in Figure 1, was used to evaluate the fracture strength under opening mode I loading. The DCB specimen consisted of two bonded titanium adherends (Ti-6Al-4V) with an initial debond length of 38 mm. This debond was introduced by a Teflon film of thickness equal to the adhesive bondline. Each adherend was obtained by bonding two 25.4 mm wide, 1.27 mm thick and 229 m long titanium alloy strips. Two 0.5 mm thick aluminum end tabs were bonded to the DCB specimen, along with two 1.3 mm thick aluminum reinforcing plates. The peeling load was applied through these tabs.

An adhesive containing a scrim cloth was used to bond the adherends. The details of preparation of adhesive scrim cloth is given in Reference 5. It was a glass cloth with a nominal thickness of 0.1 mm and was used as a carrier for the adhesive as well as for bondline thickness control. The nominal bondline thickness was 0.25 mm. The titanium adherends (Ti-6Al-4V) to be bonded were grit-blasted with 120 mesh aluminum oxide and treated with Pasa

Jell 107† in order to form a stable oxide on the surface. A primer coating of the polyamide acid solution was applied to the adherends and they were thermally treated for one hour at 100°C and one hour at 200°C. The specimens were bonded as follows:

1 RT to 325°C/min at 7°C/min, apply 1.38 MPa at 280°C
2 Hold 15 min at 325°C
3 Cool under pressure

All specimens were prepared at NASA Langley Research Center.

Testing procedure

All tests of DCB specimens were performed in a displacement controlled test machine (Instron). Both edges of the specimen were coated with a white brittle fluid, to aid in visually locating the debond tip. Fine visible marks were put on these edges, at 1 mm intervals, to aid in measuring the debond length. The debond length was measured visually on both sides with two mircroscopes having a magnification factor of 20. Prior to testing, these specimens were fatigued to create a debond of at least 6 mm beyond the end of the Teflon film. The static test involved the application of displacement at a slow crosshead speed (10 mm/min). The load corresponding to the applied displacement was also recorded. When the load reached the critical value, the debond grew. The onset of growth resulted in a deviation from linearity in the load *versus* crosshead displacement record. The applied displacement was then decreased until a zero load reading was observed. After each static test, the specimen was fatigued until the debond grew at least 6 mm further, thus forming a sharp crack for the next static test. A series of static tests was performed on each specimen, which provided compliance and critical load measurements at several debond lengths. These measurements provided the critical strain energy release rate, as explained in the section entitled ANALYSIS.

Fracture tests were performed at the ambient temperature, at −73°C, and at 149°C. For low- and high-temperature tests, the specimen was mounted in the Instron test machine with an environmental chamber and heated or cooled from room temperature to the test temperature at a rate of 2°C/min. Then, the

---

† Trade name for a titanium surface treatment available from Semco, Glendale, CA.

temperature was held for at least thirty minutes to ensure thermal equilibrium before the specimen was tested.

Debond surfaces

All specimens in the present study failed in the cohesive manner. The bondlines were examined after the fracture test, and there was no evidence of voids in any bond.

ANALYSIS

Fracture tests on DCB specimens, conducted as described above, provided the critical load, P_{cr}, and the compliance, C, for each debonded length. The measured values of P_{cr} and C were used with linear beam theory to compute the fracture toughness, G_{IC}. The details of this procedure are elaborated by Wilkins, *et al.*[7] A brief description of the Wilkins, *et al.*, technique is given below. Figure 2 shows the variation of compliance, C, with the debond length, a, in a typical DCB specimen. A compliance relation of

$$C = A_1 a^3 \tag{1}$$

was fit through the experimental data points by the method of least squares as shown in Figure 2 by the solid line. This relation, based on linear beam theory, fits very well with the experimental data.

Figure 3 shows the measured critical load as a function of debond length for a typical specimen. Based on the linear beam theory[7], a relation between the critical load, P_{cr}, and the debond length, a, is

$$P_{cr} = A_2/a \tag{2}$$

A solid line shown in Figure 3 with a slope of -1 was fit to the experimental data with the method of least squares. Then, the averaged value of G_{IC} for each specimen was computed from the relation:

$$G_{IC} = \frac{P_{cr}^2}{2w}\frac{\partial C}{\partial a} = 3A_1A_2^2/(2w) \tag{3}$$

where w is the specimen width.

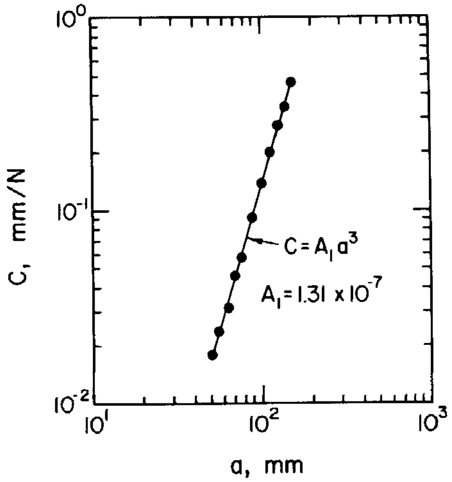


FIGURE 2 Typical relation between compliance, C, and debond length, a.

RESULTS AND DISCUSSIONS

The fracture test results are given in Table I and II. Table I shows the fracture energy, G_{IC}, of the bonded joint with polyimidesulfone, as a function of phthalic anhydride end-cap concentration. Also, Table I presents the molecular weight of polyimidesulfone with

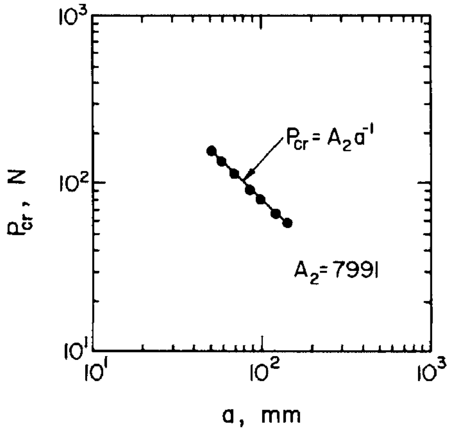


FIGURE 3 Typical relation between critical load, P_{cr}, and debond length, a.

TABLE I
Adhesive properties of polyimidesulfone ($PISO_2$) as a function of phthalic anhydride (PA) end-cap concentration

PA concentration percent	Molecular[a] weight (amu)	Tg[a] (°C)	Lap shear strength[a] (MPa)	Fracture energy[b] G_{IC} (J/m^2)
0	19,700	272	32	652
0.5	19,300	275	27	—
1.0	18,800	277	—	660
2.0	18,600	277	25	—
3.0	17,400	275	—	223

[a] From Ref. 6.
[b] Each data point represents the average of three specimens.

different phthalic anhydride concentration. In a previous study[6], the effect of endcapping of $PISO_2$ with varying concentrations (*i.e.*, 0, 0.5 and 2 percent) of phthalic anhydride on its adhesive properties was studied by measuring the lap shear strength from titanium lap shear specimens. These lap shear strength values are also given in Table I. The results of Table I are plotted in Figure 4 which shows the relationships between molecular weight and G_{IC}, as well as between molecular weight and lap shear strength. This figure clearly shows the considerable loss of the fracture energy as molecular weight decreased beyond a certain value, *i.e.*, about 18,500 amu which corresponds to PA concentration of about 1 percent. A similar phenomenon was observed with the polyphenylene ethersulfideimide (BDSDA/APB).[8] However, a difference between the present study with $PISO_2$ and previous study[8] with BDSDA/APB

TABLE II
Effect of temperature on polyimidesulfone adhesive properties

Test temperature °C	Fracture energy[a] G_{IC} J/m^2
−73	801
AMBIENT	652
149	568

[a] Each data point represents the average of three specimens.

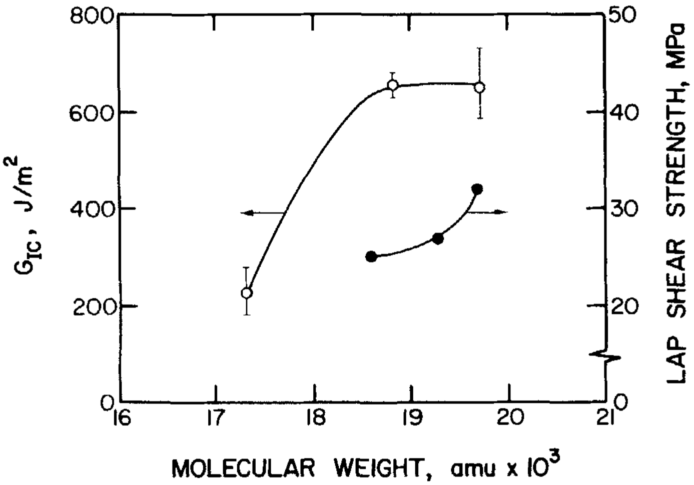


FIGURE 4 Effect of molecular weight on the fracture energy, G_{IC}, and lap shear strength of polyimidesulfone.

polyimide should be mentioned. The present study evaluated the fracture energy of the bonded joint with $PISO_2$, while in the previous study fracture energy of moldings from BDSDA/APB polyimide was measured as a function of its molecular weight. These studies, thus, show that the higher molcular weight system of polyimides resisted cracking more than the lower molecular weight system.

The lap shear strength of $PISO_2$ also decreased with the decrease of its molecular weight as shown in Figure 4. However, the trend in this decrease was not similar to the case of critical fracture energy, G_{IC}. This may be attributed to the fact that the lap shear test does not evaluate the resistance of a bonded joint against fracture failure based on fracture mechanics principles, rather it provides the resistance of a bonded joint to fracture failure in terms of the average shear stress. This average lap shear stress given in Figure 4 is not a direct measure of critical fracture energy G_{IC}. As mentioned earlier, the higher molecular weight system of polyimides increased the resistance against cracking, hence its proper quantitative and qualitative evaluation requires the measurement of G_{IC} instead of average applied stress.

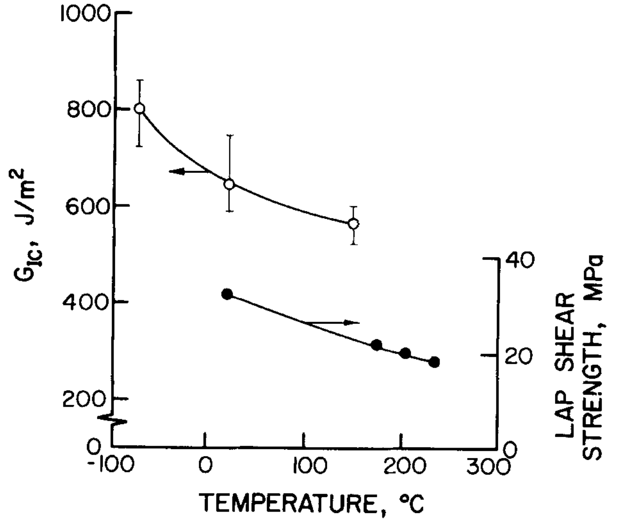


FIGURE 5 Effect of temperature on the fracture energy, G_{IC}, and lap shear strength of polyimidesulfone.

The temperature dependence of the adhesive fracture energy is given in Table II. These results are also shown in Figure 5. This study of temperature dependence was conducted only with polyimidesulfone with no endcapping, *i.e.*, zero percent of phthalic anhydride. Figure 5 also shows lap shear strength with increasing test temperature.[5] The results, given in Figure 5, show an increase in the fracture energy and lap shear strength of polyimidesulfone with decreasing temperature.

CONCLUSIONS

Polyimidesulfone is a promising polymer developed at NASA Langley Research Center for several aerospace applications. In an effort to evaluate it as an adhesive, the critical fracture energy, G_{IC}, was measured as a function of endcapping with varying concentrations of phthalic anhydride and temperature. This investigation led to the following conclusions:

1) There was a considerable loss of fracture energy as molecular weight decreased beyond a certain value which corresponded to phthalic anhydride concentration of one percent.

2) The fracture energy decreased with increasing temperature.

Acknowledgements

The work reported here was supported by NASA Langley Research Center, Hampton, VA. The author wishes to acknowledge the support and encouragement of Drs. T. L. St. Clair and W. S. Johnson, NASA Langley Research Center, during the course of this investigation.

References

1. E. J. Thrall, Jr., *Adhesives Age* **22**(10), 22–23 (1979).
2. A. J. Kinloch, *J. Matls. Sci.* **17,** 617–651 (1982).
3. S. J. Shaw, in *Adhesion* 7, K. W. Allen, Ed. (Appl. Sci. Publishers, Barking, Essex, 1983).
4. A. A. Griffith, *Proc. of* 1*st. Intern. Congress on Appl. Mech*, 1925, pp. 55–63.
5. T. L. St. Clair and D. A. Yamaki, in *Polyimides, Vol.* 1, K. L. Mittal, Ed. (Plenum Press, NY, 1984), pp. 99–116.
6. J. J. Singh, T. L. St. Clair and S. Walker, "A Study of the Effects of End-Cap Molecular Species on Environmental Characteristics of Polyimidesulfones", NASA Tech. Memorandum 86283, August, 1984.
7. D. J. Wilkins, *et al.*, in *Damage in Composite Materials*: *Basic Mechanisms, Accumulation, Tolerance and Characterization,* ASTM STP 775 (Am. Socy. for Testing and Materials, Philadelphia, 1981), pp. 168–183.
8. H. D. Burks, T. L. St. Clair, *J. Appl. Polym. Sci.* **29,** 4037–4053 (1984).