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Adhesively Bonded Butt Joint Strength: Temperature Dependence

E. D. Reedy, Jr., L. G. Hughes, J. M. Kropka, M. E. Stavig
Sandia National Laboratories
Albuquerque, NM, USA
edreedy@sandia.gov

Introduction

A tensile-loaded, adhesively bonded butt joint is commonly used to evaluate adhesives and is also a relatively simple geometry to analyze. Here we examine how joint strength varies with test temperature. One potential source of the observed dependence of strength on temperature has been identified.

Experimental

Tensile-loaded, adhesively bonded cylindrical butt joint

The adhesively bonded butt joints were formed by bonding two 6061-T6 aluminum rods together with an epoxy adhesive. The adherends are solid cylinders (28.6 mm diameter by 38.1 mm long) that have been precision machined to guarantee that the ends are flat and perpendicular to the cylinder axis (the edges were left sharp). The bonding surfaces of the aluminum adherends were lightly grit blasted (60 grit alumina oxide at 50 psi). The surfaces were cleaned by sonicating in deionized water for 10 minutes, immediately removing and wiping clean with isopropyl alcohol, wiping again with isopropyl alcohol, and finally drying with nitrogen. Each joint was assembled in a V-block fixture to ensure alignment of the two adherends, and a room temperature vulcanization (RTV) silicone (RTV 630, Momentive) boot was used to hold the epoxy resin in the bond gap during cure. The adherends were clamped to the alignment fixture during the filling operation to prevent motion. Clamps were removed prior to curing, such that the silicone boot was the only constraint to adherend axial motion. The silicone boot contained an injection hole and a reservoir to accommodate epoxy shrinkage. The epoxy adhesive is a diglycidyl ether of bisphenol A (EPON® Resin 828, Hexion) cured with Jeffamine® T-403 polyetheramine (Huntsman) at 43 parts per hundred resin. The adhesive was cured according to the following schedule: 24 hr. at 23^{o}C, followed by 3 hr. at 50^{o}C, followed by 15 hr. at 80^{o}C. After cure, the joints were annealed at 110^{o}C for 15 min. to erase the processing history and then cooled to 23^{o}C at 2^{o}C/min. to define a known thermal history of the structure prior to testing. The adhesive exhibits a glass transition that exhibits a midpoint at 85^{o}C when measured by thermal mechanical analysis. Compression tests of strain-gauged, molded epoxy plugs cured in the same manner and tested at room temperature (RT) and at a strain rate of 0.0001/s were used to measure the epoxy's elastic properties. The measured Young's modulus E equals 3.15 GPa while the Poisson's ratio ν equals 0.39. The epoxy's measured RT compressive yield strength at a strain rate of ~0.0003/s is 80 MPa.

Asymmetric Double Cantilevered Beam Sandwich Specimen

The Asymmetric Double Cantilevered Beam Sandwich (ADCBS) specimen was used to measure interfacial toughness. Interfacial toughness is a material property that characterizes the energy to propagate an existing interfacial crack. One aspect of interfacial fracture mechanics that distinguishes it from traditional linear elastic fracture mechanics is the role of crack-tip mode-mixity [1]. Asymmetries with respect to the interface are responsible for the inherently mixed-mode condition found at the tip of an interfacial crack. Even for a symmetric loading, elastic asymmetry generates both normal and shear stress on the interface ahead of the crack tip and the ratio of these stresses changes with distance from the crack tip. The level of mode-mixity $\psi_{r=l}$ (defined as the arctangent of the ratio of the shear stress to normal stress at a fixed distance l in front of the crack tip in the region dominated by the stress singularity) depends on the mismatch in elastic properties as well as specimen geometry and loading. Mode-mixity is important because the value of the interfacial toughness depends on the level of mode-mixity. The ADCBS specimen used in this study bonds 4.7 and 8.9 mm thick 6061-T6 aluminum beams together with a thin epoxy layer (both beams are 12.8 mm deep and 120 mm long). This specimen produces a predominantly Mode I-like loading near the crack-tip with a slight tendency to push the crack towards the interface so as to keep it on the interface. The specimen is pinned into a load train that utilizes a chain linkage and is loaded by pulling the ends apart at a crosshead displacement rate of 0.02 mm/s to propagate a crack along the interface with the thinner beam. The crack grows stably with increasing end displacement allowing several toughness measurements to be made while testing a single specimen. Crack length is inferred from specimen compliance, and the specimen is unloaded and reloaded several times during the test to establish the crack length during the loading step. The calibration used to determine toughness values from the inferred crack length and the load at the initiation of crack growth is based on published results for a homogeneous asymmetrical double cantilever beam specimen that ignores the compliance of the thin adhesive bond [2]. These results for a homogeneous specimen can be converted to those applicable to a sandwich test specimen with a middle layer that is thin relative to other dimensions [3]. Using this conversion, the sandwich specimen employed in this study has a crack-tip mode-mixity $\psi_{r=0.01\ mm}$ of about -20^o.

The same surface preparation as used for the butt joints was used for the ADCBS specimens (see the previous section). The measured root mean square surface roughness R_q was 4 μm. Small spacers are bonded to the ends of one of the adherends to define the epoxy layer thickness and the edges of the specimen are sealed with Teflon tape to form a cavity that is to be filled with epoxy. The cavity is filled by injecting epoxy through a small hole in one end of the thicker beam and allowing the epoxy to flow along the entire length of the cavity and then out of a small hole on the opposing end of the beam.

Results

Table 1 reports the results for two sets of nominally identical butt joints (referred to as set 1 and set 2), where the target bond thickness *h* was one mm. Each set of joints was split into two groups, with one half of the joints tested at room temperature and the other half tested at -50^{o}C. For a given test temperature, the average strength σ_f of set 1 and 2 joints is quite consistent, suggesting good reproducibility in the fabrication procedures. Interestingly, the tensile strength of the joints increased by 40% as test temperature was lowered from RT to -50^{o}C even though one might expect the epoxy to be more brittle and the residual stress to be higher at the lower temperature.

Table 2 presents ADCBS data that quantifies the dependence of interfacial toughness Γ on test temperature, epoxy cure cycle, and bond thickness. Set 1 samples have a 1.1 mm-thick bond while Set 2 samples have a 0.5 mm-thick bond. All Set 1 samples were cured for 24 hr. at 23^{o}C, followed by 3 hr. 50^{o}C, followed by 15 hr at 80^{o}C. One of these samples was also annealed at 110^{o}C for 15 min and cooled to 23^{o}C at 2^{o}C/min prior to testing. Half of Set 2 samples were cured in the same way as Set 1 samples (and all of these samples were subjected to the annealing step prior to testing). The other half of the Set 2 samples was cured using an alternate curing schedule that had been used in previous studies [4]. These samples were cured for 24 hr. at 23^{o}C, followed by 24 hr. at 50^{o}C, followed by 24 hr. 40^{o}C. Test temperature had the most striking effect. The interfacial toughness increased by 85% as the test temperature decreased from room temperature to -65^{o}C (Fig. 1, averaged Table 2 toughness at each test temperature). On the other hand, there was no significant change in toughness when the bond thickness was decreased from 1.1 mm to 0.5 mm. Likewise; the samples fabricated using either of the cure cycles had similar measured toughness.

Analysis

A first cut estimate of the strength of adhesively bonded butt joints like those tested (Table 1) can be made using the long-crack estimate of the strength of a rigid adherend butt joint subjected to a bond-normal loading

$$\sigma_f = \sqrt{2E_u \Gamma / h} \qquad (1)$$

where E_u is the uniaxial strain modulus [5]. Note that when crack is sufficiently long, the strain energy at the stress-free edge should be negligible and the residual stress is "locked in" since one side of bond remains attached to the rigid adherend (i.e., there is no contribution to the energy release rate as the long crack extends). The fact that residual stress in a thin-layer-sandwich does not drive crack growth has been previously noted by others [1]. Furthermore, for a long crack, the crack is subjected to a primarily a mode I-like loading, so mode-mixity effects are minimized.

Table 3 lists the values of the parameters used in the estimate as well as the estimated strength. The elastic properties correspond to those measured for the same Epon 828/T403 epoxy as used to fabricate the butt joints (100:43 pbw, cured 24 hr. at 23^{o}C, followed by 3 hr. 50^{o}C, followed by 15 hr at 80^{o}C). The Young's modulus was assumed to increase by 20% as the joint is cooled from RT to -50^{o}C. Figure 1 shows that the measured interfacial toughness increases from 90 J/m^2 to 150 J/m^2 as the temperature is deceased from RT to -50^{o}C (i.e., Γ increases by ~ 67% as the test temperature as decreased). Based upon these parameters, the estimated joint strength increases from 34 MPa at RT to 48 MPa at -50^{o}C; a 40% increase. The measured butt joint strength was 27 MPa at RT and 38 MPa at -50^{o}C, also a 40% increase. The first cut, long-crack estimate for joint strength is ~ 25% too high. This is not surprising since the fracture surfaces indicate that crack growth is 3D in nature with initiation from a single point on the outer, bond periphery. The plane strain, long-crack idealization is clearly a gross simplification. Nevertheless, this result seems to suggest that the increase in joint strength with decreasing temperature may be largely attributable to the increase in interfacial toughness with decreasing temperature.

Acknowledgements

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Figures

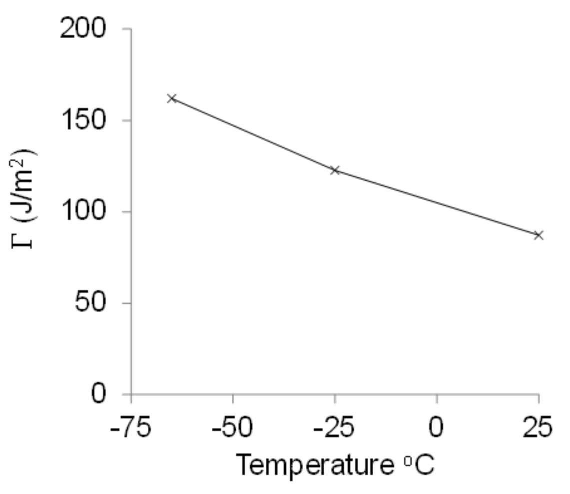

Figure 1. Measured temperature-dependent interfacial toughness ($\psi_{r=0.10\ mm}$ = -20°).

Tables

Table 1. Butt joint strength data.

set	h mm	test temp. °C	# tested	avg. σ_f MPa	st. dev. σ_f MPa
1	1.05	23	5	27.4	1.8
1	1.10	-50	5	37.8	5.1
2	0.97	23	10	26.9	3.6
2	1.01	-50	9	38.1	2.5

Table 2. Interfacial toughness vs. test temperature and cure cycle.

set	#	h mm	test temp °C	cure[1]	avg. Γ J/m^2	st. dev. Γ J/m^2
1	1	1.1	RT	1-no anneal	87	4
1	2	1.1	-25	1-no anneal	129	4
1	3	1.1	-65	1-no anneal	159	3
1	4	1.1	-65	1- an-nealed	154	5
2	1	0.5	RT	1- an-nealed	92	4
2	2	0.5	RT	2	87	3
2	3	0.5	-25	1- an-nealed	124	4
2	4	0.5	-25	2	121	2
2	5	0.5	-65	1- an-nealed	169	3
2	6	0.5	-65	2	162	3

[1] cure 1: 24 hr. at 23°C, followed by 3 hr. 50°C, followed by 15 hr. at 80°C. If annealed, annealed at 110°C for 15 min. and cooled to 23°C at 2°C/min. prior to test.

cure 2: 24 hr. at 23°C, 24 hr. at 50°C, followed by 24 hr. 40°C.

Table 3. Long-crack estimate of butt joint strength for a one mm-thick bond.

°C	E (MPa)	ν	E_u (MPa)	Γ (J/m^2)	σ_f (MPa)
23 (RT)	3150	0.39	6280	90	34
-50	3780	0.39	7540	150	48