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Adhesion Characterization of micro components fabricated by Micro stereo lithography

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Abstract

Interfacial adhesion of UV curable polymers is important in Micro stereo lithography (MSL) due to the sticking problems during separation from their substrate and insuring adhesion between two dissimilar materials used in micro-fabrication. Therefore, standardizing quantitative ranking among different substrates helps to select the required substrate for fabrication of polymers according to their application. In this report, Nano Indentation techniques is proposed and used to perform experiment on interfacial adhesion of HDDA (1, 6 Hexanediol Diacrylate) polymer over Teflon Substrate. The Nano-indenter is used to perform bending test on HDDA polymer from which the interfacial adhesion is easily analysed. Finally, interfacial adhesion of HDDA on Teflon is obtained from the computerized Nano-indenter machine and measured with an appropriate measurement unit.

Keywords- Adhesion, Micro stereo lithography (MSL), UV, HDDA

I. Introduction

In recent years, there is a rapid progress in Nano/micro fabrication for the purpose of highly functionalized devices with superior mechanical and chemical properties, and it becomes interest-attracting issue continuously for neo-conceptive applications. Micro stereo lithography (MSL) is becoming an important fabrication technique in rapid prototyping technology used in the automotive and aero-space industries: for example, in fabrication of micro-gears, and micro-turbines. Furthermore, Micro stereo lithography is essential in all manufacturing areas where three dimensional micro-prototypes are required. The majority of micro-fabrications have been carried out, thus far, using organic polymers, for example, SU-8, AZ9260, urethane acrylate, and many others [1, 2].Characterization of micro components fabricated by MSL techniques helps to know all the necessary properties of micro components. Properties of micro components fabricated in MSL are important in design of microstructures, and selecting appropriate substrates for fabrication of micro-component according to their application. Some of important properties which are required in microstructure design and selecting working surface include: hardness, young modules, poison's ratio, and interfacial adhesion. This report concentrates on interfacial adhesion of polymers. Objective of this research paper was to standardize and quantify adhesion between HDDA polymers over Teflon substrates which is helpful in MSL fabrication process. Quantitative adhesion helps in ranking among the substrates tested or to set a numerical specification for adhesion strength that can be subsequently used as a standard. The numerical specifications for the interfacial adhesion between the substrate and the sample enable one to select the appropriate substrate for the fabrication of micro- components and micro structures according to their application; whether they are needed to stay stuck to the substrate or they are going to be delaminated and installed into another complicated microstructure. The scope of the study involves preparation of the samples over different substrates and carried out quantitative evaluation of the interface strength in terms of interfacial adhesion force (N), and interfacial adhesion energy (J/m^2).The samples are fabricated using MSL fabrication technique and then subjected to beam bending test using the scratch testing utility in nano-indenter. The Quantitative value of the interfacial adhesion of the HDDA polymer over Teflon substrate is evaluated.

II. Adhesion

Adhesion can be defined in different way. The precise meaning of the term is entirely dependent on the details of the measurement technique employed and the experimental and environmental condition under the measurement was made. Quantitatively, we can say the one material have good adhesion to another material based on the observation that both the material were observed remain stuck to each other under a variety of loading conditions. Quantitatively, adhesion can be defined in terms of different parameters. These definitions include:

A. Coefficient of friction at which delamination begins [1].
B. The interfacial energy of a coating-substrate system which is the energy needed to propagate a crack along the interface between the coating and the substrate per a unit area [2].

C. Force which can be described as the force needed to separate two bodies along their interface, and it is restricted therefore to the interfacial forces acting across the interface [3].

In this study, adhesion is measure by both the energy per unit area released to debond the component from its substrate, and the maximum force required to delaminate the component from its substrate for a specified interfacial area. Interfacial adhesion occurs when there are atomic interactions between two different materials [7]. The main reasons for these interactions are arisen from [3] Mechanical retention, Secondary forces, Chemical bonding. Mechanical retention arises from either mechanical interlocking of phases or interfacial diffusion of matter; whereas, second forces are interatomic forces acting across the interface of the given materials. Along the interface of two materials there is also formation of covalent and ionic bonds. In general, Interfacial adhesion is rarely a result of secondary forces, chemical bonding, or mechanical retention alone but is a combination of these [8].Two common approaches of linear elastic fracture mechanics (LEFM): stress intensity approaches, and energy release rate approach are going to be discussed. The stress intensity factor approach states that fracture toughness should be measured in terms of resistance to crack propagation [5]. According to this approach, crack initiates on a material at the time the applied stress intensity factor, *K*, exceeds a critical value, *Kc*, material property. In general, three modes of loading can be applied to the crack; Mode I as shown schematically in Figure 1 is opening mode, where the crack surfaces move directly apart. Mode II, the sliding mode, which refers to a shear stress applied in plane of the crack but normal to the leading edge of the crack. Mode III, tearing mode, is for shearing stress applied parallel to the leading edge of the crack. The components of the stress field near the crack tip can be written in the following simple form [6]:

$$\sigma_{ij} = \frac{K_I * f_{ij}(\theta)}{\sqrt{2\pi r}} \quad \ldots\ldots (1)$$

Where σ_{ij}= Stress component *i, j*
r = Radial coordinate measured from crack tip
θ = Angular coordinate about crack tip K_I =
Mode I stress intensity factor

$f_{ij}(\theta)$=Elementary function of trigonometric expressions

When we look at Equation 1, the reciprocal square root dependence of the stress components on the radial distance from the crack tip is the source of the singular behaviour. If the stress function is integrated over small volume containing the crack tip, the result is finite. For elastic solid, the energy associated with a given stress field can be written as [6]:

$$U = \frac{1}{2}\int_{v_0}^{v} \sum ij\ \sigma_{ij}\ _{ij} \mathrm{dv} \quad \ldots\ldots (2)$$

Where, The volume *V* covers a small region containing the crack, is the strain field, and i and j are integers.

The strain field near the crack tip ε_{ij} is given by:

$$\varepsilon_{ij} = \frac{1+v}{E}\sigma_{ij} = \frac{V}{E}\delta_{ij}\ tr(\sigma) \quad \ldots\ldots (3)$$

where, $tr = \sigma_{11} + \sigma_{22} + \sigma_{33}$ ..... (4)

Strain energy release rate is related with the stress intensity factors as follows:

$$G_1 = (1 - v^2)\frac{K_I^2}{E} \quad \ldots\ldots (5,a)$$

$$G_2 = (1 - v^2)\frac{K_{II}^2}{E} \quad \ldots\ldots (5,b)$$

$$G_1 = (1 - v^2)\frac{K_{III}^2}{E} \quad \ldots\ldots (5,c)$$

Where *v* is poison's ratio and *k* is stress intensity for the three modes.

The total strain energy release rate G is determined by adding all the three components. At time of fracture the energy release rate G will be equal to the critical fracture energy, $G_c\left(\frac{J}{m^2}\right)$.

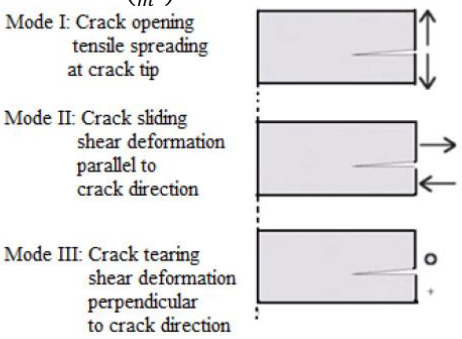

Fig. 1: Three basic mode of crack propagation

The energy release rate approach states that crack extension takes place when there is enough energy to overcome the resistance of the material [4]. An adequate amount of elastic energy stored near the crack tip is required for crack propagation. When strain energy is increased beyond a certain critical level, then sufficient energy is available to create new surface area, and the crack can propagate. The basic principle of beam subjected to bending is schematically shown in Figure 2. The

material resistances are surface energy, plastic work, and all the energy dissipated during crack growth. The work done by the external load is then equal to the material resistance. In the case of a beam, external work (W) is given by:

$$W = \frac{1}{2}F\delta \quad \dots (6)$$

Where, is displacement of the beam at the point of application of the load and F is the applied force.

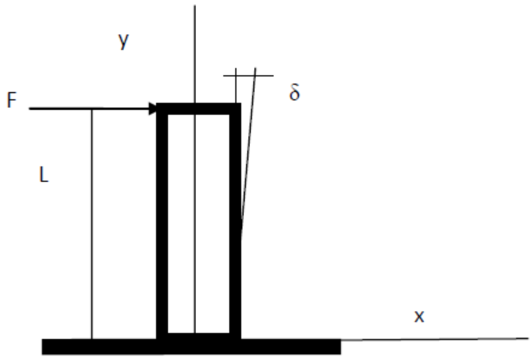

Fig. 2: Beam subjected to bending

Energy balance approach states that there is no change in the total energy (E) of the cracked body during a quasi-static increment of crack area, dA. Due to the application of the external load, potential energy (U) is induced on the material. When the stored potential energy reaches a critical value to overcome the surface energy (S), crack starts to propagate. Therefore, the total energy (E) is composed of the potential energy of deformation (U) and the surface energy (S). From the principle of conservation of energy, during crack extension:

$$dE = dU + dS = 0 \quad \dots (7)$$

For linear elastic material, the rate of change of potential energy with respect to crack area, dA, is equal to the energy release rate. The work done by the external forces is stored as elastic energy in the beam and released during delamination. Therefore, the energy required to remove the beam from its substrate is given by:

$$U = \int_0^L F dx \quad \dots (8)$$

And the interfacial adhesion energy which is the measure of the critical energy release rate or fracture energy is calculated from:

$$G_C = -\frac{dU}{dA} \quad \dots (9)$$

The energy release rate can also be calculated from the stress intensity factors as follows:

$$G = \frac{1}{2}(K_I^2 + K_{II}^2) + \frac{1}{2\mu}(K_{III}^2) \quad \dots (10)$$

Where E and μ are and are Young's modulus and shear modulus consecutively.

III. Sample Preparation-

Micro stereo lithography is used in fabricating square HDDA sample (0.5 x 0.5 mm) on the substrate of Teflon. The basic principle of Micro stereo lithography is schematically shown in Figure 3. A 3D solid model designed with CAD software is sliced into a series of 2D layers with uniform thickness. The code generated from each sliced 2D file is then implemented to control a motorized x–y stage carrying a container of UV curable solution. The focused scanning UV beam is absorbed by an UV curable solution consisting of monomer called HDDA and photo-initiator BEE (benzoin ethyl ether), leading to polymerization. As a result, the HDDA layer is formed according to each sliced 2D file. After one layer is solidified, the elevator moves downward and a new layer of liquid resin can be solidified as the next layer. With the synchronized x–y scanning and the Z-axis motion, the complicated 3D micro part is built in a layer by layer fashion. The laser wavelength used in the experiment is 351 nm from an Ar+ laser.

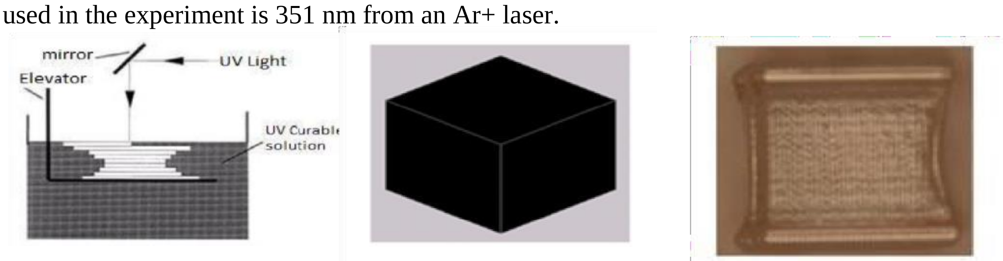

Fig. 3(a) The principle of micro stereo lithography [5], (b) HDAA sample model, (c) Optical microscope picture of the sample (0.5 X 0.5 X 0.7 mms).

After 7 layers HDDA samples are fabricated with 7µm line spacing and 0.8 mm/s scanning speed, they are taken for development with acetone. Finally, after giving enough time for drying; optical- microscope is used to take picture of the samples.

IV. Experimental Setup and Methods

The modified scratch testing is an improvement on the conventional scratch testing in such a way that there is no need of scratching on the sample. The modified testing employs Hysitron TI 900 model nano-indenter to apply lateral load on the sample. The sample is pushed horizontally while the normal load is kept as minimum as possible by setting the normal displacement to zero as schematically shown in Figure 4. (b). The modified testing is similar with a topple beam testing [6] except the applied force is uniformly distributed along theight of the sample and there is scratching on the substrate.

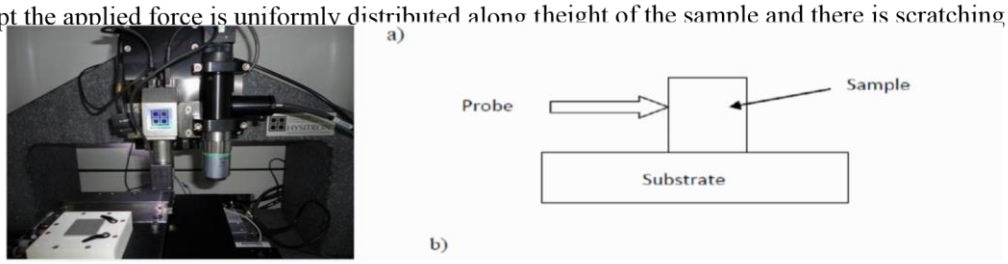

Fig.4. (a) Photo Hysitron TI 900 (b) Diamond needle pushing the sample

After attaching the samples, squares on a magnetic support with glue, they are put on the stage of the TI 900 hysitron indenter for experiments. The experiments were carried out under continuously applied load using an indenter speed of $12\mu m/sec$ until the sample is determined from its substrate. The loading is displacement controlled mode with a maximum lateral displacement of $300\mu m$. Optical Microscope installed with hysitron indenter is used to focus the indenter on the surface of the substrate close to the sample; so that sample can be pushed gently to bend. As long as the interfacial adhesion is largely related with the lateral force, the normal force was kept small. Many beam bending tests have been performed for measuring adhesion to take the advantage of relatively simple mechanics of elastic beam. Beam mechanics is simplify the analysis for stress intensity factors and strain energy release rates that drive the delamination process.

V. Results and Discussion

As Figure 5 shows the probe has touched the sample after 8 seconds and 2 seconds later the lateral force is observed to be decreased which shows the sample is detached from its substrate. The constant force after delamination is scratching force of the probe on the Teflon surface. Further experiment using the same setup with the same probe speed on the same sample showed variation in lateral force when compared to the previous experiment. Comparing Figure 5 and 6 the lateral force on second experiment is smaller than the previous experiment: which is another reason that helps one to conclude that the sample is delaminated. Furthermore, the fast increase in the lateral force in second experiment shows the lateral force is due to scratching as scratching is almost the same starting from the scratching point up to the end.

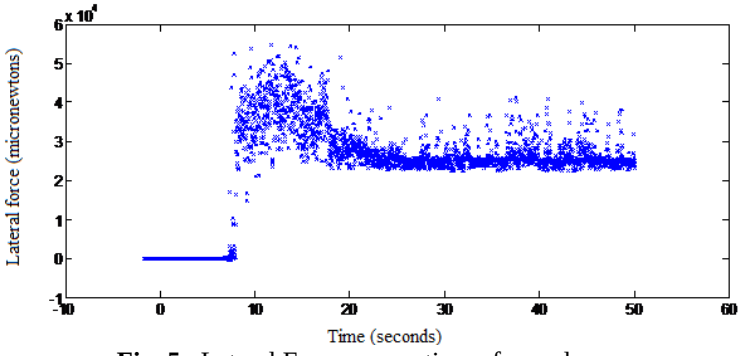

Fig. 5. Lateral Force versus time of sample one

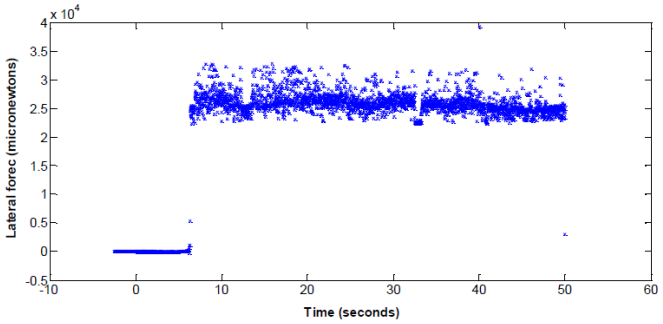

Fig. 6. Lateral force versus time of sample one after delamination

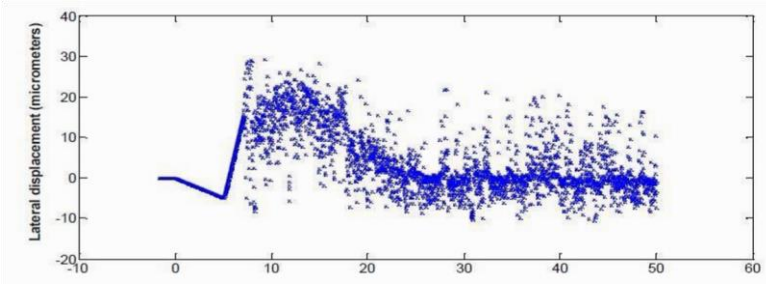

Fig. 7. Lateral force versus time plot of sample one

Even though the normal force is set to zero, the transducer was sensing normal force which could be from roughness of the substrate.

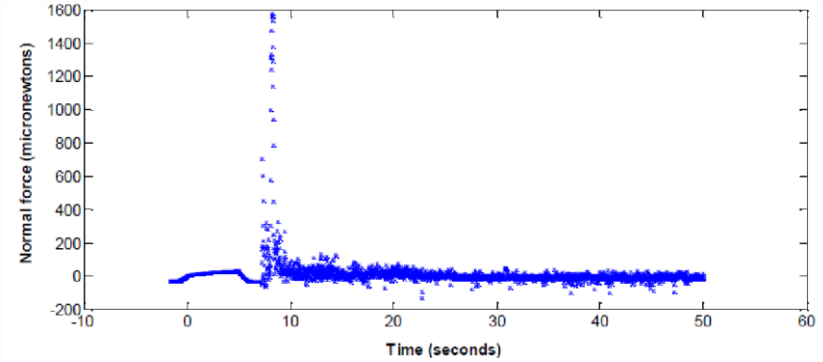

Fig. 8. Normal Force versus time plot of sample one

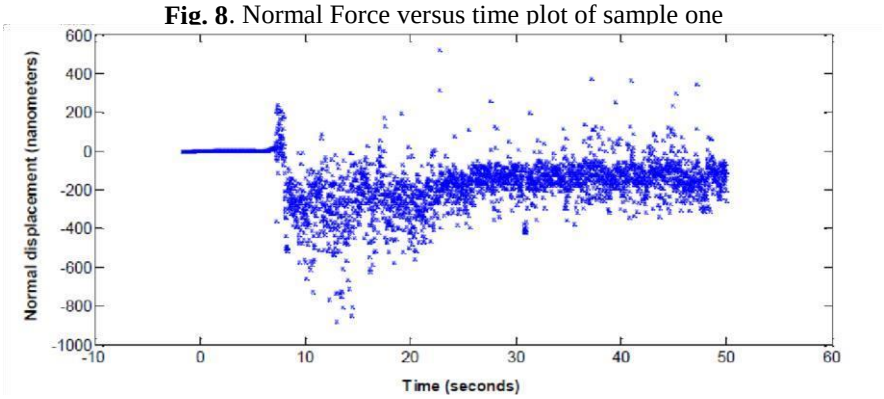

Fig. 9. Normal displacement versus time of sample one

The lateral force versus lateral displacement graph, Figure 7, which shows lateral force increases with deflection of the sample, helps to calculate the interfacial adhesion energy. The total energy required to remove the HDDA sample from Teflon substrate is the total energy released by the lateral force minus the energy dissipated in scratching the Teflon surface. Sin ce it is difficult to know the energy dissipated in scratching the Teflon; the result in the table below is the total energy released by the lateral force. But we can find the interfacial adhesion force by subtracting constant force from the maximum force in Figure 5. The calculated interfacial adhesion force obtained is about **28 MN**. This makes interfacial adhesion to be easier for the analysis when evaluated by the force required to delaminate the sample from its substrate.

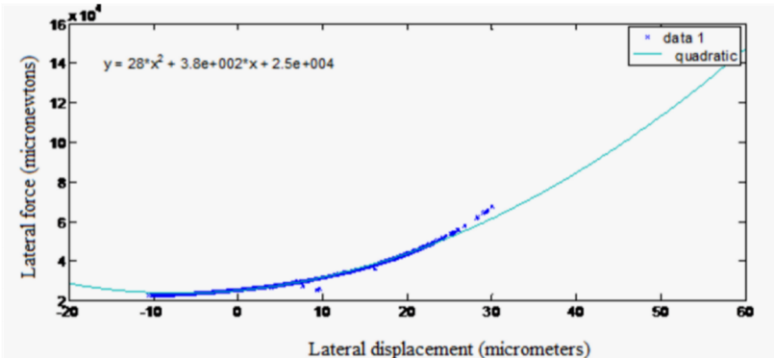

Fig. 10. Lateral Force versus deflection of sample one

The graph in Figure 10 is the lateral force versus deflection of the sample until delamination takes place. The data after the sample is detached from its substrate is not included as it is not important in the curve fitting for calculation of the total energy dissipated. The calculation for the total energy released by the lateral force is done using the fracture mechanics energy release rate approach Equation 8; which can be obtained by calculating the area under the quadratically fitted curve Figure 10. The total released energy per unit area (J/m^2), given in Table.1, is then calculated using Equation 9.

Table.1 The interferential adhesion energy (J/m^2) obtained from the beam bending test

Sample No	Material	Substrate	Interfacial Area($x10^{-7}m^2$)	Energy released($x10^{-7}$ Joules)	Interfacial energy(J/m^2)
1	HDDA	Teflon	2.5	7.56	3.02
2	HDDA	Teflon	2.5	7.56	3.0
3	HDDA	Teflon	2.5	7.54	3.0
4	HDDA	Teflon	2.5	7.55	3.01
5	HDDA	Teflon	2.5	7.55	3.01

After the samples are delaminated, the bottom surface of the samples is rougher than their upper surface. The roughness of the bottom is risen either by the roughness of the substrates or by the stepping effect due to the Gaussian laser beam distribution on HDDA polymer during fabrication. An optical microscope picture of the bottom surface of HDDA polymer after delamination is shown in Figure 11.

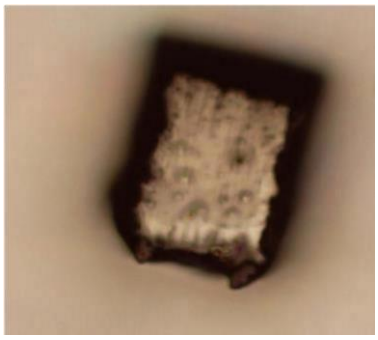

Fig. 11. Optical microscope image of the interfacial surface of HDDA after delamination.

VI. Conclusion

The report presented Nano indentation techniques to determine adhesion of polymers: modification of scratch testing to perform beam bending test of the sample. This technique avoids the complexity of highly nonlinear viscoplastic deformations, serious problem in conventional scratch testing, which arise from the act of pushing of the stylus into the sample. The closeness of the results obtained from modified scratch testing with the results from voice coil actuator inspires to standardize the proposed techniques as measuring adhesion of UV curable polymers. This technique has also an important role in detaching microstructures and mechanism, to be installed in an integrated system of components, from their substrates safely. Based on the results on adhesion of HDDA with Teflon, this is the best choice to use it as a substrate for polymers which are going to be detached and assembled with other components. Study of change in adhesion strength of UV curable polymers due to their phase change from liquid state to solid state during photo-polymerization. Optimization of MSL laser power for strength of adhesion, resolution, and quality of the component to be fabricated. Identification of the failed surface near the interface in beam bending test.

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