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Laser-Induced Forward Transfer Using Triazene Polymer Dynamic Releaser Layer

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Abstract. This article presents a short review of the use of triazene polymer as a dynamic release layer (DRL) for laser-induced forward transfer (LIFT), before looking at the latest research in more detail. The field of triazene polymer ablation only started around 20 years ago and has grown rapidly into a number of different application areas. Most promisingly, triazene ablation has been refined as a method for propulsion, bringing the benefits of LIFT to the deposition of sensitive transfer materials. The key to understanding LIFT with a triazene DRL is to understand the more fundamental nature of triazene polymer ablation in both frontside and backside orientations. This article focuses on the most recent experimental results on LIFT with a triazene DRL: the effect of picosecond pulse lengths compared with nanosecond pulse lengths; the effect of reduced air pressure; and the improvements in transfer in terms of range of transfer materials, and transfer across a gap. The results all help improve fundamental understanding of triazene-based LIFT, and the transfer of functioning OLEDs demonstrates the capability of the technique.

Keywords: triazene polymer, laser transfer, LIFT, OLED

PACS: 81.16.Mk

INTRODUCTION

Laser-induced forward transfer is a precision deposition technique utilizing the high-energy and fine-focussing of laser beams. The use of an intermediate layer, such as a triazene polymer film, to absorb the energy and provide the mechanical energy for propulsion without allowing light to impinge upon the transfer material can help minimise energy transfer into the transfer material. This extends the benefits of LIFT to more sensitive and structured thin film layers that cannot otherwise be deposited in such a controlled manner. To start off with we will look at triazene polymer ablation, before reviewing the history of LIFT and how these two separate research fields of converged to develop LIFT using an intermediate triazene polymer DRL.

Triazene Polymer Ablation

The fundamental aspect of this method is the clean laser-ablation of the triazene polymer. In the field of polymer ablation, triazene-based chemicals were originally used as dopants for the ablation of other polymers^{1, 2} using 308 nm wavelength light from the common XeCl excimer laser. Fairly soon, the group at Bayreuth incorporated the triazene chromophore into polymers themselves, firstly as a PMMA co-polymer with triazene side-chains³, and then with the triazene chromophore along the polymer backbone⁴ to improve the efficiency and cleanness of the polymer-ablation process. These novel polymers proved to be much more precise and clean in their ablation when compared to the doped PMMA⁵. In addition to the

high photosensitivity, it was seen that triazene polymers also exhibit remarkably high thermostability, up to 230 °C⁶.

Developments in triazene ablation

The ablation of triazene polymers was initially seen as a promising photoresist technology⁷. However, the photosensitivity of the standard triazene polymers is matched by their sensitivity to an acidic wet-etching processes⁸. This led to further structural alterations to triazene polymers, such as the insertion of ester groups into the backbone, enabling photo-cross-linking of the polymers without breaking up the polymers' backbones⁹. At the Paul Scherrer Institut other applications were explored, such as micro-optics formed by precise UV-ablation of the triazene, and laser-plasma thrusters for micro-satellites¹⁰. Also investigated was a form of laser transfer known as laser molecular implantation, where fluorescent molecules were contained within a triazene matrix and transferred via ablation¹¹. Imaging techniques were used to analyse the ablation process of triazene, and the first attempts were made to understand the nature of the ablation process¹². Further developments in these time-resolved methods enabled a greater understanding of the energy generated by this process^{13, 14}. An improved triazene polymer synthesis procedure¹⁵, developed at Empa, Dübendorf, has given more control to the structure of the triazene polymer, and therefore opened up the possibility of creating polymers with different structures¹⁶, and possibly different physical and chemical properties, such as solubility or reactivity.

Laser-Induced Forward Transfer

LIFT is a technique with some clear advantages. It has many of the benefits of the traditional press-printing, but uses a laser to act as the press. The laser irradiates the sample from behind as if pushing the material towards the receiving substrate. Only the material that is irradiated will be transferred, which means that the laser defines the lateral shape of the deposition. Precisely defined deposition can be obtained by controlling the path of the laser beam using a beam steerer. The important factor here is the transfer material; with the traditional printing press the transfer material was an ink which was robust and transferred quite simply. In today's age, with hand-held high-resolution display devices more factors need to be considered because we expect to transfer more things than a single ink, and in a more reliable way. So in order to be transferred efficiently it is desirable that the transfer material can absorb the wavelength of light used fairly well, and that it can also transfer this photon energy into a mechanical energy fairly efficiently. For more detailed and precise printing – at finer resolutions – this may be hard to achieve. The addition of an intermediate, dynamic release layer (DRL) to carry out the energy transfer is beneficial in terms of both the quality of the ablation, and also the reduction in degradation to the transfer material by light-absorption, even managing to maintain the film morphology through the transfer. This is the role which triazene polymer plays in laser-induced forward transfer, as a DRL.

LIFT Developments

The earliest report of LIFT, as it has been outlined above, comes from the simple "direct writing" deposition of aluminium layers using a 193 nm ArF excimer laser¹⁷. The addition of an intermediate layer was quickly adapted, originally as a thermally-activated DRL¹⁸, and then as a matrix in a process known as matrix-assisted pulsed laser evaporation direct-write (MAPLE-DW)¹⁹. The use of triazene in the molecular implantation process¹¹ was eventually followed by the use of triazene as a DRL in the transfer of polymethyl methacrylate²⁰. This

quickly led to the use of the triazene polymer as a DRL in the printing of other sensitive materials, such as living mammalian neuroblast cells in a polymer matrix²¹, quantum dots²², thin ceramic films²³, metals²⁴, polymers for sensors (in press) and organic light-emitting diodes (OLEDs)²⁵.

EXPERIMENTAL TECHNIQUES

The experimental section can be roughly divided into two large sections. The first is the study of the standard ablation (known as *frontside ablation*) properties of triazene polymers, the second is the study of the laser ablation transfer (known as *backside ablation*) mechanism. The lasers used are pulsed UV lasers. This gives the required fluence (energy per unit irradiated area) for ablation with nanosecond or picosecond pulse lengths. Whilst photo-degradation happens at all pulse lengths, the photon absorption per unit time is vital for successful ablation. Early studies of triazene degradation using Xenon lamps show photo-degradation without ablation²⁶. The optimal wavelengths for the aryl-triazene chromophore absorption are around 290-340 nm, so the preferred laser is the 308 nm XeCl excimer laser. The 3rd harmonic of the commercially-available solid-state Nd:YAG laser (at 355 nm) is also a viable option with triazene polymer absorption that is 80 % of that at 308 nm.

Sample Preparation

The substrates used are both ordinary glass and high quality quartz for the backside ablation to prevent the substrate absorbing too much of the UV light. The substrates are cleaned thoroughly before having thin films of triazene polymers deposited upon them, using spin-coating from solution. Film thicknesses obtained using this method range from < 10 nm to around 450 nm. For transfer experiments the next layer is usually aluminium, evaporated at low pressure (< 5×10^{-6} mbar), about 80 nm thick. This simple architecture is normally used for testing. For the fabrication of OLEDs, a polymer layer is spin-coated on top of the aluminium cathode. This is the light-emitting polymer, which serves a dual purpose as a mechanically stabilising layer (helping the transfer). The film quality was checked using a profilometer and an optical microscope.

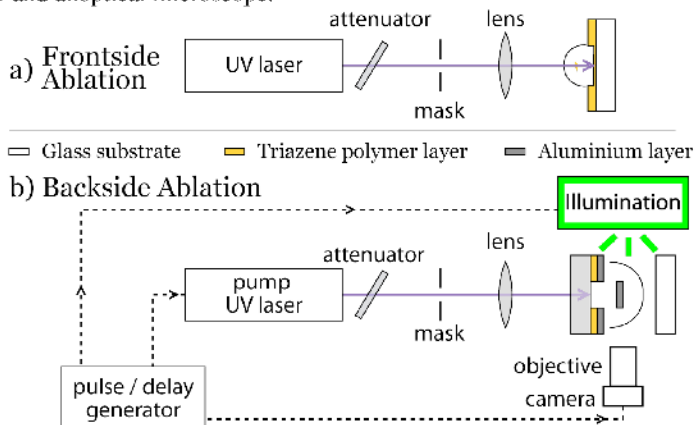


FIGURE 1. This shows the experimental setup for (a) frontside ablation and (b) backside ablation, with a receiver substrate, showing the shadowgraphy setup's illumination and camera.

Frontside Ablation

Frontside ablation experiments have been carried out using thin films of triazene polymer to help to understand the general ablation characteristics. A schematic of the experimental setup is shown in Fig. 1a. The most important information given by these experiments is the depth that is ablated for a given energy of laser pulse. The ablation depth per pulse (d) when measured as a function of fluence (Φ) can be used to give the effective absorption coefficient (α_{eff}) and the threshold fluence (Φ_{th}) when the curve is fitted to Eq. 1.

$$d = \frac{1}{\alpha_{\text{eff}}} \ln \frac{\Phi}{\Phi_{\text{th}}} \quad (1)$$

From this, further observations can be made as to the nature of the ablation, e.g. for different polymers¹⁶, or for different laser pulse lengths or wavelengths. The threshold fluence, obtained using Eq. 1, is another important property to consider. Whilst the theories for the mechanism of laser ablation are very complicated^{27,28} and by no means understood for triazene, looking at the frontside-ablation threshold for different film thicknesses of triazene has demonstrated that thermal diffusion loss can have an important effect upon the ablation of thin (< 100 nm) triazene films²⁹. These experiments were also carried out using transparent substrates with different thermal diffusivities, which further supported the argument that photothermal ablation mechanisms are significant.

Backside Ablation, Transfer and Shadowgraphy

Backside ablation is the basis for laser-induced forward transfer. Transfer is obtained by irradiating the triazene polymer from the "backside", through the quartz substrate, and propelling the transfer layer(s) to the donor substrate. A schematic of the setup is shown in Fig. 1b. In normal laboratory atmosphere, transfer is best achieved in contact at an optimised laser fluence²⁵. The experimental parameters for a successful transfer are not universal though, and strongly depend on the transfer layer's material properties²⁴. Transfer in contact has been achieved for a number of different materials (outlined above), each with their own optimal parameters (in particular, fluence and triazene thickness). Transfer with a gap is an aim, but has severe limitations at present³⁰. Successful transfer with even a small gap would be a huge improvement on current commercial OLED pixel-printing technologies such as laser-induced thermal imaging (LITI)³¹ which require very precise deposition environments (no gap and a vacuum). The investigations presented here outline research that has been conducted with the intention of improving the flexibility and reliability of the triazene-based LIFT process.

The principle investigative tool is shadowgraphy which generates side images (or "shadowgrams") of the transfer layer being ejected as a flyer. The set-up is outlined as a scheme in Fig. 1b. The ability to make these images time-resolved enables quantitative analysis of the shadowgraphy, particularly for the calculation of velocities and kinetic energies of the flyer and shockwave. Fig. 2 shows two different series of shadowgrams obtained for transfer across a gap at different air pressures. It is interesting to compare the velocities obtained from shadowgraphy to the frontside ablation depths per pulse measured as it is often assumed that the volume of triazene ablated is the same for frontside and backside and, because the pressure build up of the ablated materials is the main propelling force, the flyer and shockwave velocities should be directly proportional to the ablation depth per pulse³². This means that the velocities can be fitted to Eq. 1 as in Fig.3 and in Fig. 4.

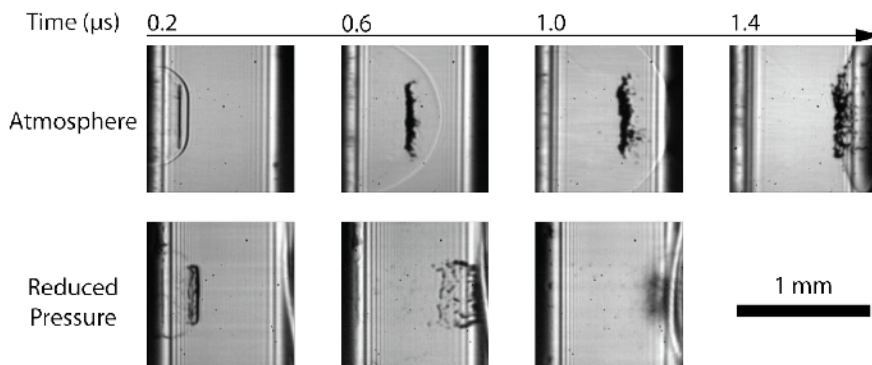


FIGURE 2. Two sets of shadowgrams of an aluminium flyer (deposited on top of 350 nm triazene polymer) being transferred across a ~ 1 mm gap. The top line shows the transfer in normal atmospheric air pressure, and the bottom line shows the transfer in a reduced air pressure ($\sim 10^{-2}$ mbar).

RESULTS SUMMARY

Low-Pressure Study

A natural development of a process like LIFT is to alter the medium through which the transfer takes place. Ordinarily, this process has been done simply in air and successful transfers have been obtained in normal laboratory conditions, but changes in the pressure or density of the medium, for example, are sure to alter the nature of the transfer. This opens up the possibility of optimizing the transfer conditions by changing the medium. The shockwave's energy has been extensively studied^{12,33}, and an interesting observation has been the effect of the shockwave, reflected back from the receiver substrate, on the destruction of the flyer when transferred across a gap³⁰. With this effect in mind, experiments have been made which aim to lessen the shockwave energy by reducing the air pressure.

Different fluences were tested, with different gaps for transfer (0.5 and 1 mm), for the LIFT process at reduced pressure (< 0.05 mbar). Changing the laser fluence has been observed to have no different effect to that already observed at atmospheric pressures³², except that the flyer speed was proportionally faster at lower pressures. Shadowgrams show the increased speed of the flyer for a given fluence, despite the fact that the threshold fluence at low pressures was not significantly altered compared with atmospheric pressures. This data is shown in the graph in Fig. 3. The speed increase is most probably due to a reduction in physical drag upon the flyer. The shockwave is probably still present, but much weakened due to the lower pressure which will both increase shockwave propagation, but also reduce the amount of energy transferred from the transfer material to the medium, in the same way in which sound wave propagation is reduced in a vacuum.

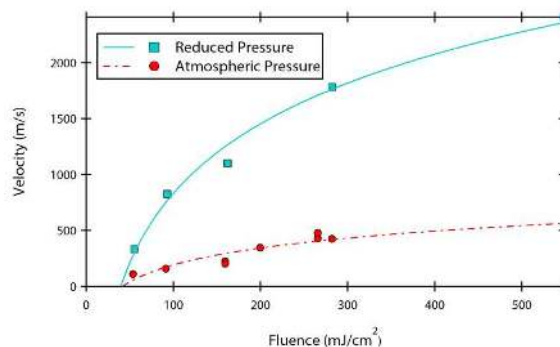


FIGURE 3. Flyer velocity as a function of fluence as calculated from shadowgrams (like those in Fig. 2). Reduced pressure flyer velocities (blue squares/blue solid line fit) and atmospheric flyer velocities (red circles/red dashed line fit) have been plotted and the fit lines were calculated according to Eq. 1.

Even though the shockwave is not present to destroy the flyer upon reflection, the flyer's increased speed increases the chance of a destructive inelastic collision with the receiver substrate. The 2 sets of shadowgrams in Fig. 2 show the difference in the transfer across a 1 mm gap for aluminium flyers at atmospheric and reduced pressures. For simple triazene polymer / 80 nm aluminium donor substrates there is no optimal fluence where a successful transfer may be obtained across a gap at reduced pressure. The addition of a stabilising light-emitting polymer layer (MEH-PPV) improves the transfer³⁰, but not enough to transfer a pixel with functionality. For any gap width, the transfer of pixels is better in air at atmospheric pressure than at reduced pressure below 0.05 mbar. The next step is to look at intermediate pressures and see how the flyer speeds and shockwave energies vary with pressure, and also to try out different gaseous densities with gases such as helium (lower density than air) and argon (higher density).

Pulse Length Effect

Whilst it is known that when the UV-irradiation is for too long a period, no ablation of triazene polymer will occur²⁶, the effect of shortening the pulse beyond the nanosecond regime had not been studied previously. This is particularly interesting following on from studies into the importance of thermal diffusion upon the laser ablation of triazene polymer²⁹. In the mathematical model of thermal diffusion the effect of the pulse length is considered. According to the model a shorter pulse length should decrease the heat loss via diffusion, and therefore increase the efficiency of energy transfer from light into gas-pressure build up of the ablation products. This investigation involved looking at a shorter pulse length laser: a mode-locked Nd:YAG laser with a pulse length of ~70 ns. This laser has a wavelength of 355 nm, slightly different to the standard 308 nm XeCl excimer laser. For this reason another Nd:YAG, with a Q-switched pulse length of 5 ns was also used to see which differences between the results could be attributed to the different pulse lengths, and which should be attributed to the wavelength difference.

Our frontside ablation threshold fluence results, shown in Fig. 4c, indicate that picosecond pulse lengths do appear to lower the large energy loss for frontside ablation of triazene thin films when compared with the observations of frontside ablation with the more typical nanosecond pulse lengths. Fig. 4d shows the ablation depth per pulse as a function of fluence and shows that the effective absorption coefficient^{34, 35} is much lower for the ps pulses. Despite the support for the thermal model by the results in Fig. 4c, the differences between

the different lasers shown in Fig. 4c should not be solely attributed to a reduction in heat loss, especially when considering the difference between the two ns pulse lasers. Although the thermal decomposition products and the photochemical reaction products are nearly identical³⁴, there is still believed to be a significant degree of energy from photochemical absorption which is not transferred into thermal energy before the triazene decomposition. In addition, the lower effective absorption coefficient for the ps pulse in Fig. 4d must play a role in keeping the threshold fluence low for thin films, although this is not an exclusive mechanism to the thermal model above (the effective absorption coefficient takes into account all ablation mechanisms).

Given the assumption that the volume of triazene removed by frontside ablation can be taken as the volume ablated by backside ablation and therefore should be proportional to the flyer velocity observed by shadowgraphy³², an apparent contradiction is seen when comparing the different pulse lengths used at the same fluence. Both Fig. 4a and Fig. 4b show that the speed of the shockwaves and flyers are greatly increased for ps laser pulses. However, frontside triazene polymer ablation depth per pulse, Fig. 4d, shows that a smaller volume of polymer is ablated for ps pulses. When faster velocities result from smaller volumes of triazene ablation, then the conversion of pressure build-up to kinetic energy can be said to be much more efficient. The triazene polymer ablation process is clearly different at shorter pulse lengths.

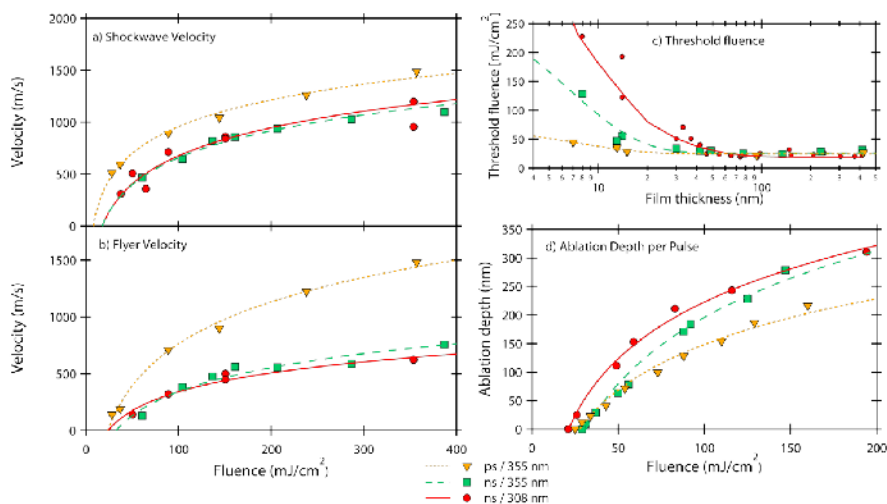


FIGURE 4. Comparisons between the standard 30 ns pulse length 308 nm XeCl excimer laser (red circles/red solid line fit), the 5 ns pulse length 355 nm Nd:YAG laser (green squares/green large dashes line fit) and the 70 ps 355 nm Nd:YAG laser (yellow triangles/yellow small dashes line fit). (a) and (b) show the shockwave and flyer velocities respectively, calculated from shadowgram analysis. The other two graphs are for frontside ablation: (c) shows the threshold fluence as a function of film thickness, and (d) shows the ablation rate per pulse for a thick film (> 400 nm). The fit lines have been calculated according to Eq. 1.

Transfer by Triazene Polymer-Based LIFT

Successful transfer of many different types of sensitive materials has been achieved using triazene-based LIFT. As the optimal transfer parameters vary greatly with the transfer material's physical properties, each transfer material must be refined. We are concentrating on the transfer of thin films of functional light-emitting polymers. Due to solvent compatibility issues and the importance of good interfaces for successful charge transfer and radiative

electron-hole recombination, we always include the non-aluminium cathode in the stack of transfer films. Being more brittle than the polymers (although more ductile than most other metals), aluminium is a good material to use as a simple test-transfer layer when looking at the LIFT mechanism. This is because a successful transfer is harder to achieve with a single 80 nm layer of Al than with polymer layers on top of the aluminium.

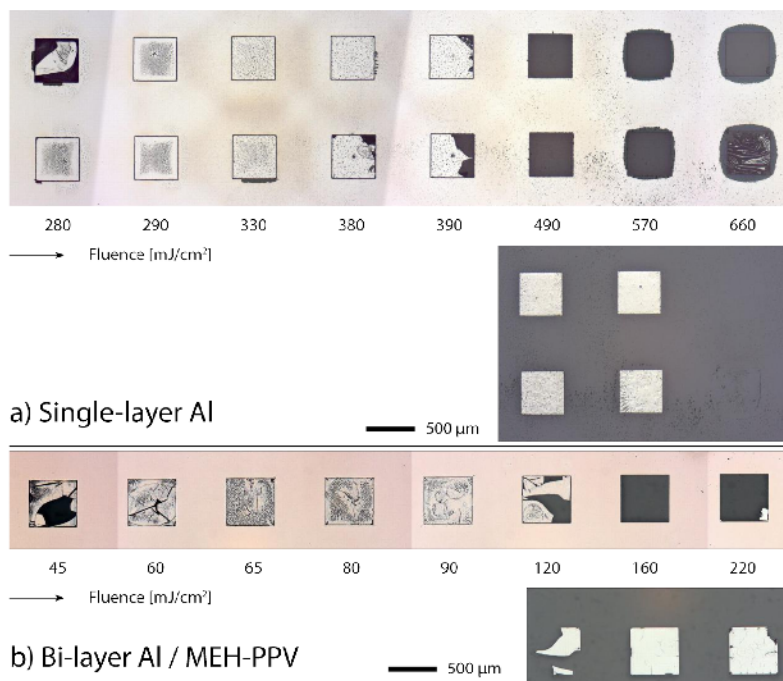


FIGURE 5. These microscopy images show the donor substrate (top) and the receiver glass substrate (bottom) for two different types of transfer materials; (a) is a single 80 nm layer of aluminium evaporated onto a 350 nm triazene polymer film²⁴, and (b) is a bi-layer comprising of 80 nm of MEH-PPV spin-coated on top of 80 nm aluminium, which was evaporated onto a 100 nm triazene polymer film.

Aluminium transfer has been achieved using the 308 nm XeCl laser with a glass receiver substrate in contact with the donor substrate, as shown in Fig. 5a. The triazene polymer thickness was 350 nm and the fluences required were in the range of 450-600 mJ/cm². With layers of triazene polymer thinner than 350 nm, it was seen that the transfer could not be obtained. The fluence required is fairly high, especially when compared with fluence required when a polymer layer is added on top of the aluminium. Fig. 5b shows the transfer of Al / MEH-PPV bi-layers with a 100 nm triazene polymer layer. The fluence required for the best transfer was 160 mJ/cm². For the transfer of pure aluminium, all the triazene should be ablated at a fluence of 450 mJ/cm², which means that some of the light energy is actually transferred into the aluminium. Some black particles can be seen around the transferred pixel, and this suggests that the aluminium may be directly ablated itself²⁴. However, the idea that the mechanical resistance to ejection by the aluminium is alone responsible for the required large fluence is not reasonable when compared with the low fluences required for the bi-layer. A more likely explanation is that an optimal fluence is required where the aluminium is forced to stick to the glass substrate. This is naturally high, but to prevent direct ablation of the aluminium, a thick layer of triazene polymer is necessary. Although the bulk of the

experiments have been carried out with a XeCl excimer laser, the 3rd harmonic of the Nd:YAG laser (355 nm) has also been used to successfully transfer aluminium / MEH-PPV bi-layers.

Transfer in contact is a good achievement, but it would become a more robust process if a reliable transfer with a small gap were achieved. This would give LIFT an advantage over competitors which require contact for transfer such as LITI. Whilst successful transfer using LIFT across a gap in reduced air pressure was found to be almost impossible, LIFT in atmospheric air pressure across a 1 mm gap has transferred the pixels in single fragments, although folded at the edges³⁰.

The proof of a successful transfer is when electroluminescent functionality of the MEH-PPV is maintained. This has been achieved in contact²⁵, and a functioning pixel is shown in Fig. 6a, along with close-ups of a well-transferred pixel in Fig. 6b and Fig. 6c. These microscopy images show how the MEH-PPV spreads out beyond the aluminium, actually helping prevent any short-circuiting by the deposition.

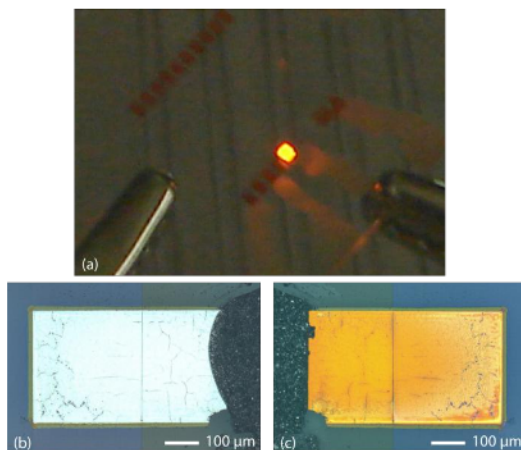


FIGURE 6. (a) shows an illuminated MEH-PPV OLED, fabricated by LIFT. The bottom pictures are microscopy images of the transferred MEH-PPV / Al stack, with (b) being viewed from above (the aluminium is on top, and the MEH-PPV squeezing out at the sides), and (c) being viewed through the glass substrate, through the ITO (left of image) and onto the MEH-PPV polymer (orange).²⁵

Summary

The history of LIFT using a triazene polymer DRL has been reviewed and the speed of development shown. Whilst LIFT has been around for 30 years, and triazene ablation has been studied for 20, the use of triazene polymers as an intermediate layer in LIFT has been only started to be fully exploited within the last 5 years. Recent results focus around the mechanism and attempts to better understand the fundamental principles behind triazene ablation and LIFT. The experiments at reduced air pressure have helped to look at the effect that removing air drag and a strong shockwave have on flyer formation and speed. The shadowgraphy results tell us that atmospheric pressure helps drag the flyer considerably, in a positive way for enabling successful transfer across a gap, and that the lack of a shockwave can reduce flyer destruction. Different pulse-length investigations have given us all sorts of interesting results – previous photothermal models have been supported, although not comprehensively. Picosecond pulse lengths have been observed to produce very fast flyers

from a smaller volume of ablated triazene polymer. This is hard to explain, and doesn't help resolve the photothermal vs photochemical (or photophysical) polymer ablation debate, but may even points to additional complexities not taken into account before. Finally, transfer of many different materials has been achieved using triazene-based LIFT. The method has proven its versatility with a wide range of sensitive (and less-sensitive) materials. Additionally, the possibility of transfer with a small gap has been investigated, and although functional OLED pixels have not yet been transferred the outlook looks bright.

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