Laser Induced Forward Transfer (LIFT) of Materials using 40 ps Pulses- Experimental and Quantitative Modelisation Study

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In this paper we report the results of experiments on LIFT performed using single pulses from a mode locked (40ps pulses), frequency doubled Nd:YAG laser (energy flux $\approx 13 J/cm^2$ at $\lambda = 532$ nm). We studied the controlled transfer of thin films of different metallic materials to a receiving substrate. Optical and scanning electron microscopy (SEM) as well as Energy-dispersive X-ray spectroscopy (EDAX) analysis were performed, showing evidence of the production of micrometric scale patterns. One of our objectives is to investigate and develop possible applications of LIFT in jewellery industry. In order to understand the experimental results we developed a simple analytical model based on the Rankine-Hugoniot relations. Comparison between experimental and simulated results is presented.

1. Introduction

Laser induced forward transfer (LIFT) was first demonstrated by Bohandy et al. in 1986 [1]. LIFT has the following three phases: (a) a laser pulse impinges on a donor film vaporizing a portion of it. (b) plasma expansion ejects a piece of donor film at high velocity (c) the ejected film, in the form of micro-sized clusters, bonds with the acceptor substrate. LIFT has been implemented by several research groups due to its efficiency in fabricating microstructures. Applications in micro patterning are indeed needed in integrated circuits industry, optical data storage, laser printing for organic and Polymer for optoelectronics, laser printing of biomaterials. Furthermore, LIFT can be an important technique for thin film deposition and micromachining.

Even if laser ablation starts at laser fluxes as low as several tens of mJ/cm^2 [2,3], laser pulses at higher fluxes (up to several J/cm^2) are needed for laser transfer. Selected works of other researchers on LIFT deposition are outlined in references [4-14].

LIFT can be compared to PLD (pulsed laser deposition).

In PLD, a laser beam of high intensity is focused on a target and an expanding coating plume is created and subsequently condenses on a substrate. Instead, LIFT is a micro printing technique, which adds mass instead of removing mass, it can be considered as inverse technique of micromachining. In particular, in the present paper, we show some preliminary results concerning the controlled transfer of thin films of different metallic materials to a receiving substrate using single pulses from a mode locked ($\tau \approx 40$ ps pulse duration), frequency doubled Nd:YAG laser. Receiving substrates were placed at a close proximity to the thin film. Optical and scanning electron microscopy (SEM) as well as EDAX analysis was performed, showing evidence of the production of micrometric scale patterns.

Our final goal was to study the transfer of precious metal to metal / crystals for possible applications in jewellery industry. LIFT is a promising technique in jewellery industry because design and manufacture requires state-of-the-art-tools. LIFT writing on rings or flat components is expected to give the level of customization

and personalization required by the customer using compact and efficient systems. Also, while almost all previous studies focused on the use of relatively long pulses, here we made use of short laser pulses. In the field of laser ablation, it is well known that short pulse (fs and ps) may have several advantages over longer ones [2, 3]. Therefore, in our study, we were also motivated by the idea of testing LIFT performances in the short pulse regime.

Since this is a preliminary study, we were not interested in reproducing definite patterns onto the substrate but we just studied the transfer of a foil portion roughly corresponding to the laser beam size on the target foil and studied its adhesion.

We also developed a simple physical model of LIFT at high laser intensity. The model is based on the idea that laser irradiation creates a shock wave travelling in the donor material. After shock breakout, the foil is accelerated and the donor material impinges and eventually sticks on the substrate.

The model is using Rankine-Hugoniot (RH) relations for shock dynamics and a specific equation of state model (we used the SESAME tables [15]). The results of the model were qualitatively compared to our experimental data showing significant comparable trends.

2. Experimental set-up

We used single laser pulses generated from a mode locked (pulse duration τ=40 ps), frequency doubled (532 nm) Nd:YAG laser. The system is described in ref. [16].
The maximum applied laser energy on foil was ≤ 25 mJ. The target (thin foil) was positioned far from the best focus to get a quite large beam size (diameter ≤ 0.5 mm) implying a laser flux on target ≈ 13 J/cm^2.

The receiving substrates were metals, semiconductors or crystals. They were placed parallel, at a close proximity to the thin film (distance of the order of 100 μm, in few cases at 1mm). Samples were mounted on a mechanically-controlled, two-axis translation stage, so to expose fresh areas of the film to each laser pulse.
All experiments were conducted in air at room temperatures. Although, irradiation under vacuum would prevent any possible role of the ambient gas, in reality already in 1988, Bohandy et al. [4] compared results obtained in vacuum and in air and noticed how LIFT could be done equally well in air. By matter of fact, today most experimental work on LIFT is done in air. Therefore, we also chose to study LIFT processes in the conditions in which it is usually done.

Optical and scanning electron microscopy (SEM) as well as EDAX analysis was performed to the substrates, pattern of many different materials on substrates.

3. Experimental results:

As stated before, this is preliminary study aiming at optimising the laser parameters and the foil/substrate couple in order to improve the efficiency of LIFT (transfer yield and transfer uniformity). Therefore we performed many irradiation experiments by changing the foil thickness, the laser energy, the foil and the substrate materials.

In the first set of experiments, we changed the substrate and maintained a fixed foil material and thickness (either Al at 4 μm or Au at 1 μm). Results are summarised in Table 1.

Table 1 First set of experiments, changing the substrate:
(a) transfer of 4μm foils of Al (b) 1μm of Au

Substrate Material	Remarks/ Adhesion	Substrate Material	Remarks/ Adhesion
Cu	good	Cu	good
Si	good	Si	good
Ni	good	Agate	good
Pb	poor	SiO2	good
Zn	poor	Zn	good
Pd	poor	Quartz	Nothing

The second set of experiments was designed to investigate the effect of foil material. Results are summarised in table 2.

Table 2 Second set of experiments: changing the foil material (4μm thickness). Comparison of transfer on Silver and Glass substrate.

Foil Material	Substrate Material	Remarks/ Adhesion
Sn	Silver	good
Au	"	good
Ti	"	poor
Al	SiO2	good
Sn	"	poor
Au	"	Poor
Cu	"	Poor

Finally, in the third set of experiments we changed the foil thickness (either 1 or 4 μm). Results are summarised in Table 3. Although here we changed the substrate too, which does not allow drawing definite conclusions (two parameters being changed at the same time), nevertheless practically in all cases, the transfer was better with 1μm foils than with 4 μm foils.

Table 3 Third set of experiments: changing foil thickness

Foil Material	Foil Thickness (μm)	Substrate Material	Remarks/ Adhesion
Au	1	Cu	good
"	"	Si	good
"	"	Pb	poor
"	4	Silver	good
"	"	quartz	nothing
"	"	SiO2	poor
Cu	1	Sn	good
"	4	SiO2	poor

In a fourth set of experiments, we investigated the effect of laser pulse energy by irradiating the same samples with 2 mJ laser pulses. In all cases, results at reduced

energy were worse than those using 20 mJ laser pulses, especially in terms of adhesion. Of course, many more energy values could have been tested but this was not really the goal of this preliminary work. Here the variation of laser energy was not intended to explore the full parameter space but rather just to try to get a "working" point where we could perform meaningful experiments.

Optical microscope images of Al deposited on Agate are shown in fig.1. SEM image at the deposited Al on Agate, as well as the EDAX spectrum are shown in fig 2.
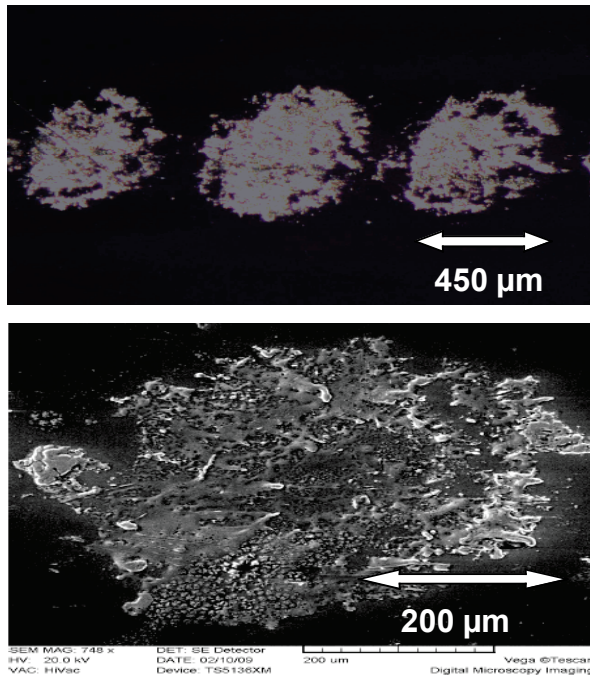
Fig. 1 Optical microscope image (top) and SEM image (bottom) of the deposited Al on Agate.
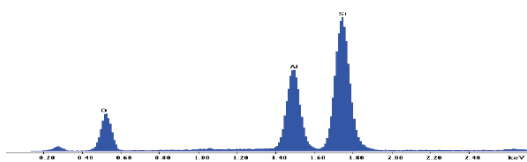

Fig. 2 EDAX spectrum showing quantitative analysis of the Agate with evidence of Al deposits

In particular, it was observed with the naked eye that forward transfer of Al was induced and Al deposited on the Agate surface. Adhesion of the deposited film was very good. It was not possible to remove it from the Agate substrate even when applying a sticking tape (the so-called "tape test" is simple but largely used to test the adhesion of deposited film [17, 18]). It is a method for evaluating the adhesion of a coating to a substrate. A sticking tape is tightly applied to an area of the coating. Adhesion is considered to be adequate if coating is not pulled off when the tape is suddenly and very quickly removed. It is proved to be a simple and reliable standard test that can measure in a reproducible manner the adhesive strength of a deposited coating to a substrate.

Gold was found to be easily transferred to the silver

substrate with high adhesion. Optical microscopy and SEM images and the EDAX spectrum of the produced deposit are shown in fig. 3 and fig. 4 respectively. Figure 5 shows instead the transfer of Cu on Ag, again with a high adhesion. It's EDAX spectrum is shown in fig 6. However, we were not able to transfer Sn or Fe to Ag. Interestingly, the measured areas of the deposited Al on Agate, Au on Ag and Cu on Ag were not comparable.
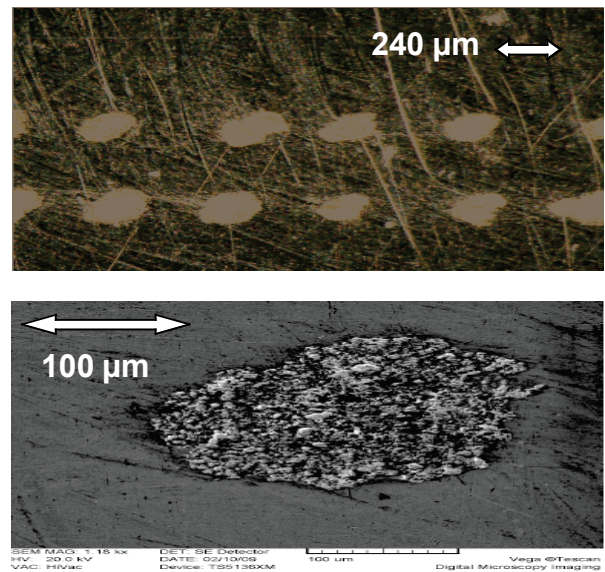

Fig. 3 Optical (top) and SEM (bottom) image showing Au deposited on Ag
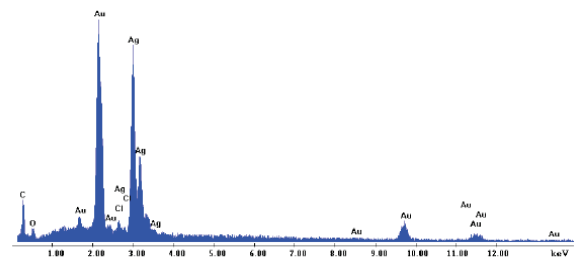

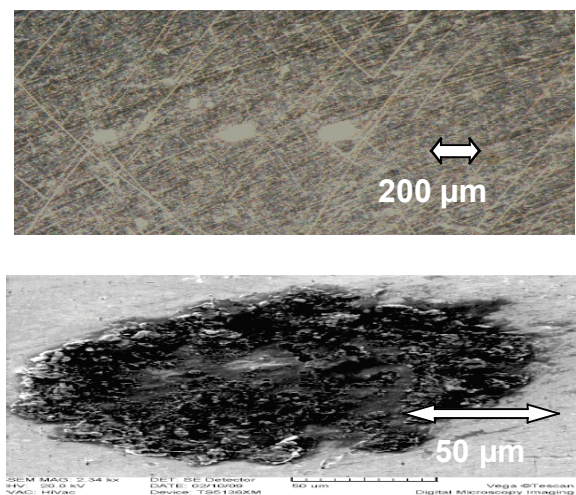**Fig. 4** EDAX spectrum of Au deposited on Ag

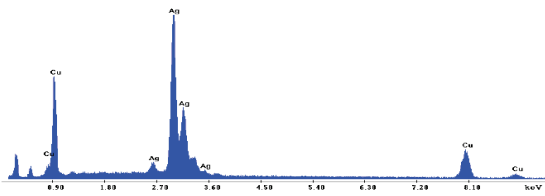**Fig. 5** Optical (top) and SEM image (bottom) of Cu deposited on Ag

Fig. 6 EDAX spectrum of Cu deposited on Ag on Ag

Finally, we also noticed that Al of 4 µm was successfully deposited on Si (see fig. 7). It is EDAX spectrum is shown in fig 8.
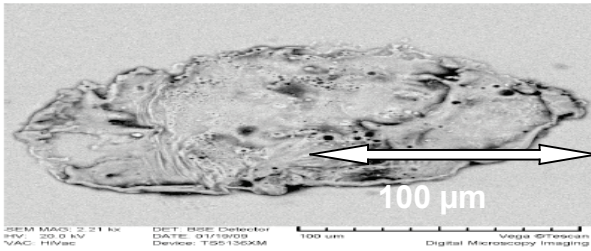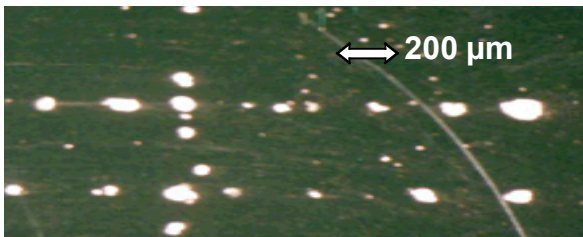

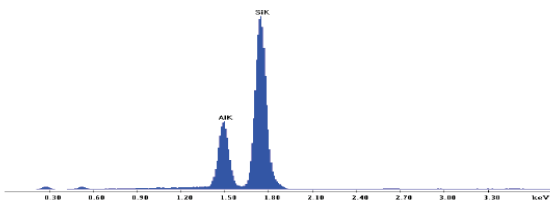**Fig. 7** Optical (top) and SEM image (bottom) of Al deposited on Si

Fig. 8 EDAX spectrum of Al deposited on Si

In contrast, the irradiation of 5 µm Au foil in close contact to Malachite showed no deposition on Malachite. Similar results were obtained when using Sn and Cu foils instead of Au foil. Again, we notice how this work is preliminary and will continue. Due to the many parameters playing a role in LIFT, it is indeed difficult to get a full picture of the phenomenon and optimize it over all such experimental parameters. However, this result seems encouraging about LIFT technique as a possible approach satisfying the needs of jewellery industry.

4. A model for LIFT

In order to gain some general understanding of LIFT in the ps laser pulse duration regime, it is important to try to physically model our results. As just explained before LIFT takes place in 3 steps [2] namely: (a) a laser pulse impinges on a donor film vaporizing a portion of it. (b) plasma expansion ejects a piece of donor film at high velocity (c) the ejected film, in the form of micro-sized clusters, bonds with the acceptor substrate (see Fig. 9)
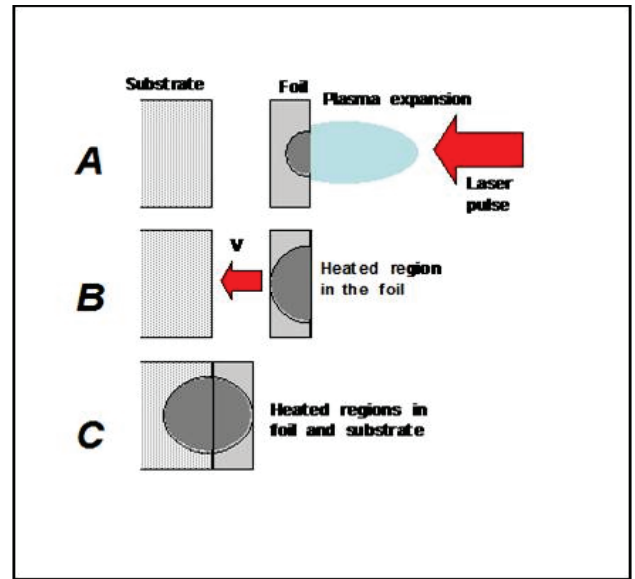

Fig. 9 Different phases of LIFT: up) ablation. The laser pulse vaporizes the surface layer causing vaporization and plasma expansion in vacuum and the propagation of a heat wave in the material; centre) motion at high velocity (V) of the film; c) impact with the substrate and creation of heated regions in foil and substrate.

The process was initially described by Bohandy et al. [1] as the propagation of a non-linear heat wave in the material. According to the authors, the necessary conditions for producing a good LIFT were that the heat wave was so sufficient to produce the melting of the foil. Clearly this model may work at relatively low laser intensities but fall in the ps regime where higher intensities are used and then the heat wave changes to a laser-driven shock. Moreover, in their model they completely neglected the phases of motion and impact between the foil and substrate, which are also essential. Indeed, several experimental results, including ours, show that the same foil (material and thickness) may adhere well to one substrate and in a worse way to another, thereby pointing to the role of the foil/substrate couple.

Let's notice that at the flux we used (≈ 13 J/cm^2), the laser intensity on target is of the order of 3×10^{11} W/cm^2 and the nonlinear heat wave really becomes a shock wave of pressure ≈ 0.3 Mbar travelling in the foil material. At the time of shock breakout, i.e. when the shock reaches the target rear side, the material relaxes and a decompression wave travels back into the material (all these process are for instance described in [19]). Now depending on the shock pressure, the material may reach a temperature either above the boiling point of the material (causing vaporization of the rear side) or above the melting point (causing fusion of the material). Finally, the target is set in motion with velocity $V = P\tau/\rho d$ (where ρ and d are density and thickness of the foil respectively and P is the shock pressure, (i.e. the ablation,

pressure) and impacts the substrate. Again, at impact, a shock is generated in both the substrate and foil, as described in [20].

The dynamic of the high intensity shock wave can be described using the Rankine-Hugoniot relations which represent mass, momentum and energy conservation across the shock front i.e. a system of three equations with six independent physical variables (pressure P, internal energy, U, fluid velocity u, shock wave velocity D, solid and shocked material density (ρ,ρ')). The RH relations in the high pressure limit $P \gg P_0$ (where P_0 is the initial pressure) are the following

$$D\rho = (D - u)\rho' \qquad (1)$$

$$P = \rho Du \qquad (2)$$

$$U - U_0 = Pu - \frac{1}{2}u^2 \qquad (3)$$

This system brings to well known formula where one use the perfect gas form EOS (equation of state). However, in order to put better description of material behaviour we used the SESAME EOS developed at Los Alamos[15]. From those tables we can obtain the relations between all the above mentioned relevant physical observables. We are interested in estimating these physical observables during the relevant phases of our experiments, which are,: (a) laser-foil interaction. (c) foil-substrate interaction. The initial experimental parameters are: the laser parameters (wavelength, Intensity, pulse duration) and the initial density of both the foil and the substrate materials.
To estimate the "expected" pressure on foil surface we used the following well known relation between the laser parameters and the induced pressure on the irradiated foil [21]:

$$P(Mbar) = 8.6 \left(\frac{I(W/cm^2)}{10^{14}}\right)^{2/3} \lambda^{-2/3} \left(\frac{A}{2Z}\right)^{1/3} \qquad (4)$$

From this using RH relations and SESAME EOS we then deduces all the others quantities. The ablation pressure induces a velocity on to the foil, which can be estimated using the Newton's second law.

$$v_f = \frac{P\tau}{\rho d'} \qquad (5)$$

Here d' (the remaining thickness after ablation) differs from the initial foil thickness d because part of its mass is removed (ablated from the foil following laser interaction):

$$\left. \begin{array}{l} d' = d - d_{abl}; \quad d_{abl} = \frac{\dot{m}\tau}{\rho} ; \\ \dot{m}\left(\frac{Kg}{cm^2 s}\right) = 150 \left(\frac{I(W/cm^2)}{10^{14}}\right)^{1/3} \lambda^{-4/3} \end{array} \right\} \qquad (6)$$

Here $\dot{m}$ is the mass ablation rate and d_{abl} is the ablation depth. However for all cases of interest $d_{abl} \ll d$, so that the difference is negligible. To obtain the induced pressure on substrate (due to the foil impact) we started from the relation between shock and fluid velocities, which can be obtained using SESAME tables. This is usually represented by the linear relation:

$$D = \Gamma_i u + \beta_i \qquad (7)$$

here Γ and β are coefficients depending on the material (in particular β is the acoustic velocity in the shocked material). The above relation can be inserted in eq. 2 to obtain the pressure as a function of the fluid velocity only, for any material "i" (the so called shock polar).(here I can either represent the foil or the substrate)

$$P_i = \rho_i u(\Gamma_i u + \beta_i) \qquad (8)$$

The shock polar relation can be written for the thin film and for the substrate in the substrate reference-frame.

$$P_f = \rho_f |u - v_f|[\Gamma_f|u - v_f| + \beta_f] \qquad (9)$$

$$P_s = \rho_s u(\Gamma_s u + \beta_s) \qquad (10)$$

where v_f is the velocity of the foil, and ρ_f, ρ_s represent respectively foil and substrate initial densities.

We assume that during the impact between foil and substrate, the pressure and fluid velocity on the foil and on the substrate surfaces are the same. This is due to the momentum conservation laws. The fluid velocity ($u=u_f=u_s$) and the pressure ($P=P_f=P_s$) at the moment of the impact can then be obtained from eqs. (8) and (9).

$$u = f(R, a, b, c, v_f) \approx f'(R, a, b, c)v_f \qquad (11)$$

$$P = F(R, a, b, c, v_f) \approx F'(R, a, b, c)v_f^2 \qquad (12)$$

where

$$\left. \begin{array}{ll} R = \frac{\rho_f}{\rho_s}; & a = \frac{\Gamma_f}{\beta_f}; \\ b = \frac{\beta_s}{\beta_f}; & c = \Gamma_s / \Gamma_f \end{array} \right\} \qquad (13)$$

The functions f and F are shown in the appendix of this paper. The free gas limit is also shown. The parameters in (13) can be estimated using equation of state EOS contained in SESAME tables; in particular we get the induced pressure on substrate due to the collision with the moving foil. Tables 4 and 5 show the theoretical estimations for different materials, based on our analytical model and using the SESAME tables.

Table 4: theoretical estimation of laser induced pressure (col. 2, eq. 4), velocity of the foil due to the ablation pressure (col. 3, eq. 5) and the reached temperatures on foil surface (col. 4, SESAME). In column 5, the foil temperatures compared to the material melting point (T_M) is shown.

Foil	P(Mbar)	v_0(m/s)	T(C^O)	T_{foil}/T_M
Al (1 μm)	~0.29	~424.4	~315	~0.62
Al (4 μm)	~0.29	~104.0	~315	~0.62
Cu (1 μm)	~0.30	~131.4	~1329	~1.15
Cu (4 μm)	~0.30	~32.60	~1329	~1.15
Au (1 μm)	~0.31	~63.30	~267	~0.39
Au (4 μm)	~0.31	~15.80	~267	~0.39

Table 5 Theoretical estimation of the foil velocity due to the ablation pressure (col. 2, eq. 5), foil collision induced pressure on substrate (col. 3, eq. 12), the reached temperatures on foil surface during the collision (col. 4 SESAME), and the reached temperatures on substrate surface during the collision (col. 5 SESAME). In columns 6 and 7 the temperatures compared to the material melting point (T_M) are shown.

Foil/Substrate	v_0 (m/s)	P_{sub} (kbar)	T_{foil} (C°)	T_{sub} (C°)	T_{foil}/T_M	T_{sub}/T_M
Al (1 μm)-Cu	~424	~44	~27	~27	~0.321	~0.230
Al (4 μm)-Cu	~104	~10	~27	~57	~0.321	~0.234
Cu (1 μm)-Al	~131.4	~10	~57	~27	~0.353	~0.289
Cu (4 μm)-Al	~32.6	~2.6	~87	~27	~0.386	~0.300
Au (1 μm)-Cu	~63.3	~11	~57	~57	~0.353	~0.320
Au (4 μm)-Cu	~15.0	~2.4	~27	~87	~0.321	~0.256

The model suggests that the ablation-induced foil velocity and the reached temperature on substrate have strong influence on the LIFT processes. This compares qualitatively well with our experimental results, as an example of good adhesion (see experimental results in Table1.a) occur always with thin aluminium foil which is characterized by the highest foil velocity (line 1 Tables 3,4) We can argue also that the initial laser parameters influence the final foil velocity only, while the physical state of the foil-substrate system is foil velocity dependent only (see eq.12). The temperature on foil depends on the laser parameters while the temperature on substrate during the foil-substrate interaction depends on the foil velocity tuned by the materials proprieties then the model allows playing with the material properties in order to improve the LIFT performance. It is important to note that from the experimental results, Cu seems to be the best substrate material. This behaviour cannot be explained by our model, which involve hydrodynamic variables only. Possibly this could be due to the high thermal conductivity of the Cu material. Finally our model should allow for an optimization of LIFT experiment in that i) one should maximize foil velocity and pressure (according to eqs. 4 and

5) and ii) one should then maximize the highest pressure and temperature on foil and substrate on impact.

5. Conclusions

SEM and EDAX analysis of our experimental results show that we could realize a controlled transfer of thin films of several materials to receiving substrates using single laser pulses. At our irradiation flux ≈ 13 J/cm^2 we observed how both the laser (pulse energy) and the target parameters significantly influence the process. In particular, it is possible to have good adhesion of metals on crystals (Al on Agate) but also of precious metals onto other precious metals (Au on Ag). These results seem encouraging about LIFT technique as a possible tool in jewellery industry. We also developed a modellisation of LIFT, which qualitatively explains our experimental results. In particular in agreements with experimental results, 1 μm foils are accelerated more effectively and impact at higher speed onto the substrate. Also at reduced energy both the shock is weaker (possibly producing a temperature below the melting point) and the velocity imparted to the foil is lower. We started from the relation between shock and fluid velocities, which was obtained using the EOS SESAME. Our model shows that the ablation induced foil velocity and the reached temperature on substrate influences the LIFT processes more than the other parameters. Finally, we show that the initial laser parameters influence the final foil velocity only, while the physical state of the foil-substrate system is foil velocity dependent only (see eq.12). Moreover we can play with the material proprieties in order to improve the LIFT performance.

Appendix

We start from eqs. (9,10) and the following A1 and A2:

$$P_f = \rho_f |u - v_f|[\Gamma_f|u - v_f| + \beta_f] \tag{A1}$$

$$P_s = \rho_s u(\Gamma_s u + \beta_s) \tag{A2}$$

Since $u < v_f$ then $|u - v_f| = v_f - u$.

At the impact moment the pressure and the fluid velocities on the foil and substrate surface are the same then A_1 and A_2 gives:

$$Au^2 + Bu + C \Rightarrow u = \frac{-B - \sqrt{B^2 - 4AC}}{2A} \tag{A3}$$

Where

$$\left. \begin{array}{l} A = a_f(R_{f,s} - c_{f,s}); \quad B = -[b_{f,s} + R_{f,s}(1 + a_f)]; \\ C = v_f R_{f,s}(1 + a_f v_f); \end{array} \right\} \tag{A4}$$

$$R_{f,s} = \frac{\rho_f}{\rho_s}; \quad a_f = \frac{\iota_f}{\beta_f};$$
$$b_{f,s} = \beta_s/\beta_f; \quad c_{f,s} = \Gamma_s/\Gamma_f \qquad \text{(A5)}$$

By inserting the solution of eq. A3 into A2 we can obtain the final pressure on foil-substrate surface. As an example let us assume the free gas approximation then $\beta_f = \beta_s = 0$ and $\Gamma_f = \Gamma_s = \Gamma = (\gamma+1)/2$. The fluid velocity depends only on the relation between the material densities:

$$u = \frac{\sqrt{R_{f,s}}}{\sqrt{R_{f,s}}+1} v_f \qquad \text{(A6)}$$

If R= 1, then

$$u = \frac{1}{2} v_f \qquad \text{(A7)}$$

Assuming the same material for foil and substrate (R=1), the fluid velocity is found to be half of the foil velocity as in the inelastic collision between two objects with the same mass. Fig.A1 shows the final pressure on substrate as a function of the foil velocity for the general case compared with the free gas approximation.
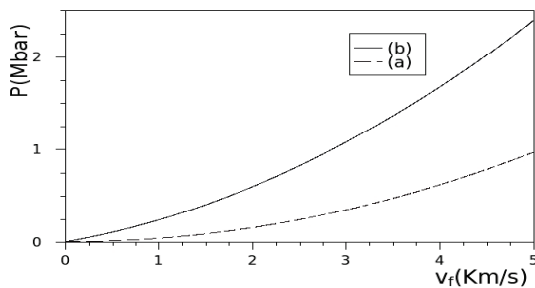

Fig. A1 Pressure on substrate as a function of the foil velocity (eq.12 or A2) Gold on Copper (a) general case (b) free gas approximation.

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