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A time-resolved shadowgraphic study of laser transfer of silver nanoparticle ink

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ABSTRACT

The dynamics of liquid phase laser induced forward transfer (LIFT) of silver nanoparticle (NP) ink (particle size 30–50 nm) was investigated by time-resolved shadowgraphic imaging. LIFT was carried out by a KrF excimer laser (248 nm, 35 ns) using two donor substrate configurations: (a) a wet silver NP ink layer spread on a quartz substrate and (b) a wet silver NP ink layer spread on a quartz substrate covered by a 40 nm thick titanium layer. This comparative study revealed a completely different ejection mechanism for the two different donor configurations. The use of the titanium dynamic release layer (DRL) resulted in a highly directional and low velocity ejection of the material for a wide range of laser fluences. On the other hand, LIFT of silver NP ink without using the titanium DRL provoked supersonic velocity ejection and shock wave generation for laser fluences even slightly above the ejection threshold. The velocity of the ejected material (13–240 m/s) in the case of titanium DRL assisted LIFT was significantly lower than the one observed without using DRL (106–830 m/s). The use of the titanium DRL layer expanded, significantly, the processing window for directional and low velocity ejection of the silver NP ink.

1. Introduction

The excellent properties of the commercial silver nanoparticle (NP) inks such as high electrical conductivity, stability and low sintering temperature make these materials ideal for applications like organic photovoltaic, plastic electronics and thin film transistors. The fabrication of those microelectronic devices requires high precision printing of silver NP ink based miniaturized interconnects.

Ink-jet printing of metallic NP inks provides a direct and maskless patterning method, which has been widely used for the fabrication of conductive interconnects [1,2]. Despite its broad appeal, the ink-jet printing technology presents several limitations regarding to the spatial resolution of the deposited patterns (25 μm for printing continuous lines [3]) and the rheological properties of the complex NP ink solutions. In particular, the viscosity and the solid content (NPs concentration) of the metallic NP ink suspensions should be carefully selected to avoid clogging of the nozzles [1].

Recently, laser induced forward transfer (LIFT) [4–6] of silver NP inks is receiving growing interest as it offers an alternative non-lithographic technique for printing uniform and well-defined conductive patterns [7–13]. Liquid phase printing of low viscosity silver NP inks has been performed for patterning conductive lines and dots using both conventional [8–13] and dynamic release layer (DRL) assisted LIFT [7]. Three-dimensional printing of complex patterns has also been demonstrated by employing LIFT of high viscosity silver NP pastes [10,12].

A critical issue for the LIFT of liquid solutions is the definition of the optimum laser processing parameters (i.e. pulse duration, wavelength, laser fluence, and beam size) and donor substrate characteristics (i.e. use of suitable DRL, liquid film rheological properties and thickness) to print uniform and reproducible droplets. To this direction, time-resolved imaging of the LIFT process provides an advanced method to investigate the dynamics of the material ejection and optimize printing capabilities. Most of the studies dealing with liquid phase LIFT dynamics have been performed by using model biological solutions and several metallic and polymeric DRL layers [14–18]. It is a common observation for these studies that under the optimum laser fluence the material ejection takes place through the formation of a long and stable jet. Recently, the jetting behavior has been simulated using hydrodynamic modeling [19,20].

Despite the extended work on the LIFT dynamics of glycerol-based model solutions, there are only few studies dealing with LIFT dynamics of composite solutions [13,21,22]. Our study was focused to the investigation of LIFT dynamics of a commercially available complex Ag NP ink solution, which is widely used for digital printing of interconnects. The dynamics of both conventional and titanium layer assisted liquid phase LIFT of silver NP ink were investigated by using time-resolved shadowgraphic imaging. This comparative study was carried out in order to investigate the effect of a titanium layer on the ejection mechanism. We have found a

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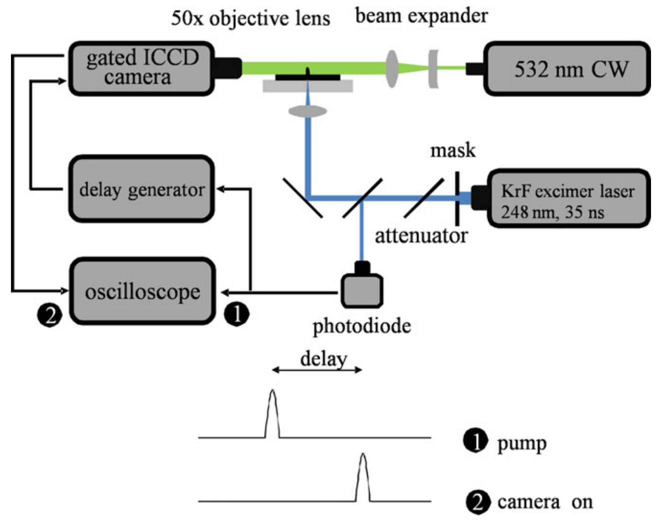

Fig. 1. A sketch of the shadowgraphic time-resolved imaging setup. A pump KrF eximer laser (248 nm, 35 ns) initiates the ejection of silver NP ink. The images are captured with a gated ICCD camera, which is synchronized to the pump laser through a photodiode and a pulse delay generator. Both photodiode and ICCD signals were sent to an oscilloscope for accurate measuring of the delay time.

completely different ejection mechanism for the two different LIFT configurations. In the case of the titanium layer assisted LIFT of the silver NP ink the material was ejected with a directional way and significantly low velocities. On the contrary, LIFT of silver NP ink without using DRL resulted in directional ejection of the material only for a narrow laser fluence range. A slight increase of the laser fluence over the ejection threshold provoked supersonic material ejection and shock wave formation.

2. Materials and methods

Two different donor substrates were used in order to investigate the effect of the titanium absorbing layer on the dynamics of the silver NP ink ejection. The first type of donor substrates was 1 mm thick quartz plates (25 mm in diameter) purchased from UQG Optics. The second type donor substrates was prepared by using the same type of quartz plates coated with a 40 nm titanium laser absorbing layer. The titanium layer was deposited by electron beam evaporation (typical thickness variation was 2 nm). A thin liquid film of silver NP ink (U5603, SunChemicals, 20 wt.% silver content, solvent: mixture of ethylene glycol, glycerol and ethanol, viscocity: 12 mPa s, NP size: 30–50 nm) was applied to both types of donor substrates by using spin coating (2900 rpm, 30 s). Spin coating ensured reproducibility and high uniformity of the film thickness ($\sim$5.5 μm) across the donor target surface. No additional treatment was applied at the donors, which were used for a maximum period of 30 min after their preparation to avoid silver ink film drying. Measurement of the donor mass over the time revealed that solvents' mass loss rate was about 4.5 μg/min. This caused an insignificant reduction (about 4.5%) of the mass of the spin coated silver NP ink layer within the 30 min period. Therefore, we have considered the silver NP ink film as wet and uniform within the time period that each donor was used.

The donor substrates were placed in a "face up" liquid phase LIFT configuration without receiving substrate (Fig. 1). Then, the silver NP ink ejection was initiated by using single pulses of a KrF excimer laser source (248 nm, 35 ns, 1–100 Hz, 0.5 J) and a mask projection optical system. The projected laser beam spot on the donor substrate was rectangular with an edge width of 220 μm. The dynamics of the LIFT process was studied using the experimental configuration which is depicted in Fig. 1. An expanded laser beam of a cw Nd:YAG laser was used to illuminate homogeneously the ejected material. Images were captured by a gated intensified charge-coupled device ICCD (ICCD camera, Princeton Instruments) equipped with a 50X long working distance objective lens. The integration time for each image frame was 20 ns (gate time). The camera synchronization was initiated by the pump KrF excimer laser by using a photodiode and a pulse delay generator. Both photodiode and ICCD signals were sent to an oscilloscope for accurate measuring of the delay time, which was ranged from 145 ns to few microseconds (Fig. 1). Three to five images were taken for each delay time in order to ensure reproducibility. Finally, the images were analyzed using the ImageJ [23] software to extract the front distance of the ejected material as a function of the time.

3. Results and discussion

3.1. Dynamics of titanium layer assisted LIFT of silver NP ink

In the first part of this work we investigated the ejection of the silver NP ink using titanium layer assisted LIFT. Time-resolved shadowgraphic images of the ejected silver NP ink under various laser fluences are depicted in Fig. 2. Pictures were obtained at different time delays (0.145–18.5 μs) with respect to the pump laser pulse.

For a laser fluence range from 100 mJ/cm^2 (ejection threshold) up to 130 mJ/cm^2, which is below the titanium layer ablation threshold (experimentally defined around 200 mJ/cm^2), a directional ejection of the silver NP ink was initiated due to the absorption of the laser energy by the titanium layer. In accordance with the absorption coefficient ($a \sim 6.13 \times 10^5$ cm^{-1}) of the Ti layer at 248 nm [24], the incident laser radiation will not exceed a penetration depth of about 16 nm ($1/a$). As a result of the localized temperature rise at the titanium DRL, a vapor pocket was formed at the titanium–liquid film interface due to the vaporization of the silver NP ink solvents. Since the laser pulse duration was much shorter than the materials dynamic response we consider the expansion of the vapour pocket as the main impulse force for the materials ejection. The observed dynamics can be related to the jet formation in liquids due to the expansion of a bubble near a free surface [25], which has been reported in several experimental [14,15] and simulation modeling works [19] dealing with DRL assisted LIFT of liquid model solutions. However, the jetting behavior observed in our experiments is less directional and presents several discontinuities. This is attributed to the nature of the silver NP ink material that consists of two separated phases; the solid silver NP component embedded in the organic solvents matrix as the liquid phase. The slight right bend of the flyer that is observed for low laser fluence in Fig. 2 (also appeared in Fig. 4) is probably due to laser bean intensity variation within the laser spot. Despite the relative uniform beam profile achieved by the mask projection system, the large laser spot size (220 μm) compared to the Ag NP solution film thickness (5.5 μm) enhanced the effect of the laser intensity variation on the flyer ejection.

For higher energy fluences, above the 130 mJ/cm^2, a different dynamic behavior is observed. The shape of the initial flyer at the silver NP ink layer–air interface clearly reproduced the size and the shape of the laser spot indicating a transfer behavior that is common for solid phase LIFT dynamics [26,27]. The expansion of the initial protrusions of the silver NP ink material was completed in a microseconds time scale. It is also noticed that no shock wave is observed even for a laser fluence of 330 mJ/cm^2 that resulted in plasma formation. The absence of shock wave is in agreement with the calculations for the ejection velocity of the silver NP ink material (240 m/s) that was well below the supersonic velocity threshold (343 m/s).

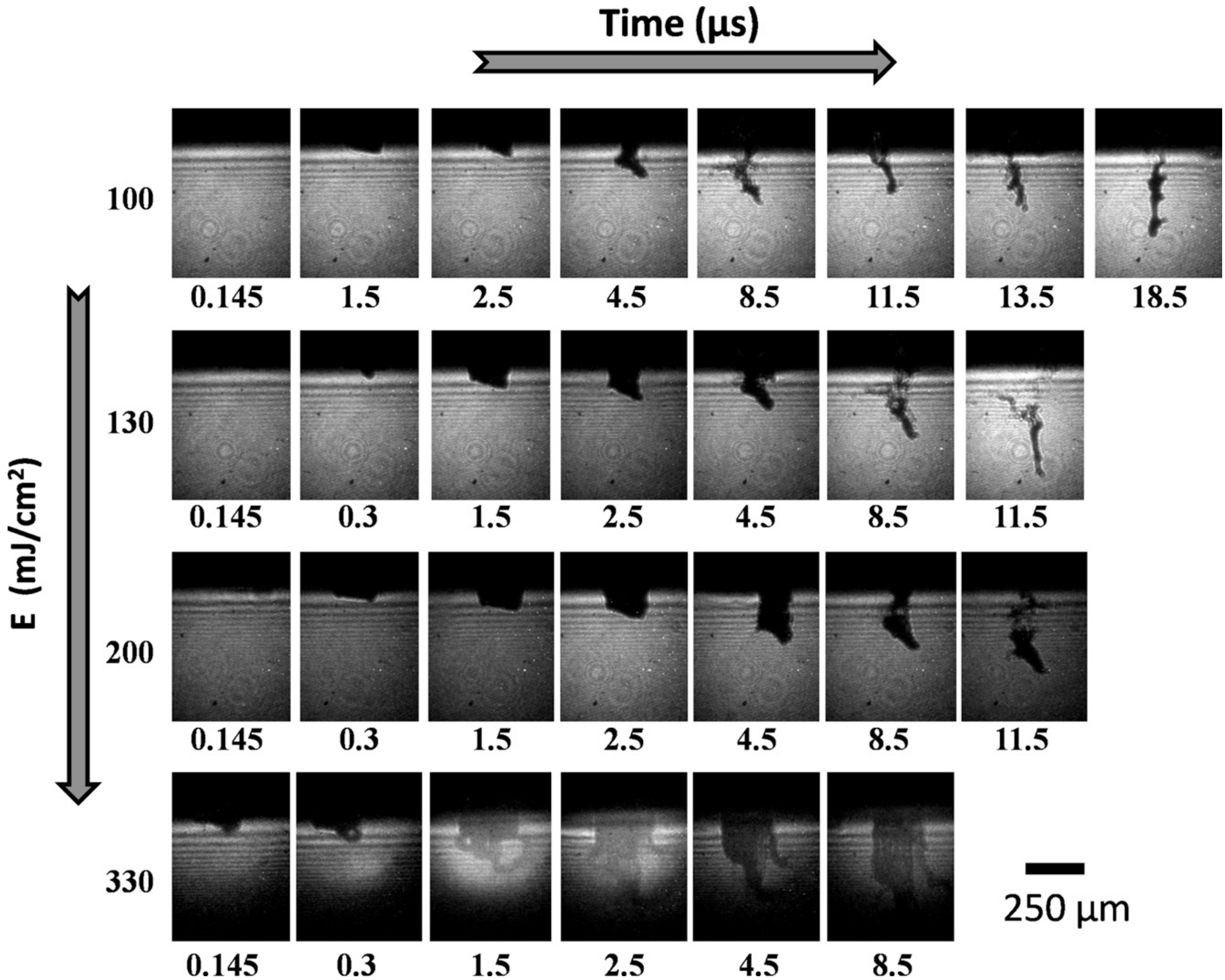

Fig. 2. Shadowgraphic time-resolved images of the silver NP ejection using titanium DRL assisted LIFT for various laser fluences. Pictures were taken at different time delays with respect to the pump laser pulse.

The front distance of the ejected silver NP ink material as a function of the delay time with respect to the pump laser pulse is depicted in Fig. 3. As it can be seen the dependence is approximately linear for 200 mJ/cm^2 and 330 mJ/cm^2. Therefore, the ejection velocity was calculated by a linear fit of the data. For laser flunces of 100 mJ/cm^2 and 130 mJ/cm^2 linear fitting was applied for delay time $\geq$2.5 μs. The existence of two different velocity regimes for this laser fluence range can probably be attributed to viscous forces due to the jet like ejection of the silver NP ink material. Similar behavior has been observed in LIFT works dealing with jetting behavior of model solutions [14,20]. The calculated velocities ranged from 13 m/s to 240 m/s for laser fluences ranging from 100 mJ/cm^2 (ejection threshold) to 330 mJ/cm^2 (plasma generation threshold), respectively. The use of the titanium DRL resulted in a broad laser fluence processing window (100–200 mJ/cm^2) for low velocity ejection of the silver NP ink. As a result, LIFT printing of silver NP ink dots and lines can be achieved with high quality and reproducibility [7].

The significant low ejection velocities of the silver NP ink for the laser fluences below the plasma generation threshold (330 mJ/cm^2) are characteristic of the “indirect” (i.e. DRL assisted) ejection mechanism, which involves titanium layer heating and vapor pocket formation and expansion. In particular, the use of the 40 nm thick titanium layer prevents the direct exposure of the overlying silver NP ink layer to the laser irradiation. This is due to the

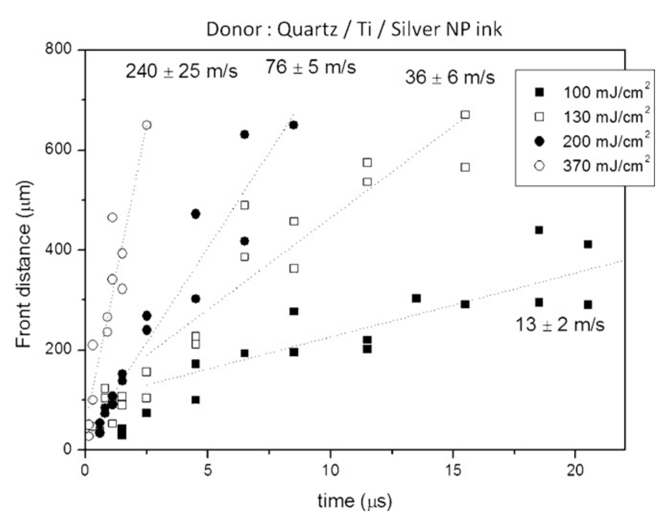

Fig. 3. Titanium layer assisted LIFT of silver NP ink. Plot of the front material position as a function of the time delay with respect to the pump laser pulse. The standard error of the slope was used to determine the corresponding standard error for the velocity.

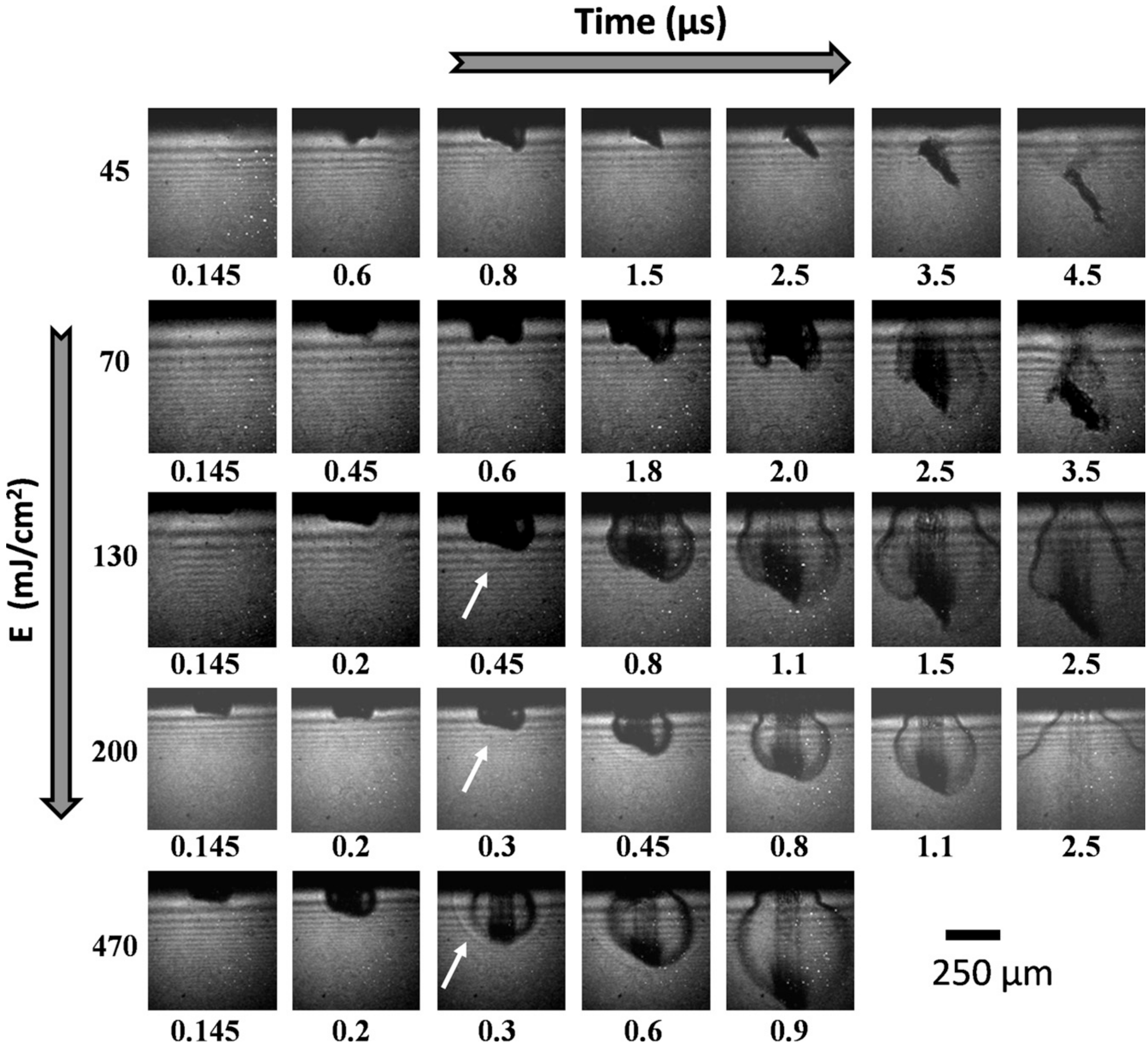

Fig. 4. Shadowgraphic time-resolved images of laser transfer (without titanium DRL) of silver NP ink for various laser fluences. Pictures were taken at different time delays with respect to the pump laser pulse. The laser fluence of 130 mJ/cm^2 is the threshold for a shock wave propagation, which is indicated by white arrows.

significant short penetration depth of the laser irradiation (16 nm) in the titanium layer compared to its thickness (40 nm). Therefore, heat diffusion to the silver NP ink layer is considered as the dominant mechanism for the bubble formation at the titanium layer/silver NP ink layer interface.

3.2. Dynamics of LIFT of silver NP ink without using DRL

In the second part of this work we investigated the ejection of the silver NP ink without using the titanium DRL layer. Time-resolved shadowgraphic images of the ejection of the silver NP ink under various laser fluences are depicted in Fig. 4. Pictures were obtained at different time delays (0.145–4.5 μs) with respect to the pump laser pulse.

For laser fluences ranging from 45 mJ/cm^2 (ejection threshold) up to 70 mJ/cm^2 a directional ejection of the silver NP ink was initiated due to the absorption of the laser energy by the silver NP liquid film. It can be assumed from the experimentally determined value of the absorption coefficient ($a \sim 5.88 \times 10^3\ cm^{-1}$) at 248 nm that the incident laser pulse energy is absorbed within the first ~1.7 μm of the silver NP ink layer (1/a). The laser energy was mainly absorbed by the silver NP content of the silver NP ink solution. The laser-induced heating of the silver NP causes the vaporization of the silver NP ink solvent. As a result, a vapor pocket was formed at the quartz–liquid film interface. The expansion of the vapor pocket led to a directional ejection of the silver ink material similar to the one observed for titanium assisted LIFT at low laser fluences. However, the observed dynamics is different from the jet and bubble behavior, which has been reported in the literature for LIFT of silver NP ink [21] and barium titanate nanopowder ink [22]. This is attributed to the relative high ratio of the spot diameter to the silver NP ink layer thickness (~40). More specifically, the laser irradiation of a relative large donor area results in a rapid rise of the height of the vapor pocket, which expands prior to the formation of a well defined jet even for the ejection threshold laser fluence. The above observation has been also noticed in the work of Mézel et al. [19], in which the influence of the bubble diameter and film thickness on the jetting behavior were investigated.

For higher energy fluences the experiments showed a completely different behavior of the ejection dynamics. Shock wave

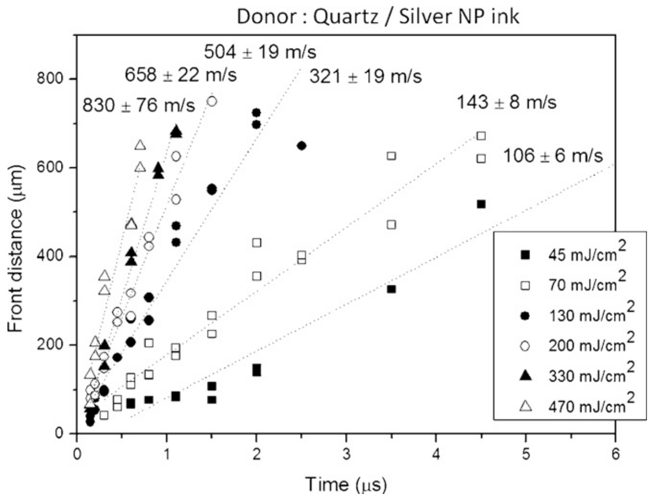

Fig. 5. LIFT of silver NP ink without using DRL. Plot of the front material position as a function of the time delay with respect to the pump laser pulse. The standard error of the slope was used to determine the corresponding standard error for the velocity.

propagation was observed as it is indicated by white arrows in Fig. 4. The ejection of the material took place with a high diversion hemispherical cloud due to the violent expansion of the vapor pocket. At the central part of the hemispherical cloud, concentrated silver NP ink material travels parallel to the laser pulse direction. The edges of the cloud consist of less concentrated material that expands with a hemispherical shape. We consider the following mechanism to explain this complex ejection behavior of the silver NP ink material. The violent expansion of the vapor pocket propels the overlying silver NP ink layer parallel to the laser beam direction with a supersonic velocity (Fig. 5). The initially ejected silver NP ink material forms the central concentrated part of the observed hemispherical cloud. At the same time, the supersonic displacement of the ejected material produced a strong shock wave that propagated with a hemispherical shape. The propagation of the shock wave initiated a hemispherical ejection of the surrounding silver NP ink layer. That formed the less concentrated edges of the observed hemispherical cloud.

Fig. 5 depicts a plot of the front distance of the ejected silver NP ink material as a function of the delay time with respect to the pump laser pulse. The ejection velocities were derived from a linear fit of the data. It is mentioned that no obvious deceleration effect is observed due to friction in air. This is probably due to the more aerodynamic ejection of the silver ink material (i.e. cloud of discontinuous separate NPs) compared to the ejection of solid rectangular flyers [28], where a characteristic "bend" at the plot is observed due to friction in air. The ejection velocities were ranging from 106 m/s to 830 m/s for a laser fluence range from 45 mJ/cm^2 (ejection threshold) to 470 mJ/cm^2, respectively. The threshold fluence for the supersonic ejection regime of the silver NP ink material was at 130 mJ/cm^2, where hemispherical ejection was observed.

It is significant to mention that the material ejection velocities in the case of LIFT without using DRL were calculated to be one order of magnitude higher than the DRL assisted LIFT for a similar laser energy fluence range. The direct exposure of a significant part of the silver NP layer to laser irradiation is considered to be crucial. When LIFT was performed without using DRL the laser irradiation penetrated within the fist ~1.7 μm of the silver NP ink layer. The laser induced heating of the NPs as well as the strong near field enhancement around the vicinity of the NPs (nanolens effect) contributed to the generation of several nano-bubbles within the laser penetration depth. The dynamics of the nano-bubbles generation around metallic NPs has been reported to be extremely fast (nanoseconds time scale) [29,30]. The synergetic action of the bubbles, which were generated immediately after the laser irradiation, is considered as the main impulse force for the observed "explosion-like" dynamics including supersonic material ejection and shock wave generation. The strong scattering effects of the incoming laser beam, caused by the silver NPs, may have also contributed to "explosion-like" dynamics by producing an optical "dispersing" effect. The importance of the scattering effects will be further investigated.

4. Conclusion

The dynamics of both conventional and titanium DRL assisted LIFT of silver NP ink was investigated by shadowgraphic time-resolved imaging. A completely different ejection behavior was observed for the two different LIFT configurations. The indirect mechanism of the materials ejection in the case of the titanium DRL assisted LIFT resulted in a high directional ejection of the silver NP ink for a wide range of laser fluences (100–200 mJ/cm^2). The ejection velocity was measured to be significant low (13–74 m/s), which is ideal for high uniformity printing of silver NP lines and dots. On the contrary, LIFT of silver NPs ink without using the titanium DRL revealed directional ejection of the material for a narrow fluence range (45–70 mJ/cm^2). LIFT without DRL at higher laser fluences resulted in a high diversion hemispherical ejection of the silver NP material due to shock wave propagation. Our results indicate titanium DRL assisted LIFT of silver NP ink as the optimum selection for printing conductive lines and dots from liquid phase. The use of the titanium DRL has a "deceleration" effect to the ejection dynamics due to the indirect ejection mechanism that involves DRL heating and vapor pocket formation and expansion. As a result, the effective laser fluence range for directional and low velocity transfer of the silver NP ink is significantly expanded.

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