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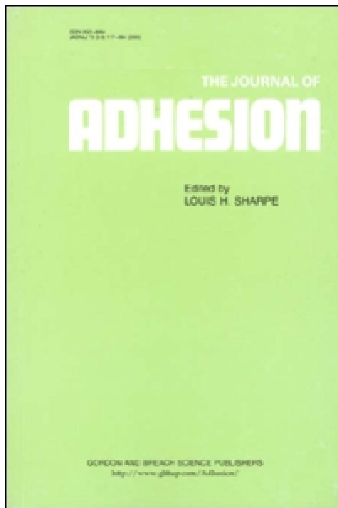

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Thin-Disk Test for Adhesion-Bond Strength in Solvent Environments

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Thin-Disk Test for Adhesion-Bond Strength in Solvent Environments

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A number of the popular tests for adhesive strength are difficult to apply to the study of adhesion under solvent environments. Complex applications, in which two different substrate materials need to be bonded and for which the substrates are thin sections, can be particularly difficult to study. The thin-disk test described here uses a thin annular disk of adhesive to bond two dissimilar materials while exposing the bond line to a circulating solvent. The new test was evaluated for a typical inkjet print-head application using surrogates for inkjet water-based inks. The joint is an epoxy adhesive joining a silicon wafer to a thermoplastic part (Rynite®), in which the silicon substrate, the thermoplastic, or the various adhesive interfaces might fail.

A conventional lap-shear test was compared with the thin-disk test for samples exposed to four different solvent systems plus water at two different temperatures. Lap-shear test failures occurred mostly in the thermoplastic part, with the exception of two samples exposed to the most aggressive solvents at high temperature. By contrast, thin-disk test failures occurred either in the silicon substrate or in the thermoplastic–adhesive interface. The thin-disk failure strengths at the thermoplastic–adhesive interface correlated with the equilibrium solvent swelling that could occur in the adhesive under the test conditions. This method could be adapted to other mixed-substrate bonding systems and would be particularly appropriate for thin section solids and thin adhesive layers.

Keywords: Adhesive bond; Adhesive swelling; Blister test; Lap shear; Thin disk

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INTRODUCTION

The adhesive attaching the die to the inkjet print head plays a critical role in meeting the printing system's print quality and reliability requirements. This adhesive bond holds the silicon heater chip to the body of the print head and functions as a seal to prevent cross-contamination between the cyan, yellow, and magenta inks. The die attach adhesive must maintain its properties throughout the life of the device for the print head to function properly [1]. Because the assembly is directly exposed to ink, adhesion can be lost because of increases in internal stresses within the adhesive phase as solvents diffuse through exposed surfaces. Therefore, the die attach adhesive must demonstrate excellent compatibility, *i.e.*, have a low sorption response with each component in the ink. Inkjet inks are water-based with small amounts of solvents that act as humectants and surface-tension modifiers. Inks also have dyes and pigments to produce color and permanence after printing. Sorption of solvents and dyes from these complex mixtures can cause failure in the bulk adhesive or at any adhesive–solid interface such as the body materials or the silicon heater chip. Understanding the failure mechanisms for the complete system, silicon chip–die attach adhesive–print head body material, in the presence of ink is critical for creating a properly operating print head. A successful test could be applied to a number of similar high-technology adhesive applications for thin section materials exposed to solvent and corrosive environments.

There are a variety of methods for measuring adhesion between an adhesive and a particular substrate. Typical test geometries include single lap-shear joints, double lap-shear joints, butt joints, 90/180-deg peel tests, blister tests, the double cantilever beam (DCB), the tapered double cantilever beam, die-shear tests, and wedge tests [2–5]. Each of these joint configurations has particular purposes. For instance, the DCB is often used to determine the cohesive strength of the adhesive [6] whereas the peel test is used primarily to determine the adhesion of a low-modulus adhesive [7].

The wedge test is used to determine the effect of surface treatments on various metals [8]. In microelectronic packaging, the die-shear adhesion test is used to determine the adhesion between a silicon die and substrate. This popular test is subject to large errors because the applied shear load of the fixture on the extremely brittle silicon die often causes the silicon die to fracture before any cohesive or interfacial failure occurs [9, 10]. The wedge test developed by Boeing Corporation [8] has been used in the past to determine the durability of various adhesives to metal surfaces in different environmental

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conditions and liquid media. This test also is useful in evaluating various surface pretreatments and coatings [11].

Lap-shear joints, possibly the most commonly used test method, can determine loss in adhesion as a function of environment [12]. Their popularity relates to their ease of sample preparation, simple fixturing, and low cost. Although lap-shear joints are useful for determining dry adhesion strengths, the joint has a very small cross-sectional area exposed to solvent. Therefore, changes in adhesive strength caused by diffusion of ink solvents into the test specimen require long exposure times to achieve equilibrium swelling. This geometry contributes to numerous experimental errors and high standard deviations in the measured adhesion strengths. Effects that contribute to large errors include the amount of eccentricity, internal stresses in the adhesive film, and elastic bending of adherends. Lap-shear joint data are affected by the sample preparation, *i.e.*, cleanliness, surface roughness, excess adhesive around the edges, and the spew fillet.

Unfortunately, diffusion and solubility interactions with the adhesive in lap-shear-joint tests are limited because of the low surface area of fluid in contact with the ink or solvents. Only the perimeter of the lap-shear joints, which has a relatively low bond-line surface area, is exposed to the ink. Typical adhesive thicknesses for lap-shear joints are 0.3 mm. Diffusion of solvents across the small surface area of the thin bond line gives low fluxes, requiring long testing times to achieve significant sorption. Lap-shear joints are constructed using two pieces of the polymeric substrate and the adhesive, because the silicon die is too thin to be used in this test. Therefore, only polymer-adhesive bonding is tested and the strength of the silicon-adhesive bond is not known. Another fault of the lap-shear geometry is that the applied stress to the joint is in shear, which is not the case for the silicon die–adhesive–polymeric substrate.

The purpose of this research was to develop an adhesion test (the thin-disk test) that could measure the bonding between the adhesive and two different substrates (both the polymeric substrate and the brittle silicon substrate), while replicating a similar adhesive-bonding area that would be found on an inkjet print head. This test applies hydrostatic pressure at both the silicon–adhesive and the adhesive–substrate interfaces as well as at the bulk adhesive and makes use of the blister test configuration [4]. By monitoring the pressure, an accurate determination of the force at which loss of adhesion occurs can be obtained. The benefits of the thin-disk test are that the weakest phase of the substrate–adhesive–substrate system is always determined, while achieving low experimental error and standard deviation. This paper reports thin-disk test results for the adhesion strength of

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the die-attach adhesive to the substrate (print-head body material) and for the loss of adhesion as a function of exposure to various liquid media at various temperatures in an ideal configuration. We have tested this with pure solvents and water solutions similar to inkjet inks.

EXPERIMENTAL

Materials

Lap-shear joints and die-adhesion samples were prepared by using a commercial one-part system of Bisphenol A (DGEBA) with a methyl tetrahydrophthalic anhydride curing agent. The thermoplastic substrates used for both the die-adhesion samples and the lap-shear joints were purchased from Dupont under the trade name of Rynite® FR515 (Dupont, New Castle, DE, USA). Rynite FR515 is a glass-filled blend of poly(ethylene terephthalate) and poly(butylene terephthalate). Blank polished silicon wafers were used for the die-adhesion joints.

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Lap Joints

Lap-shear joints were prepared by using the American Society for Testing and Materials (ASTM) standard D 3163-96 test method [13]. Rynite plaques were molded to a dimension of 3.35 in. (85 mm) by 0.787 in. (20 mm) and a thickness of 0.0591 in. (1.5 mm). The substrates were cleaned and degreased using methanol. The adhesive was dispensed using a CAM/ALOT™ automated positive displacement pump system (Speedline Technologies, Franklin, MA, USA) that allows accurate adhesive placement and weights. The thickness of the lap-shear joint was controlled by using two 0.0118-in. (0.3-mm)-thick spacer wires. Peak load to failure was tested using a MTS Qtest/10 LP tensile tester (MTS, Eden Prairie, MN, USA) at a rate of 0.0496 in./min (1.26 mm/min) with a 10KN load cell. Five samples for each test condition were measured to compute the average response. The solvent exposure period was 30 days.

Die-Adhesion Joints

Figure 1 shows a cross-section of the die-adhesion joint (silicon substrate, adhesive ring, and Rynite substrate), with the solvent applying hydrostatic pressure on the assembly. The thin-disk test system consisted of a screw pump, a pressure transducer [rated at 6.89 MPa (1000 psi)], a heater, a thermocouple, and a computer (Figure 2). The screw pump and pipe were filled with water and the system was

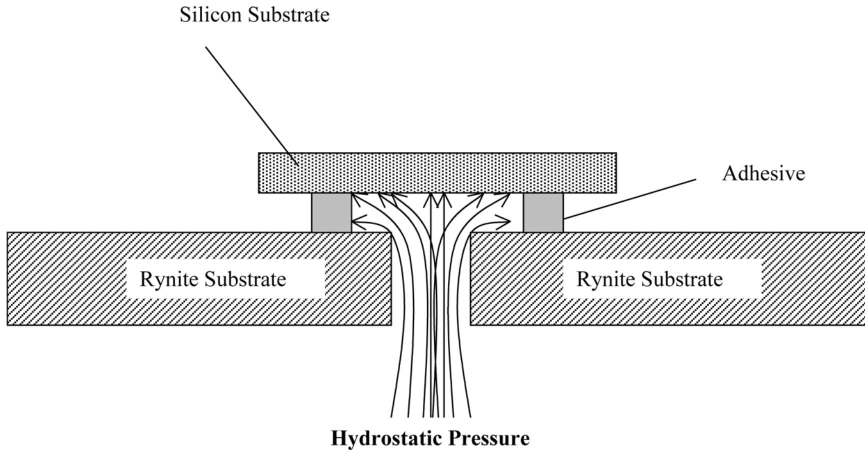

FIGURE 1 Cross-section of thin-disk joint assembly (not to scale).

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pressured as water circulated at a flow rate of 0.0305 cubic in./min (0.5 ml/min). Liquid pressure *versus* time data were obtained and recorded each second by a digital/analog converter with Lab View data acquisition software (Lab Tech, Andover, MA).

The silicon part was prepared by dicing a wafer to a square 0.748 in. (19 mm) on each side with a thickness of 0.0268 in. (0.68 mm).

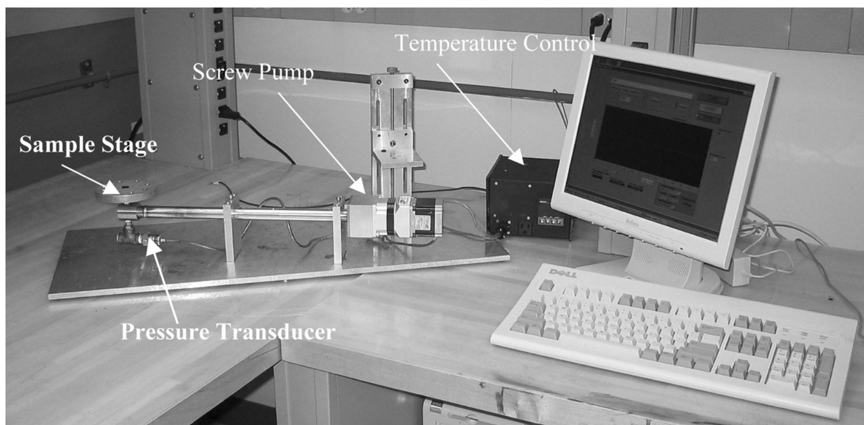

FIGURE 2 Die-adhesion-test system: 1) motorized screw pump, 2) substrate platform, 3) pressure transducer, and 4) analog–digital interface with computer.

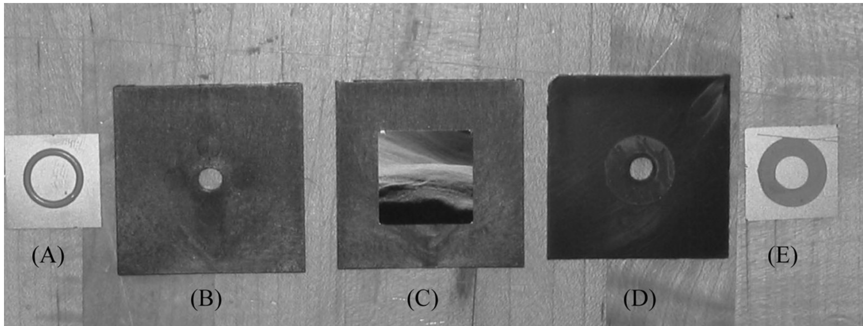

FIGURE 3 Photograph of (a) adhesive dispensed in a circular pattern on silicon, (b) Rynite substrate with 5-mm hole, (c) constructed joint, (d) Rynite failure surface, and (e) silicon failure surface; one of two spacer wires is shown.

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The die-attach adhesive pattern was in the form of a 'circle', which weighed roughly 20 to 30 mg (Figure 3a). Square die-adhesion plaques were molded to the dimension of 1.57 in. (40 mm) on a side with a thickness of 0.138 in. (3.5 mm) and a center hole 0.197 in. (5 mm) in diameter (Figure 3b). Figure 3c shows the assembly after the silicon part was inverted and placed on the Rynite substrate. Figure 3d shows the Rynite substrate after the test. The faint pattern of the adhesive on the Rynite corresponds to that seen on the silicon part (Figure 3e). Two spacer wires on opposite sides of the adhesive were used to control the adhesive thickness (one is shown in Figure 3e). A standard weight of 50 g (0.11 lb) was placed on each silicon–adhesive–Rynite joint to ensure similar adhesive contact area. During the test, the specimen failed catastrophically after 20 to 30 s [10].

Solutions

The solvents used in this experiment represent ink additives of different chemical characteristics and molecular weights: hexyl carbitol (97%, di(ethylene glycol) hexyl ether; [112-59-4]), 2-pyrrolidone (99%, 2-pyrrolidinone; [616-45-5]), and polyethylene glycol (Mw of 400; [25322-68-3]) from Aldrich Chemical Company, Milwaukee, WI, USA. Solutions of each solvent were prepared by mixing 0.8 wt.%, 5.0 wt.%, and 5.0 wt.% of hexyl carbitol, 2-pyrrolidone, and polyethylene glycol 400, respectively, in deionized water. A simulated inkjet ink mixture (referred to here as the ternary system) was prepared by mixing 0.8% hexyl carbitol solution, 5.0% 2-pyrrolidone, and 5.0%

polyethylene glycol 400 into deionized water. These levels are typical of such solvents in inkjet inks. Both lap-shear and die-adhesion samples were tested at room temperature and 60°C for a period of 30 days to allow long term exposure to the synthetic ink.

Thermal Properties

Samples for thermal analysis were taken from each of the adhesive films after exposure to the various environments. Thermal properties were obtained using a modulated TA Differential Scanning Calorimeter (New Castle, DE, USA). The heating rate was 5°C/min to 150°C and temperature was modulated ±0.80°C every 60 s. The glass transition temperature, T_g, was determined by finding the inflection points of the thermal scans. The glass transition temperature of the adhesive without solvent was 70°C. Adhesive samples with sorbed solvent had lower glass transition temperatures in all cases. Solvent evaporation during DSC scans can cause false readings when the temperature nears the boiling point of the solvent. All of the solvents had normal boiling points higher than water, so that the loss of solvent would be a concern only if the measurement temperature exceeded ~100°C, the boiling point of the lowest boiling solvent in the mixture (water).

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Adhesive Volume Fraction Samples

Swelling of the adhesive by solvent generally puts extra stresses on cohesive bonding in its bulk phase and on the adhesive bonds at the adhesive–solid interfaces. Thin adhesive films were prepared by casting a 0.0197-in. (0.5-mm)-thick film on a preheated block of Teflon®, and then placing it in a 135°C oven for 60 min. The epoxy film was cut into a 0.787-in. (20-mm) square while the adhesive was above the glass transition temperature. Each sample was allowed to cool to room temperature. They were then weighed with an analytical balance and placed into the various solvent solutions along with the adhesive joints. Both the joint and the volume fraction uptake samples were taken out of each solution after 30 days. The outer surface of the soaked samples was patted dry and weighed with a standard deviation of 0.2 mg. The equilibrium volume fraction uptake was calculated from the percent change in mass as a function of time/temperature in each solvent or solution. The calculation was based on the weights of the adhesive and solvent in the swollen system, their densities, and the assumption of no volume change on mixing.

RESULTS AND DISCUSSION

Test Temperature *vs.* Glass Transition Temperature of the Adhesive–Solvent Mixture

One of the challenges in designing a test for adhesion failure under solvent attack is the changes that solvent swelling causes in solvent uptake and in the swollen adhesive properties. Test configurations with low exposed adhesive surface area may not reach equilibrium swelling over the time period of the experiment and may not mimic the performance of the actual adhesive joint. Glass transition temperature measurements are one way to identify changes in the diffusion mechanisms, which depend on whether the sample is in the rubbery ($T > T_{g,mixture}$) or glassy ($T < T_{g,mixture}$) regions. The solvent diffusion coefficients in the adhesive phase increase by orders of magnitude when the measurement temperature increases above the glass transition temperature of the polymer–solvent mixture.

The last two columns of Tables 1 and 2 show the swelling volume fractions and glass transition temperatures of the swollen adhesive phases in pure solvents. Similar data appear in Tables 3 and 4 for solvent–water mixtures. For pure solvent swelling at room temperature, only the hexyl carbitol and 2-pyrrolidone systems were in the rubbery region. At 60°C, all the pure solvents except water caused the swollen adhesive to have a glass transition temperature lower than the test temperature. Adhesive swelling was significantly less when the solvents were dispersed in water at typical ink concentrations. At 25°C, solvent diffusion into glassy polymers occurred in all of the adhesive samples exposed to water mixtures. At 60°C, the hexyl carbitol, the 2-pyrrolidone, and the ternary mixture samples experienced solvent diffusion into rubbery polymers. In some cases, the glass transition temperature of an adhesive exposed to a particular liquid is higher than the control. This can be rationalized by the possibility that the bulk liquid phase displaces some low-molecular-weight material from the adhesive, but this is not critical to test development. Tensile tests results should reflect these differences in reaching equilibrium.

The performance of lap-shear joints and the new thin-disk test were compared in pure solvent and simulated inkjet ink solution environments at room temperature (25°C) and 60°C.

Lap-Shear Joints

Pure-Solvent Environment

Room Temperature. Sample exposure to pure solvents should give high solvent flux rates into the adhesive phase. The room-temperature,

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TABLE 1 Lap-Shear Strengths in Pure-Solvent Environments

Solvents	Lap-shear strength, psi (MPa)		Solvent volume fraction (%)		Measured T_g (°C)	
	25°C	60°C	25°C	60°C	25°C	60°C
Control	370 ± 22 (2.55 ± 0.15)	370 ± 22 (2.55 ± 0.15)	0 ± 0.2 mg	0 ± 0.2 mg	$70 \pm < 1$°C	$70 \pm < 1$°C
H_2O	351 ± 29 (2.42 ± 0.20)	348 ± 35 (2.40 ± 0.24)	1.1	3.24	70	73
PEG 400	365 ± 33 (2.52 ± 0.23)	362 ± 29 (2.50 ± 0.20)	2.17	19.48	46	23
Ternary	357 ± 34 (2.46 ± 0.23)	356 ± 38 (2.45 ± 0.26)	4.71	33.96	68	N/A
Hexyl carbitol	388 ± 31 (2.68 ± 0.21)	186 ± 56 (1.28 ± 0.39)	43.59	45.64	–1	N/A
2-Pyrrolidone	341 ± 22 (2.35 ± 0.15)	96 ± 44 (0.66 ± 0.30)	43.04	80.34	4.2	N/A

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TABLE 2 Die-Adhesion Strengths in Pure-Solvent Environments

	Die-adhesion strength, psi (MPa)		Solvent volume fraction (%)		Measured T_g (°C)	
Solvent	25°C	60°C	25°C	60°C	25°C	60°C
Control	(622 ± 44) (4.29 ± 0.30)	(622 ± 44) (4.29 ± 0.30)	0 ± 0.2 mg	0 ± 0.2 mg	70 ± <1°C	70 ± <1°C
H_2O	(607 ± 42) (4.18 ± 0.29)	(561 ± 45) (3.87 ± 0.31)	1.1	3.24	70	73
PEG400	(697 ± 40) (4.81 ± 0.28)	(437 ± 42) (3.01 ± 0.29)	2.17	19.48	46	23
Ternary	(617 ± 21) (4.25 ± 0.14)	(359 ± 23) (2.48 ± 0.16)	4.71	33.96	68	N/A
Hexyl carbitol	(413 ± 31) (2.85 ± 0.21)	(117 ± 22) (0.81 ± 0.15)	43.59	45.64	–1	N/A
2-Pyrrolidone	(557 ± 28) (3.84 ± 0.19)	(18 ± 10) (0.12 ± 0.07)	43.04	80.34	4.2	N/A

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TABLE 3 Lap-Shear Strengths in Water Solutions

	Lap-shear strength, psi (MPa)		Solvent volume fraction (%)		Measured T_g (°C)	
Solution	25°C	60°C	25°C	60°C	25°C	60°C
Control	(370 ± 22) (2.55 ± 0.15)	(370 ± 22) (2.55 ± 0.15)	0 ± 0.2 mg	0 ± 0.2 mg	$70 \pm <1$°C	$70 \pm <1$°C
H_2O	(351 ± 29) (2.42 ± 0.20)	(348 ± 35) (2.40 ± 0.24)	1.1	3.24	70	73
PEG 400	(359 ± 42) (2.48 ± 0.29)	(384 ± 42) (2.65 ± 0.29)	0.83	4.1	77	73
Ternary	(357 ± 34) (2.46 ± 0.23)	(356 ± 38) (2.45 ± 0.26)	3.11	19.48	74	23
Hexyl carbitol	(413 ± 22) (2.85 ± 0.15)	(393 ± 33) (2.71 ± 0.23)	1.48	11.8	76	34
2-Pyrrolidone	(377 ± 30) (2.60 ± 0.21)	(389 ± 56) (2.68 ± 0.39)	0.81	4.1	75	67

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TABLE 4 Die-Adhesion Strengths in Water Solutions

	Die-adhesion strength psi, (MPa)		Solvent volume fraction (%)		Measured T_g (°C)	
Solution	25°C	60°C	25°C	60°C	25°C	60°C
Control	(622 ± 44) (4.29 ± 0.30)	(622 ± 44) (4.29 ± 0.30)	0 ± 0.2 mg	0 ± 0.2 mg	70 ± <1°C	70 ± <1°C
H_2O	(607 ± 42) (4.18 ± 0.29)	(561 ± 45) (3.87 ± 0.31)	1.1	3.24	70	73
PEG 400	(700 ± 38) (4.83 ± 0.26)	(702 ± 43) (4.84 ± 0.30)	0.83	4.1	77	73
Ternary	(617 ± 21) (4.25 ± 0.14)	(437 ± 39) (3.01 ± 0.27)	3.11	19.48	74	23
Hexyl carbitol	(681 ± 45) (4.70 ± 0.31)	(597 ± 34) (4.12 ± 0.31)	1.48	11.8	76	34
2-Pyrrolidone	(511 ± 34) (3.52 ± 0.23)	(466 ± 23) (3.12 ± 0.16)	0.81	4.1	75	67

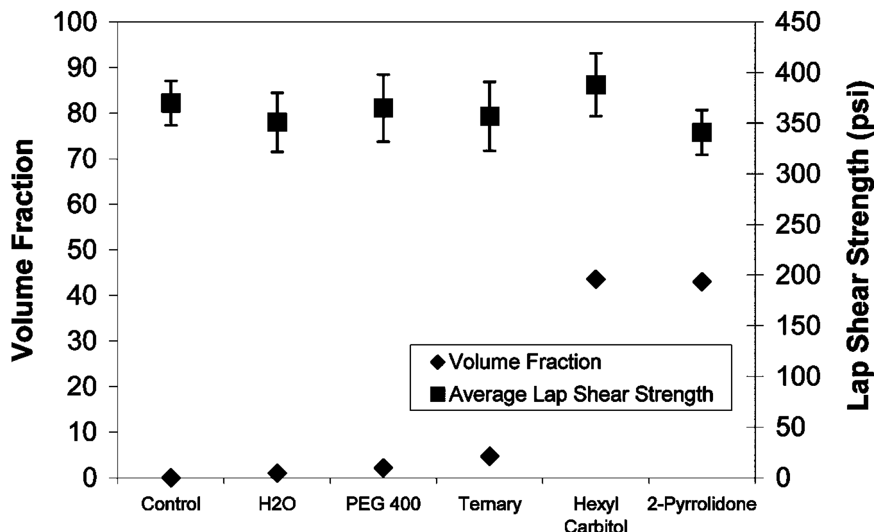

FIGURE 4 Room-temperature, pure-solvent, lap-shear strength and sorption data.

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pure-solvent, average lap-shear strengths and their standard deviations are shown in Figure 4 along with the respective equilibrium volume fraction uptakes. The measured lap-shear strengths for all samples are essentially the same, as the mean values for the replicate experiments fall within two standard deviations of each other. The failure mechanism for each lap-shear test was within the Rynite substrate, explaining the rather low standard deviation of the lap-shear strength. Failure within the Rynite substrate indicated that little solvent diffused into the adhesive during the 30-day test.

The glass transition and swelling volume fraction data showed that, at equilibrium, both hexyl carbitol and 2-pyrrolidone caused the adhesive to swell to a rubbery state. Although the failure mode of the lap-shear test could have shifted to adhesive failure for these solvents, there was insufficient flux of the solvent through the exposed adhesive in this test configuration.

60°C. At higher experimental temperatures, the flux of solvent through the exposed bond line should have been larger, and changes in failure mechanisms should have occurred at shorter experimental times. Figure 5 shows the equilibrium volume fraction uptake, average lap-shear adhesion strengths, and standard deviations for control, H_2O, ternary, PEG400, hexyl carbitol, and 2-pyrrolidone pure-solvent

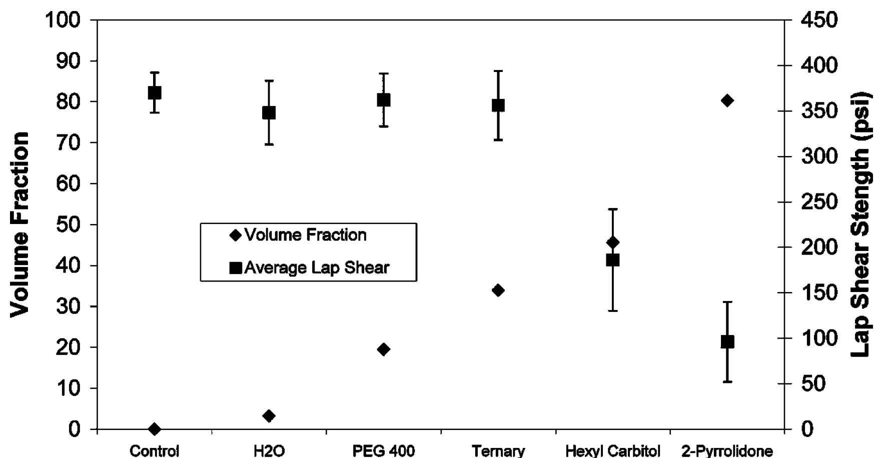

FIGURE 5 60°C, pure-solvent, lap-shear strength and sorption data for 30-day samples.

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solutions at 60°C. Based on the glass transition temperatures, all of the solvents except for water caused the adhesive to swell to rubbery states at equilibrium. The mode of failure for the control, H_2O, ternary, and PEG400 systems were within the Rynite, whereas the hexyl carbitol and 2-pyrrolidone had cohesive failures within the adhesive. These two samples had larger average standard errors (the standard deviation divided by the mean value) in lap-shear strength than the samples that failed within the Rynite, confirming a change in failure mechanism. Although both the PEG 400 and ternary solvents could lower the glass transition temperature of the adhesive below the test temperature, there apparently was not enough solvent flux in the 30-day test for the swelling to result in a change in failure mechanism.

Water-Solution Environment

Room Temperature. When the various solvents were dispersed in water at concentrations similar to those found in inkjet inks, their activities would be much smaller and their diffusive flux rates into the adhesive phase would be much lower than those for the pure solvents. At room temperature, the adhesive–solvent mixture would always be in the glassy region (based on the glass transition temperature measurements in Table 3), causing even lower diffusive flux rates. Average lap-shear strengths for the PEG-400, hexyl carbitol, and 2-pyrrollidone room-temperature solvent solutions showed no loss in adhesion relative to the control (Table 3). All failures were within

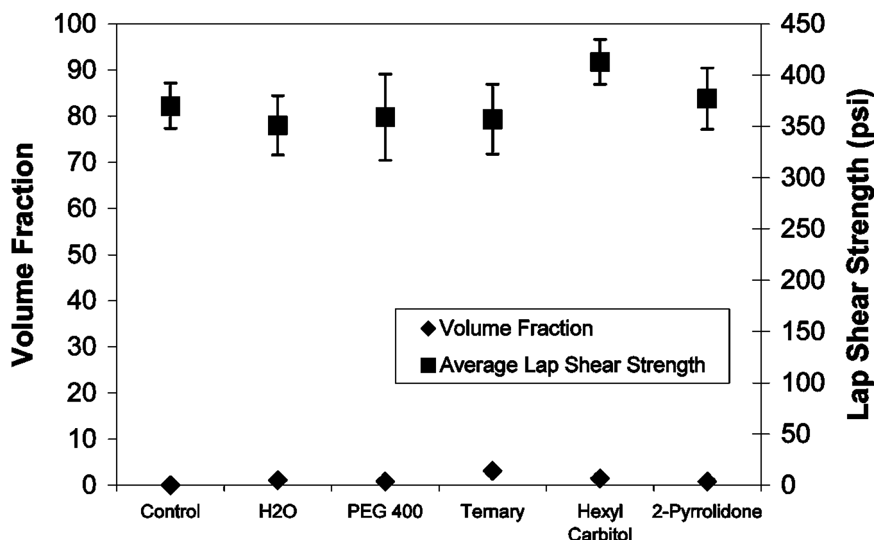

FIGURE 6 Room-temperature, lap-shear strength and sorption data for water-solution samples.

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the Rynite substrate. Also, the equilibrium volume fraction uptakes for the adhesive were relatively low compared with the pure-solvent samples. The average lap-shear strengths, standard deviations, and equilibrium volume fraction uptakes in Figure 6 confirm that the lap-shear strengths were essentially the same for all samples.

60°C. The ternary– and hexyl carbitol–water mixtures should cause the adhesive to be rubbery at equilibrium swelling, while the rest of the systems should have the adhesive in the glassy state. The solvent solutions' lap-shear strengths, standard deviation, and adhesive sorption for the 60°C samples are shown in Figure 7. All failures were within the Rynite substrate. Although the average lap-shear strength of the ternary solution appeared to be slightly lower, there were no significant strength differences among any of the samples. Although changes in the failure mechanisms for the ternary and hexyl carbitol systems could be possible, they were not observed over the time scale of these experiments.

Identification of Failure Mechanisms by the Lap-Shear Test

Experimental conditions in which the adhesive swells significantly and is in the rubbery state can lead to loss of adhesion at solid–adhesive interfaces or in the bulk adhesive itself. Exposure to pure

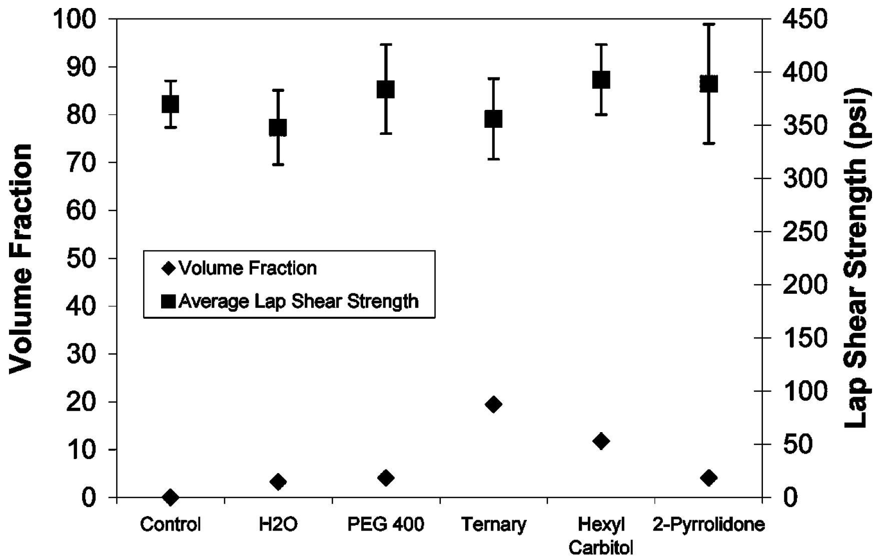

FIGURE 7 Water-solution, 60°C, lap-shear strengths and sorption data.

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solvents could lead to adhesion failures for the hexyl carbitol and 2-pyrrolidone experiments at room temperature and for all solvents except water at 60°C. The lap-shear test showed no adhesion failures at room temperature and only the hexyl carbitol and 2-pyrrolidone systems showed adhesion failures at 60°C. Exposure to simulated inkjet mixtures could have led to adhesive failure for the ternary and hexyl carbitol systems at 60°C. However, all of these systems failed in the Rynite and no change in failure mechanism was detected. There was no correlation between equilibrium solvent swelling of the adhesive and the lap-shear strength.

Die-Adhesion Joints

From the previous discussion, it was evident that an alternative joint geometry was needed for a better understanding of the adhesion of silicon–adhesive–Rynite joints in the presence of solvent. The new joint geometry was developed to increase the solvent sorption in the adhesive. This test incorporated both a larger *exposed* surface area per volume ratio (a factor of $\sim$4 larger for the thin-disk test) and a much shorter diffusion path (an order of magnitude lower than the lap-shear test). The thin-disk test uses both the Rynite polymeric substrate and the silicon die, mimicking a real inkjet print-head

configuration. This test can show whether one of the substrates, one of the adhesive–substrate interfaces, or the adhesive failed. For example, in the absence of solvent, the Rynite–adhesive–silicon system always failed by fracture of the silicon. This type of failure was not observed when solvent was present.

Pure-Solvent Environment

Room temperature. Table 2 shows the equilibrium volume fraction uptake, average die-adhesion strengths, standard deviations, and the glass transition temperatures of the swollen adhesive for control, H_2O, ternary, PEG 400, hexyl carbitol, and the 2-pyrrolidone for the room-temperature, pure-solvent solutions. In contrast to the lap-shear data, the die-adhesion data (Figure 8) suggested a correlation between the die-adhesion strength and the solvent volume fraction at both room temperature and 60°C. The average standard error of the adhesion strength changed little with solvent swelling. The systems with hexyl carbitol and 2-pyrrolidone, which were in the rubbery state at equilibrium swelling, showed reduced die-adhesion strength. For these systems, the failure mechanism changed from within the silicon to the Rynite–adhesive interface (not the silicon–adhesive interface).

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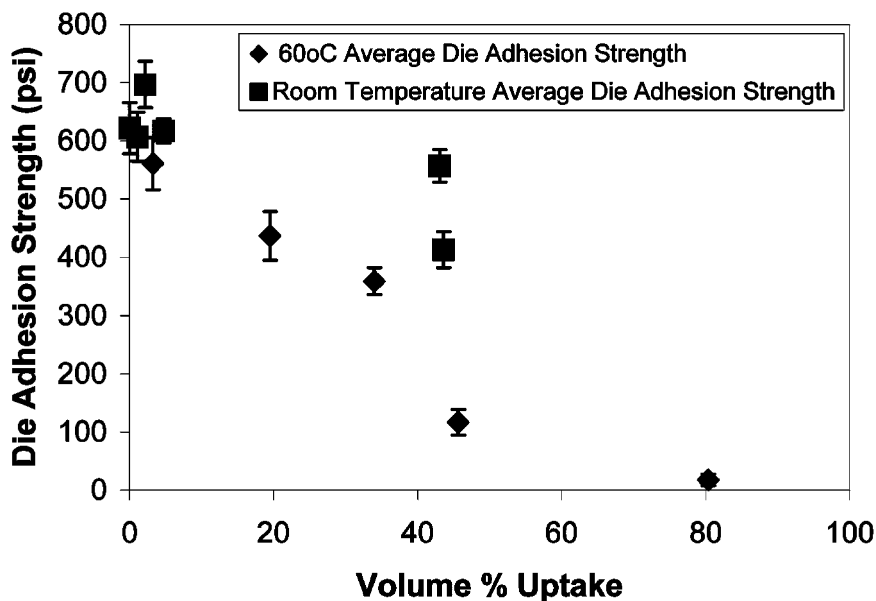

FIGURE 8 Die-adhesions strength *vs*. volume% solvent uptake for pure solvents at room temperature and 60°C.

This is a different failure mechanism from that determined by the lap-shear test (failure in the Rynite).

60°C. The adhesive strength decreases directly with solvent swelling at 60°C, suggesting that the adhesives are near the solvent-swelling equilibrium in the thin-disk test configuration. The largest loss in adhesion occurred in the 2-pyrrolidone sample, which lost 97% in die-adhesion strength relative to the control while the solvent volume fraction was 80%. Large decreases in adhesion were also observed for the hexyl carbitol, PEG 400, and the ternary systems. The locus of failure occurred consistently at the Rynite–adhesive interface. In the lap-shear test, only the hexyl carbitol and 2-pyrrolidone systems demonstrated cohesive failure of the adhesive.

Water-Solution Environment

Room temperature. Average die-adhesion strengths for the PEG-400, ternary, and hexyl carbitol solutions showed little to no loss relative to the control (Figure 9). All failures occurred at the Rynite–adhesive interface. The fact that no changes in die-adhesion strengths were observed indicates that the combination of low temperature and low solvent activity in the water phase reduced the water and solvent fluxes into the adhesive networks compared with the pure solvent systems.

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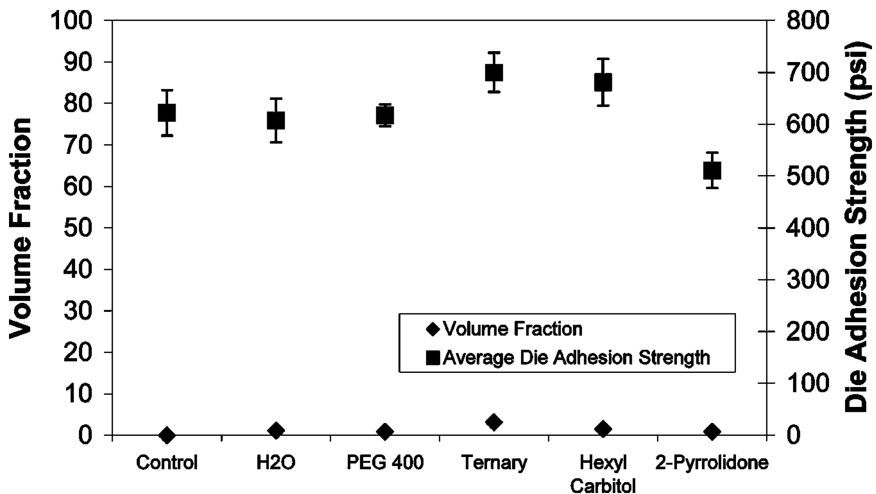

FIGURE 9 Die-adhesion strengths and sorption data for water solutions at room temperature for 30 days.

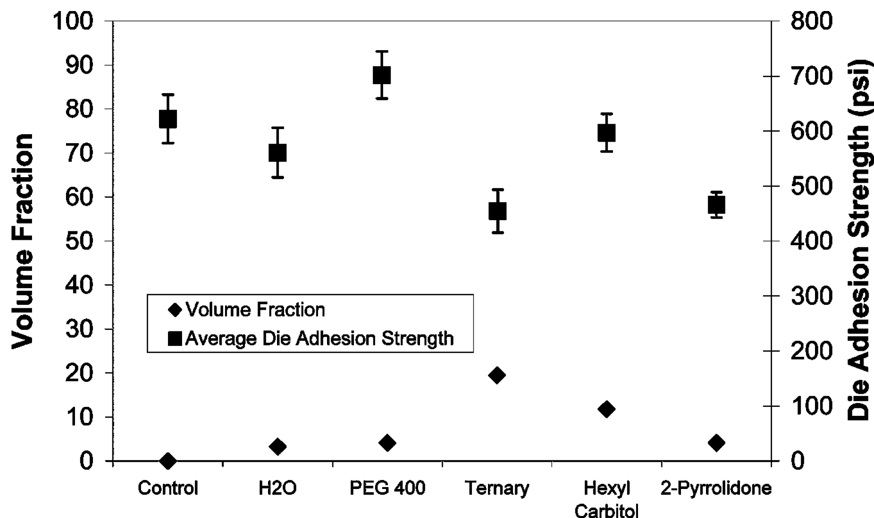

FIGURE 10 Die-adhesion strengths and sorption data for water solutions at 60°C.

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60°C. Decreases in adhesion were also observed for several of the mixtures as shown in Figure 10. The low volume fraction and the small changes in the T_g, shown in Table 4, also indicate that the solvent swelling was the critical parameter for loss in adhesion. Each failure occurred at the Rynite–adhesive interface.

The lap-shear and thin-disk test results for water solutions are best compared in Figures 6 *vs*. 9 and Figures 7 *vs*. 10. At both temperatures, the lap-shear samples failed in the Rynite substrate, whereas the die-adhesion samples failed at the Rynite–adhesive interface. The error bars on lap-shear strength simply reflect the average standard error of the Rynite strength and do not relate to the Rynite–adhesive strength. The thin-disk test is measuring the Rynite–adhesive bond and should be sensitive to differences in adhesive swelling. Both the ternary mixture and the 2-pyrrolidone systems are known to swell the adhesive, and both systems show lower die-adhesion strengths at 60°C. Although PEG 400 also swells the adhesive, its die-adhesion strength is not lower than that of the control.

CONCLUSIONS

Adhesion testing using lap-shear joints in the presence of solvents or solvent solutions requires long periods for the liquid to diffuse into

the bond line (greater than 30 days at room temperature). Even the more aggressive pure solvents, hexyl carbitol and 2-pyrrolidone, have only moderate effects on the lap-shear adhesion after 30 days' exposure at 60°C. The lap-shear joint specimens failed primarily within the Rynite, implying that adhesive failure did not occur. Also, the lap-shear test is difficult to use with different materials such as silicon.

The thin-disk test can easily incorporate both a polymer substrate (Rynite) and the silicon die into the joint. This test showed good reproducibility and accuracy for both the controls and the soaked samples. The weakest interface of the joint and the loss of adhesion as a function of time, temperature, and solution environment were easily determined. The thin-disk test, having geometric similarities to actual parts, was sensitive enough to distinguish the proper failure mechanism in short, accelerated tests. The die-adhesion method allows a system with two different substrates to be tested in geometries and scales similar to the actual part. This method could be adapted to other substrate 1–adhesive–multiple substrate 2 systems and would be particularly appropriate for thin section solids and thin adhesive layers.

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