

Ink and Moisture Sorption Study in UV-Curable Polyurethane Acrylate

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ABSTRACT: A study of the interaction of commercial black ink with a UV-curable polyurethane acrylate (PUA) is presented. Diffusivity and solubility in PUA cured using different UV dosages, and subjected to different post-UV curing conditions, were studied, as was the swelling produced by such sorption. Experiments were also conducted using water, to serve as a benchmark and compare with the effect of ink. The percentage ink absorption was found to decrease with higher degree of cure. In addition, through desorption measurements, ink induced swelling of PUA processed under different condi-

tions was determined. The degree of swelling per unit solvent uptake was observed to increase with the degree of cure. PUA was determined to be more prone to moisture rather than solvent uptake. However, the coefficient of moisture expansion (CME) was found to be ~35% lower than the coefficient of ink expansion (CIE) for a fully cured PUA. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 1985–1991, 2007

Key words: polyurethane acrylate; moisture uptake; solvent uptake; coefficient of moisture expansion

INTRODUCTION

Growing concern for environmental protection has driven industry toward the use of solvent-free polymerization systems, where UV-radiation curing has become a viable alternative to conventional thermal curing of solvent-containing polymer formulations. Added benefits of UV-cured materials including cure on demand properties, ultra fast polymerization, and combination of good material properties have led to their increased use in high-volume assembly processes.

UV-curable systems are increasingly employed as coatings in various industrial applications to provide surface protection of metals, plastics, and optical fibers. Among UV curable polymers, polyurethanes (PUs) are well-known for their combination of good physical and mechanical properties, such as adhesion to various substrates, flexibility, high chemical resistance, and impact strength, which make them ideal candidates for coating and adhesive applications.¹ Because of the above-mentioned advantages, UV cured polyurethane acrylate (PUA) is therefore used as one of the alternatives to thermal cured encapsulant such as epoxy, in

thermal inkjet print head assembly, a Si-based microelectromechanical system (MEMS).² UV-cured aliphatic polyurethanes, in particular, were found to withstand environmental degradation well, whereas their aromatic counterparts proved to be much more sensitive to UV radiation, leading to discoloration, cracking, and embrittlement upon weathering.^{3,4}

A disadvantage with polymer coatings is that they cannot provide hermetic protection; they possess the ability to transmit liquids, gases, and vapors to a greater or lesser extent. Permeability of moisture in polymers is orders of magnitude higher than in metals and ceramics.⁵ Improvements have been made in recent years to enhance the moisture resistance of PUA coatings. One method is by incorporating fluorinated block or graft copolymer within the copolymer chains. Fluorine atoms concentrate at the surface of the polymer blends, imparting strong hydrophobic character, giving rise to low-surface energy, and thus enhancing the resistance to moisture.^{6,7} The addition of small amounts of clay nanoparticles into polymer matrices has also been found to confer dramatic property enhancements; stiffness and strength, thermal stability, flame retardancy, solvent and UV resistance, and gas barrier properties^{8–10} can all be improved using this approach.

In thermal inkjet assembly applications, PUA is dispensed on exposed metal sections of flexible circuit leads. A schematic cross-sectional diagram of an inkjet printer is shown in Figure 1. The purpose of PUA is to encapsulate the conductors so as to prevent any con-

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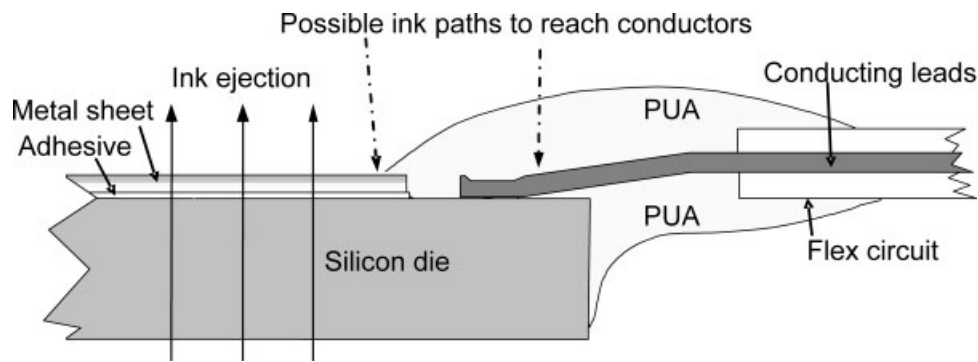


Figure 1 Schematic cross-sectional view of a typical printer head.

tact with ink or moisture from the ambient environment. Without proper insulation, ink/moisture can access the bond pads and conductors, potentially leading to corrosion related shorting between conductors.

The presence of moisture is the root cause for many package failures. On moisture permeation, polymer interfacial adhesion with the substrate deteriorates, accelerating the onset of delamination, and promoting corrosion and ionic-migration processes at interconnects underneath the coating.^{5,11,12} Moisture absorption also induces hygroscopic swelling of the polymer. Swelling mismatch between different materials in the package causes development of hygroscopic stress, which can escalate the delamination process.^{11,13,14} Exposure to ink, which is composed of a mixture of solvents and water, causes similar sorption behavior, swelling, and property changes in the polymer. Although the effect of specific solvents on sorption in polyurethane has been reported,^{15,16} thus far, there are no reports on the interaction of ink with UV encapsulants used in print head assemblies or other microelectronics packages. A study in this area will benefit the industry, either through new encapsulant evaluation methods or through better control of the assembly process.

The aim of the present work is to study transport properties, i.e., diffusivity and solubility, as well as sorption-induced strains induced when a commercial UV-cure polyurethane acrylate interacts with black ink. Sorption-induced strains were determined through measurement of coefficient of expansion during immersion in ink. For moisture, coefficient of moisture expansion (CME) is determined, which is defined as the fractional change in length per unit mass variation due to moisture desorption or absorption. A similar term, coefficient of ink expansion (CIE), is introduced here, which is defined as the ratio of length variation to mass variation due to ink absorption or evaporation. Values are derived from desorption measurements recorded using two standard thermal analysis instruments, the dynamic mechanical analyzer (DMA) and the thermogravimetric analyzer (TGA) that characterize the swelling behavior of polymers, as proposed by

Wong et al.^{17,18} The effect of UV curing intensity and post cure annealing on the sorption behavior of PUA is considered in this work. Measurements were repeated using water, to serve as a comparison to the study of ink.

EXPERIMENTAL

Sample preparation

A UV-polymerizable polyurethane acrylate (PUA) encapsulant, supplied by Hewlett–Packard (HP), was used for the study. Samples were prepared by manual screen-printing with a laser cut metal stencil onto a Teflon block, followed by UV curing. As the PUA encapsulant does not adhere to Teflon, the cured film could be peeled from the Teflon substrate to become a freestanding PUA film. The cured films have final dimensions of 6.5 mm × 30 mm and a thickness of 170 ± 10 μm, which fulfilled the JEDEC standard No. 22-A120 requirements for moisture uptake measurements, to approximate one-dimensional Fickian diffusion behavior. Curing was carried out by exposing samples to a Mercury UV lamp of wavelength range from UVB to UVV (280–445 nm). The UV dose was varied, with values of 2025, 3975, or 6525 mJ/cm² being employed. These UV dosages were obtained by adjusting the distance between PUA sample and UV lamp to achieve different UV intensities of 135, 265, and 435 mW/cm² with a constant exposure time of 15 s. This is to simulate production conditions, where UV intensity decays with operating hours of the UV lamp. The UV intensities were measured using a high energy UV integrating radiometer from EIT Inc.

Immersion/weight gain experiments

Sorption tests were conducted at room temperature (25°C) and 60°C. Before the immersion test, freshly cured films were aged in ambient atmosphere for 3 days, followed by 2 days drying at 40°C. Sorption experiments were performed by immersing PUA samples in 20 mL of black ink, contained in screw-tight

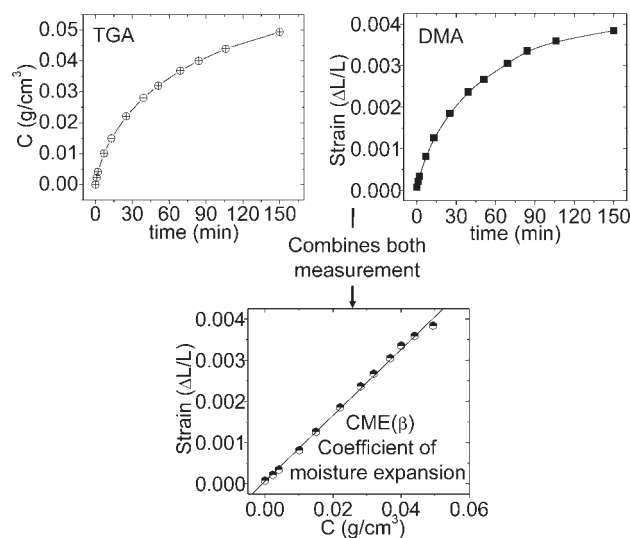


Figure 2 Process for determination of hygroscopic swelling properties.

bottles and kept in a thermostatically controlled oven ($\pm 1^\circ\text{C}$) at the desired temperature. The black ink used in this study is extracted from HP print cartridges (HP 45 Inkjet Print Cartridge, Black (PN: 51645A)). The ink is an aqueous-based formulation consisting of < 28% mixture of carbon black, isopropyl alcohol, 2-pyrrolidone, and trade secret materials. Samples were periodically removed from the ink, wiped with a particle free cloth to remove surface attached solvent and pigments, and weighed using a high accuracy (± 0.0001 mg) analytical balance from Mettler Toledo. The whole measurement process was completed within 50 s after removal from the temperature-controlled oven, to minimize solvent evaporation. Measurements were continued until the PUA samples attained saturation level, which took more than 5 days. Five samples were used for each test condition. The weight gain during water absorption was measured in similar manner using deionized water instead of ink.

CIE and CME measurement

After 2 days drying at 40°C , samples were removed from the oven and soaked in black ink until saturated. Samples were removed from bottles and transferred immediately to the DMA chamber, to measure dimensional changes using the film–fiber in tension mode setting with a controlled static force of 0.001 N and clamping distance of 20 mm. Other samples were transferred to the TGA to measure the rate of solvent weight loss. Both tests were conducted at laboratory temperature ($23.5 \pm 1^\circ\text{C}$). The time taken to transfer samples from bottle to equipment was minimized to prevent excessive solvent evaporation. DMA data provides the rate of change of dimension ($\Delta L/\Delta t$) while TGA data gives the rate of change of moisture

weight loss ($\Delta M/\Delta t$). By extracting DMA and TGA data at the same desorption time intervals, CME or CIE was determined from the gradient of the strain versus concentration curve, as illustrated in Figure 2.

The impact of post annealing was studied by subjecting cured samples to 80°C for 12 h after UV treatment.

RESULTS AND DISCUSSIONS

Ink sorption at 25°C

Figure 3 shows the ink sorption trend at 25°C for PUA cured using different UV doses. Initially absorption occurs rapidly, reaching a plateau at longer times. Samples that had received a higher UV dose were found to exhibit lower weight gain. In previous work, it was determined that lower UV dose yields lower degree of cure,¹⁹ which results in a less compact intermolecular packing and more microvoids. With increased intermolecular packing in samples cured at higher intensity, less sorption spaces are available in the matrix to accommodate solvent molecules, thus inhibiting their penetration into the PUA.

Sorption data were analyzed in terms of an empirical relation to establish the diffusion mechanism:²⁰

$$\log(M_t/M_\infty) = \log k + n \log t \quad (1)$$

where M_t is the mass uptake at time t ; M_∞ represents the corresponding equilibrium value; k gives an indication of the extent of polymer–solvent interaction; n is a numerical value from which the transport mechanism can be established. If the estimated value of n is close to 0.5, results are indicative of a Fickian mode of transport.¹⁵ By plotting $\log(M_t/M_\infty)$ vs. $\log t$ (Fig. 4), n is computed from the gradient of the line. All three trendlines show very good R^2 correlation, with gra-

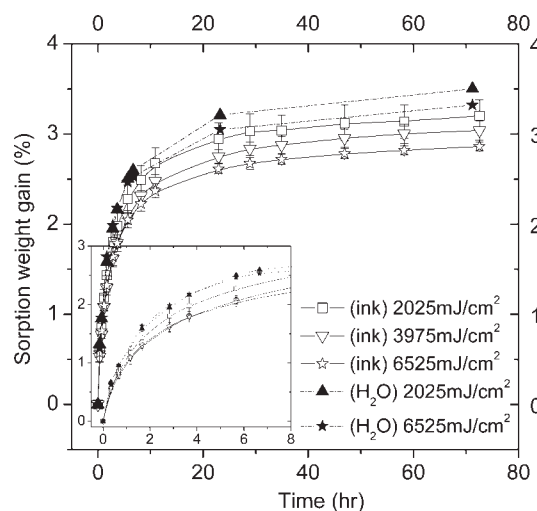


Figure 3 Ink and moisture uptake test results at 25°C .

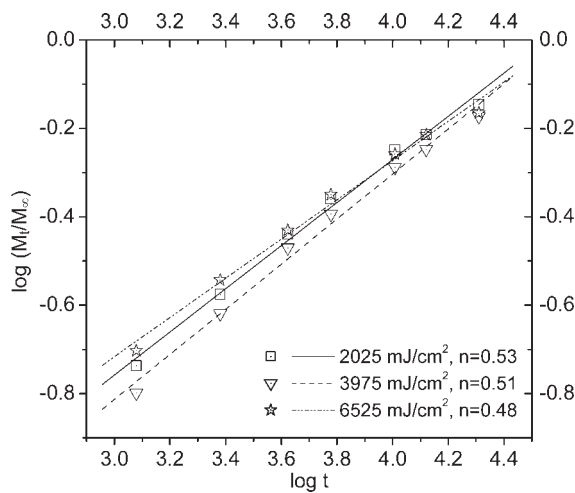


Figure 4 Plot of $\log (M_t/M_\infty)$ vs. $\log t$ for ink uptake test at 25°C.

dients from 0.48 to 0.53, suggesting that the transport of ink can be classified as Fickian type at 25°C.

Maximum solubility C_{SAT} is calculated from:²¹

$$C_{SAT}(T, \%RH) = \frac{\text{WetWt}(f) - \text{DryWt}}{\text{Vol}} = \frac{M_{SAT}(T, \%RH)}{\text{Vol}} \quad (2)$$

where $C_{SAT}(T, \%RH)$ is the solubility at temperature T and percentage relative humidity %RH (mg/cm^3); WetWt (f) is the final wet sample weight (mg); DryWt is the final dry sample weight (mg); Vol is the sample volume (cm^3); and $M_{SAT}(T, \%RH)$ is the saturated solvent content at temperature T and %RH (mg/cm^3). In this study, C_{SAT} was measured at room temperature and 100%RH.

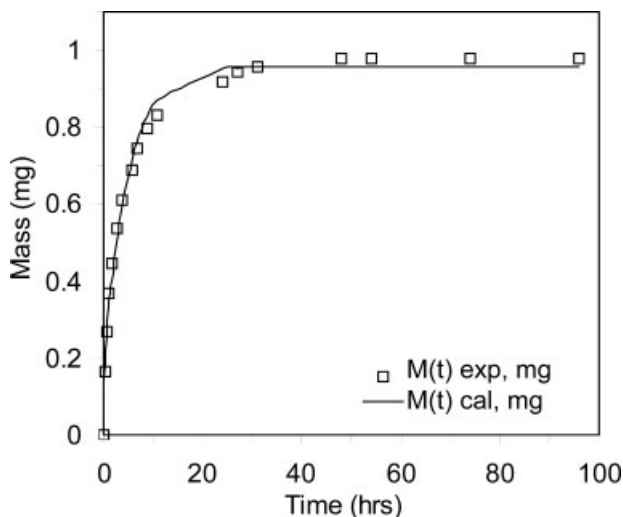


Figure 5 Curve fitting using eq. (3) for PUA encapsulant cured at 3975 mJ/cm^2 to determine the diffusivity of ink.

TABLE I
Diffusivity D and Solubility C_{SAT} (100%RH)
Values from Ink Sorption Tests

UV dosage (mJ/cm^2)	25°C		60°C	
	D (mm^2/s)	C_{SAT} (mg/cm^3)	D (mm^2/s)	C_{SAT} (mg/cm^3)
2,025	1.57E-07	34.24	2.00E-07	45.37
3,975	1.55E-07	33.92	1.51E-07	45.30
6,525	1.51E-07	30.27	1.35E-07	42.61

Diffusivity D at 25°C was determined by the best-fit curve fitting approach from experimental weight gain data, using the following equation,^{21,22} where h is the thickness of the polymer sheet.

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[\frac{-(2n+1)^2 \pi^2 D t}{h^2}\right] \quad (3)$$

Figure 5 shows the curve fit for PUA encapsulant cured at 3975 mJ/cm^2 . Square blocks represent experimental data collected from the ink absorption test. Data points were best-fitted using eq. (3) to obtain the diffusivity value.

Table I summarizes diffusivity and solubility values calculated for ink uptake at different temperatures. Samples cured at higher UV energy show lower diffusivity and solubility values, which are attributed to the reduction in mobility of polymer chains with increasing degree of crosslinking. This leads to greater activation energy for solvent transfer between absorption sites and restricts the movement of penetrant.²³ It also results in a reduced number of sorption spaces, as discussed earlier, and hence lowers solute ingress.

Ink sorption at 60°C

The absorption trend at 60°C (Fig. 6) is different from that observed at 25°C. Weight increases during the ini-

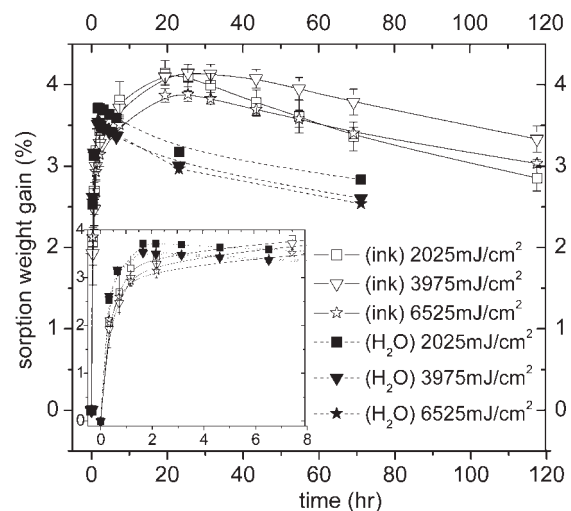


Figure 6 Ink and moisture uptake test results at 60°C.

tial stage of ink absorption, until a maximum point is reached and a gradual reduction in weight is recorded thereafter. Mode of transport was determined by plotting $\log (M_t/M_\infty)$ versus $\log t$ to attain values for n , as shown in Figure 7. n values were found to fall between 0.20 and 0.31, suggesting that diffusion at 60°C deviates from Fickian type. This indicates that different reactions may occur at elevated temperature.

Owing to the non-Fickian absorption behavior at 60°C, eq. (3), which suggests an asymptotic nature for moisture sorption where a practical saturation limit must be defined for characterization, becomes inappropriate. A 99% saturation approach, is used to calculate the diffusivity at 60°C^{11,24}

$$t_{99\%} = \frac{0.45 \times l^2}{D} \quad (4)$$

The 99% saturation approach helps to define the limit of Fickian diffusion, hence eliminating error caused by non-Fickian sorption. Calculated values for D and C_{SAT} are compiled in Table I, maximum solubility was calculated from the maximum values recorded in the ink sorption test before the commencement of weight loss. Owing to the weight drop at 60°C, values for D and C_{SAT} calculated might not reflect the actual values. Possible reasons for the weight drop and the shifting of absorption behavior to non-Fickian are discussed in a later section.

Comparison of ink absorption with moisture sorption

Ink sorption is compared to moisture sorption at 25 and 60°C in Figures 3 and 6 respectively. PUA is seen to be more prone to moisture rather than ink ingress, as signified by the measured higher weight gain percent recorded in DI water. As weight gain plots are

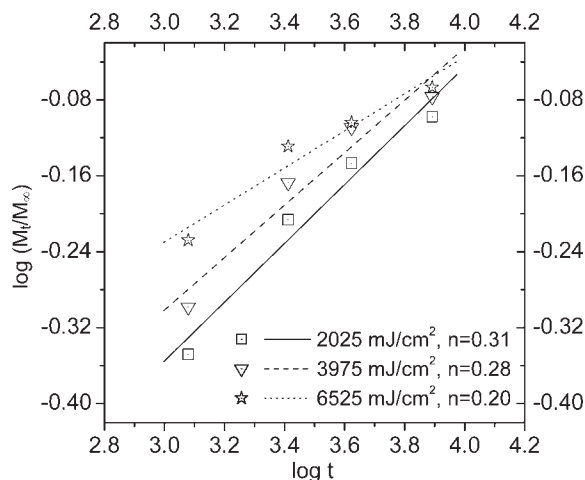


Figure 7 Plot of $\log (M_t/M_\infty)$ vs. $\log t$ for ink uptake test at 60°C.

compared without considering the density effect, the volumetric uptake would be varied if water and ink have different densities. The density of ink, from which the pigment had been removed, was found to be 0.9898 g/cm³. Given that the densities of moisture and ink are very close, plotting respective volume uptake versus time would yield little difference to the final conclusion.

The difference in weight gain between ink and moisture can be explained in terms of the molecular size of the solvents. Water has relatively small molecular size when compared with the monomer unit of PUA. A limited rotational oscillation of only one or two monomer units would be sufficient to give a cross section for the water penetrant to jump thermally from one position to a neighboring one, allowing the molecules to penetrate more easily into the polymer matrix.²³ Furthermore, its smaller size also allows more water molecules to cluster and reside within one PUA sorption site. In moisture saturated polymethyl methacrylate (PMMA), it was deduced that each sorption site can accommodate about 5 molecules of water.^{25,26} However, for ink, which is composed of a mixture of organic solvents, the molecular size is larger than water, so fewer molecules can be accommodated at the sorption sites available in the PUA and hence lower absorption is observed.

Experiments to elucidate the mechanism for weight drop trend at 60°C in absorption measurements and non-Fickian transport behavior in Figure 6 were carried out. One mechanism proposed was the leaching of low-molar mass indigenous compounds from the encapsulant sample in the presence of solvent.²⁷ To verify this theory, UV spectra were recorded for DI water in which encapsulant samples had been immersed at 60°C for 3 days. Pure DI water allows close to 100% transmission across the whole wavelength range. However, for water in which PUA was soaked, light transmission dropped to 0% below 260 nm (Fig. 8), indicating that materials leached out of the film during soaking. This lends support to the hypothesis and provides a possible explanation for imprecision in the calculations of D and C_{SAT} . As ink is non-translucent, UV-vis testing could not be carried out. Since both ink and moisture gave same sorption behavior at 60°C, it may be postulated that the same leaching effect may occur during ink sorption experiments.

Further analysis was performed on the final weight drop percentages tabulated in Table II and shown in Figure 6. A decreasing trend in the final weight drop percentage with increasing UV intensity was observed. This can be attributed to the fact that stronger UV intensity yields more complete PUA curing and increases the density of intermolecular packing. The more densely packed and crosslinked structure serves as an effective barrier, reducing the content of low

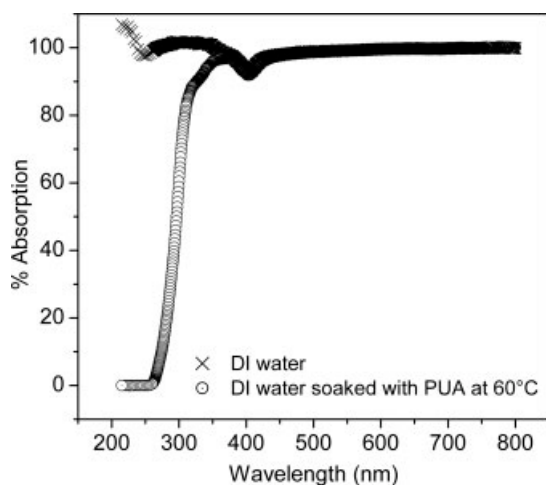


Figure 8 UV-vis spectra of DI water with and without soaking with PUA for 3 days at 60°C.

molecular weight species and preventing the material from leaching out. Hence, reduced weight loss is observed for higher intensity curing.

Coefficient of ink expansion

From DMA (swelling) and TGA (weight loss) measurements, CIE values were calculated and are compiled in Table III. The effect of UV dose and post cure annealing are shown in Figure 9. It has been reported previously that swelling strain will increase with absorbed moisture content;²⁸ for PUA samples UV cured at 6525 mJ/cm², the percentage solvent absorbed was less than for those cured at 2025 mJ/cm², but the rate of increase in swelling with concentration of absorbed solvent was higher. With increasing degree of cure, a more densely packed and cross-linked polymer network is formed, leading to lesser sorption spaces and reduction in solvent diffusion through the polymer chains. Consequently, larger distortion strain and expansion in the network is expected upon absorption.

When a post cure annealing step is introduced, residual crosslinking reactions are accelerated, increasing the degree of cure and hence the CIE value. This is clearly exhibited by the samples cured at 2025 mJ/cm² UV energy with and without annealing (Fig. 9), where

TABLE II
Difference Between Maximum Weight and Final Weight of the PUA Samples During the Ink Sorption Test at 60°C

UV dosage (mJ/cm ²)	Max wt gain (%)	Final wt gain (%) (at 121 h)	Δ wt %
2,025	4.27	2.99	1.28
3,975	4.21	3.30	0.91
6,525	3.96	3.09	0.87

TABLE III
Compiled CIE and CME Values

Sample condition	Total intensity (mJ/cm ²)	Coefficient of ink expansion (CIE)	Coefficient of moisture expansion (CME)
No anneal	2025	0.075	0.070
	6525	0.144	0.090
Anneal	2025	0.117	0.076
	6525	0.142	0.090

the CIE value increases substantially due to greater increase in the percentage cure during the annealing process. For samples cured at 6525 mJ/cm², since the PUA encapsulant has already attained a relatively high degree of cure, further annealing therefore has little impact on degree of cure and swelling behavior.

Comparison with coefficient of moisture expansion

Ink- and moisture-induced swelling are compared in Figure 10. Although moisture absorption levels in PUA are higher, moisture has less impact on the induced swelling than solvent. Measured CME values were 35% and 36.6% lower than the respective CIE figures for UV doses of 2025 and 6525 mJ/cm² respectively. This difference may be attributed to the molecular size difference between moisture and ink, as mentioned previously. Water molecules require only limited rotational oscillation of only one or two monomer units to jump thermally from one position to a neighboring one. By contrast, a larger movement of polymer segments is required for solvent permeation into the polymer matrix. Thus, solvent ingress will

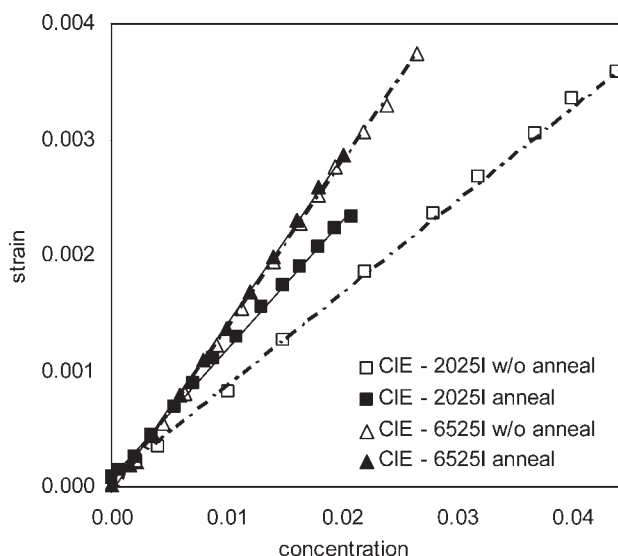


Figure 9 Plots showing linear relation between swelling and ink content—comparison between annealed samples and those subjected to different degrees of cure.

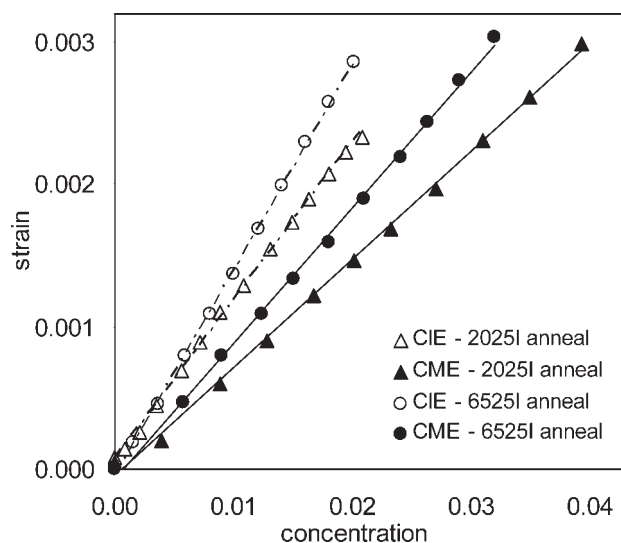


Figure 10 Plots showing linear relation between swelling and moisture/ink content—comparison between CME and CIE.

introduce larger distortional strains in the matrix during permeation, expanding the polymer network and leading to greater swelling effects.

CONCLUSIONS

Ink and moisture sorption characteristics of a UV-curable polyurethane acrylate, used in inkjet print head assemblies, have been investigated. The impact of different penetrants, exposure temperature, UV dose and post cure annealing on ink sorption trends, and on PUA swelling was studied. Two sorption related parameters studied for PUA, diffusivity D and maximum solubility C_{SAT} , decreased for PUA cured with higher doses of UV, which exhibit higher degrees of cure and increased crosslinking density.

Deviation from Fickian absorption behavior in ink and moisture at 60°C was attributed to material leaching from the samples. Samples cured at high UV dosage are subjected to lower ink absorption, but at the expense of higher sorption induced swelling, as determined from CIE measurements.

A higher swelling coefficient often results in greater stresses in the encapsulants, promoting defects such as delamination, which are detrimental to the reliability of the MEMS-based inkjet print head devices. Curing conditions adopted and their influence on the swelling coefficient should be taken into consideration to ensure reliable PUA encapsulation. Previous experimental works proved^{19,29} that the effect of annealing is beneficial, helping to increase the degree of cure and interfacial adhesion strength. It is hence recommended to subject PUA encapsulant to an annealing step to achieve optimum conversion for product reliability.

The PUA was found to be more prone to moisture rather than ink ingress, as indicated by higher uptake trends. Nevertheless, moisture has a lesser impact on swelling; the CME values measured being ~ 35% lower than CIE values for annealed samples.

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