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Characterization of Thin Films Produced by Laser Induced Forward Transfer of Iron

Nafie A. Almuslet*1 and Mohammed Othman Awadallah2

1 – Institute of laser – Sudan Universirty of Science and Technology.

2 – Department of Physics – Red Sea University, Sudan.

ABSTRACT

In this work thin films were produced by Laser Induced Forward Transfer (LIFT) and characterized. Picoseconds Nd – YAG laser was used to irradiate pure samples of Fe and the plasma plume was deposited as thin films on two different types of substrate; the first one from copper and the second one from agate (SiO_2). The thin films were characterized using scanning electron microscope (SEM) and Energy Dispersive Analysis of X-rays (EDAX) in addition to scratch and scotch-tape for adhesion test. The effects of laser power density, the target thickness and the type of substrate on the homogeneity and adhesion of the films were investigated. The best conditions were: 2 X10^{13} W/cm^2 laser power density, 2 µm target thickness and agate substrate. Thin films with high quality were deduced using these conditions.

Keywords: *laser plasma interaction, LIFT, thin films, laser matter interaction.*

1. INTRODUCTION

Plasma is a gas in which molecules and atoms have been ionized to a considerable degree and for this reason plasma often is referred to as an ionized gas [1]. About 99% of our entire universe is found in the plasma state. In nature, plasmas are encountered in stars and the interstellar medium, but also closer to us in the Earth's ionosphere and in lightning. Man-made plasmas also exist in light bulbs, discharges, flat-screen televisions, and are created in short-pulse laser experiments.
When the laser interacts with the target surface, it becomes immediately a plasma environment related to many complicated physical phenomenon. The main important phenomena is the plasmas laser absorption due to different mechanisms such like rapid inverse bremsstrahlung heating of the plasma electrons [2].

Laser induced plasma has a very short temporal existence and is transient in its nature, with a fast evolution of the characteristic parameters. That is heavily dependent on irradiation conditions such as incident laser intensity, irradiation spot size, atmosphere gas composition and pressure. It is also true that these parameters vary drastically with axial or radial distance from the target surface under the same irradiation conditions. The key parameters of laser-ablated plasma plumes are density and temperature. As the degree of ionization under ordinary ablation conditions is not negligible, one needs to consider the density and the temperature of the several species constituting a plasma i.e. ions, electrons and neutral atoms. But at early times, the characteristics of the plume are governed primarily by electron contributions to temperature and density. The temperature can be estimated for the entire duration of the expansion of the plume with the aid of x-ray and visible spectroscopy [3].

The principle of Laser Produced Plasma (LPP), in contrast to the simplicity of the system setup, is a very complex physical phenomenon. It involves all the physical processes of laser-material interaction during the impact of the high-power radiation on a solid target. It also includes the formation of the plasma plume with high energetic species, the subsequent transfer of the ablated material through the plasma plume in the laser direction. Thus LPP generally can be divided into the following two stages [4]:

1. Laser radiation interaction with the target.
2. Dynamic of the ablation materials.

Each process step is very material dependent as well as dependent on experimental parameters such as laser wavelength, laser energy and pulse width. In the first stage, the laser beam is focused onto the surface of the target. At sufficiently high energy density and short pulse duration, all elements in the target surface are rapidly heated up to their evaporation temperature. Materials are dissociated from the target and ablated out with stoichiometry as in the target. The instantaneous ablation rate is highly dependent on the power density of the laser irradiating the target. Different LPP application areas, with some specific examples, are listed below [4]:

- arc plasma processing: arc welding, plasma jet cutting, steel refinery processes.
- material fabrication: single crystal growth, fabrication of small particles, powders and thin films.
- plasma chemistry: synthesis of acetylene, cyanide, oxygen and nitrogen containing compounds.
- semiconductor processing: etching, deposition and formation of thin films.

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The most important techniques used as plasma ablation for thin films formation are Pulsed Laser Deposition (PLD) and Laser Induce Forward Transfer of material (LIFT).

The general principle of the LIFT process is outlined in Figure (1). The thin film of the material to be transferred is deposited on a substrate of metal or crystal [5]. The receiving substrate is placed parallel and facing the target at a very short distance. This distance can vary from in contact to several micrometers. The laser wavelength is selected to the range of minimum substrate absorption and maximum target absorption. The mechanisms and dynamics of the transfer of material from the carrier to the receiver substrate are complex. The type of laser (i.e. its wavelength, intensity, focal spot size and pulse length or scanning speed), the type of the material (i.e. optical absorption coefficient, thermal diffusivity) and the geometry of all components determine the quality of the transferred material arrives onto substrate.

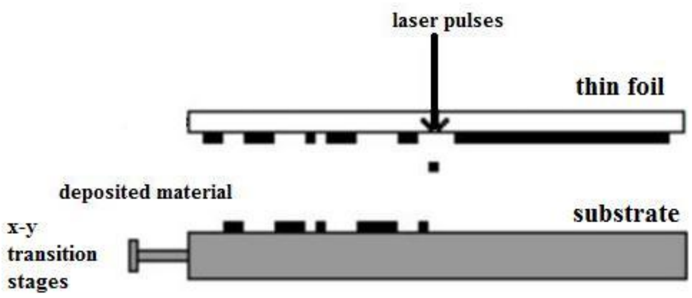

Fig (1): Scheme of the laser-induced forward transfer (LIFT) process.

A shock wave is created in a medium that suffers a sudden impact, like LIFT, or in a medium that releases large amounts of energy in a short period of time (e.g. high explosives). When a high-power laser interacts with matter, very hot plasma is created. This plasma exerts a high pressure on the surrounding material, leading to the formation of an intense shock wave, moving into the interior of the target. The momentum of the out-flowing plasma balances the momentum imparted to the compressed medium behind the shock front. The thermal pressure together with the momentum of the ablated material drives the shock wave [6].

Since the passage time of the shock is short in comparison with the disassembly time of the shocked sample, one can do shock wave research for any pressure that can be supplied by a driver, assuming that a proper diagnostic is available [7].

In 1974 the first direct observation of a laser-driven shock wave was reported [8, 9]. The science of high pressure is usually analyzed in a medium that has been compressed by a one-dimensional shock wave. For a one-dimensional shock wave traversing a known medium (density and temperature are known before the shock wave passes through), the density, pressure and energy of the shocked material are uniquely determined from the conservation of mass, momentum and energy, and the measurement of the shock and particle flow velocities. The starting points in analyzing the one-dimensional shock waves are the conservation laws of mass, momentum and energy [9]. Shocks play a dominant role in the compression phase of plasmas, especially in coating by plasma.
The aim of this work was to produce iron thin films based on plasma ablation by Laser Induced Forward Transfer of metal (LIFT), as a dynamic technique for coating, and then to characterize these thin films. We tried to make benefit from the plasma dynamic process to demonstrate micro-ablation and pattern micro-deposition production of Fe thin films and to find the optimum parameters for producing good quality homogeneous films.

2. EXPERIMENTAL PART

To successfully achieve LIFT experiments, a certain suitable setup has been performed. The basic components of the laser plasma setup are shown in figure 2.

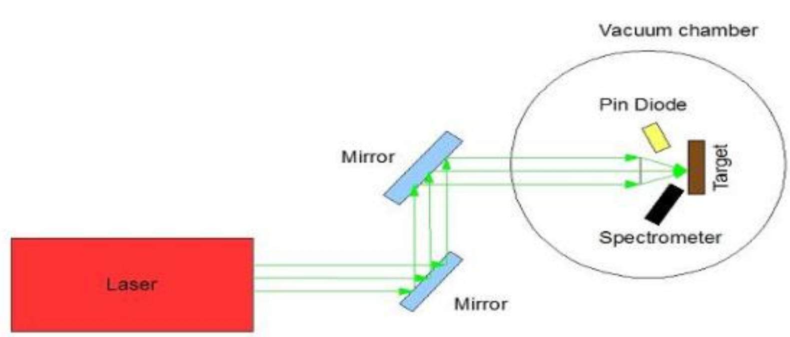

Fig (2): Sketch diagram of the experimental setup used for LIFT experiments.

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2.1 The Nd:YAG Laser Source

Q-switched Nd:YAG laser (model ND 40, supplied from spectra physics company - USA), was used in this study. The technical specifications of this laser are listed in the table (1).

Table (1): Specification of Nd: YAG Laser Model ND40

Parameter	Magnitude
Laser model	Q-switched Nd:YAG Laser
Laser wavelength	532 nm second harmonic
Pulse energy	from 15 to 35 mJ
Pulse width	40 ps
Repetition frequency	from 1 to 10 Hz

2.2 The Targets

The used targets were Fe foils. Each foil was fixed on the substrate by using double sticker tape. These foils were of 1, 2, and 3 µm thickness and 2cm×2cm dimensions. The adjustment of target-substrate distance provides an opportunity to improve the deposition quality. In this system, the target foil was placed in contact parallel to the substrate.

2.3 The Substrates

Two different types of substrates were used for deposition in LIFT experiments. They are copper and agate which is semi-pellucid crystallized quartz of SiO_2. Each substrate was well cleaned in distilled water and then dried, before the laser shoot, in order to remove the impurities and residuals from their surface to get pure deposition. The most important feature of a substrate holder was its movement relative to the target and plasma plume, so the user will be able to adjust the distance between the target and the substrate.

The target holder keeps the ablated material fixed in the vertical orientation during the LPP process.

2.4 Thin Films Characterization

The characterization of the films, deposited from the ablated material on the substrate, was done using Scanning Electron Microscopy (SEM), the ratio of the chemical components was investigated by Energy Dispersive Analysis of X-rays (EDAX) while the film thickness were measured by travelling microscope.

The SEM investigation was carried out by (FEL Quanta 200, Netherlands Company) equipped with Energy Dispersive Analysis of X-ray (EDAX).

2.5 Procedure of LIFT Deposition Experiments

The materials used as ‘source’ for the micro-deposition experiments were Fe foils with different thicknesses. The micron deposition was formed as thin film on substrates from copper and agate. Scratch and scotch-tape were used to test the adhesion degree of the film on the substrate surface**.** The distance between the target and the substrate was not varied and always fixed in the in-contact manner. The source-substrate pair was placed in a miniature cell parallel to each other under a normal atmosphere. This miniature cell was fixed onto a manual controlled linear translation stage, allowing a maximum of 45 mm movement, by means of manual feed throw. Thus, serial deposition of metal-islands and isolated dots were expected, as well as complicated pulse-generated diffractive cluster structures.

3. RESULTS AND DISCUSSION

This part is focused on the effect of LIFT parameters on the homogeneity of the deposited films. The effects of different parameters were investigated through sets of experiments.

The Ablation Threshold Effect on the Film Homogeneity:

It was important to specify the minimum laser intensity needed to produce the thin film on the substrate provided that no damage (crater) is introduced into the substrate. The LIFT threshold was examined using a single laser pulse and it was found to be, more or less, around 1 to1.5×10^{13} W/cm^2. For intensity equal to the transfer threshold, the achieved deposition was accumulated in one point and the agglomerates became denser in the central region of the transferred films. This made the morphology of the films very rough. At laser intensity higher than the threshold a deep crater on the substrate was existed and the deposited film was re-ablated again; therefore no deposition was achieved, as shown in Figures (3) and (4). Here, the target was put exactly at the focal point of the lens resulting in a phenomenon of non-linear heat effect.

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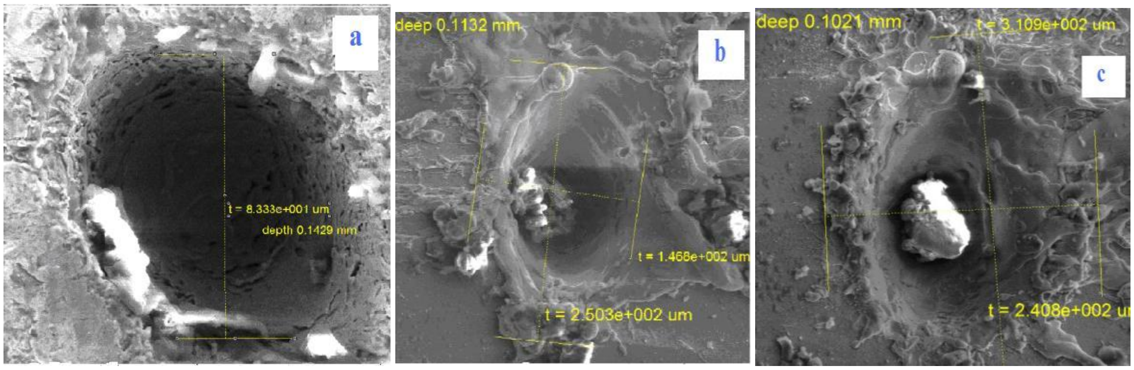

Fig (3): SEM images of Fe plasma deposited on Cu substrate, showing the laser intensity (higher than threshold) influence on the deposition quality. The corresponded laser intensity for each image was; (a): 5×10^{13}W/cm^2, (b): 4×10^{13}W/cm^2 and (c): 3×10^{13}W/cm^2.

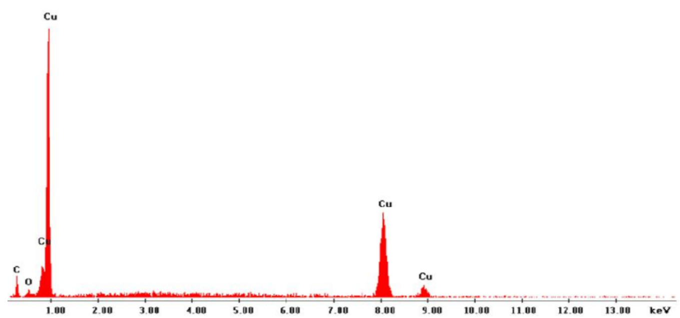

Fig (4): EDAX spectrum of Fe plasma deposited on Cu substrate at laser intensity higher than threshold. The spectrum shows peaks for Cu only, which means that no Fe was deposited on the substrate.

Using laser intensity of 2×10^{13} W/cm^2 as an optimum value gave lower crater depth and more homogenous deposited film. This was true for the examinations of the deposited film morphology using EDAX morphology test where the film has symmetric distribution.

The Effect of Target Thickness

To investigate the effect of target thickness on the film homogeneity, and to know the optimum thickness, two experimental sets were carried out with different targets thickness while all other parameters were fixed in the assumed optimum values. The laser intensity value was 2×10^{13} W/cm^2 whilst the target was put 2 cm before the focal point of the lens. The deposition results are presented in figures (5), (6) and table (2).

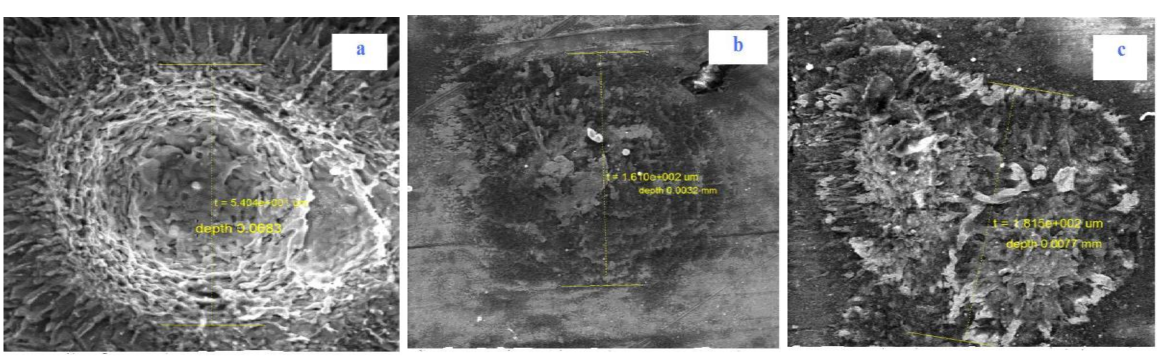

Fig (5): SEM images of Fe plasma deposited on Cu substrate, showing the foil thicknesses influence on the deposition quality. The corresponded thickness for each image was;
(a): 1µm, (b): 2µm and (c): 3µm.

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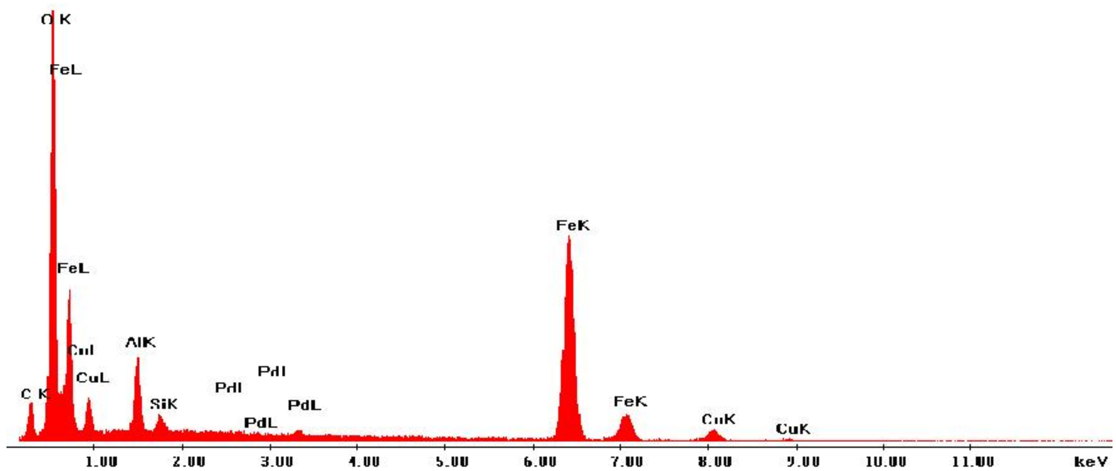

Fig (6): EDAX spectrum of Fe plasma deposited on Cu substrate. The spectrum shows a symmetric shape of Fe peaks corresponding to image (b) in Figure (5), and other peaks for Cu as a substrate.

A semi-symmetric shape of Fe peak deposited on Cu substrate in figure (6) proved the enhancement of the film homogeneity using 2×10^{13} W/cm^2 laser intensity and 2µm target thickness as optimum parameters.

Table (2): Enhancing the Deposition of Fe on the Cu substrate by changing the Fe Thicknesses

Foil thickness (µm)	Deposition diameter (µm)	Crater depth (mm)	Remarks/adhesion	homogeneity
1	39.7	0.0683	Poor	Bad
2	11.8	0.00320	Good	Good
3	59.9	0.0077	Poor	Bad

Figure (5) and table (2) insure the achievement of the deposition for all experiments with low crater depths but some defects were observed for each. In image (a), it was found that a large amount of the deposited material was not homogeneous and accumulated in one point. In image (c) the deposited material was distributed on a large area, but still not homogeneous. In image (b) there was a little enhancement in the film homogeneity as shown in figure (6). Confidently, it can be said that the achieved deposited film corresponded to the 2 (µm) as a target thickness was better than that achieved with other thicknesses.

Table (2) and image (b) in figure (5) made it clear that more homogenous film and less crater depth were achieved with 2µm thickness. Therefore, this thickness was considered to be one of the optimum values in this work. At a thickness of 1 µm the ablation threshold value reduced and hence the ejected particles have high temperature which might caused the re-ablation of the deposition and deep crater on the substrate as shown in the center of image (a) in figure (5). On the contrary, for the thickness of 3 µm, the threshold became high and the ejected particles have low temperature and hence accumulated in one point as presented in image (c) in figures (5).

The Effect of Substrate

Set of experiments was carried out to investigate whether the achieved values of the optimum parameters are valued for substrates other than copper. All parameters were kept in the optimum values mentioned before whilst the Fe foil thickness deposited on crystal from agate was varied as shown in figure (7).

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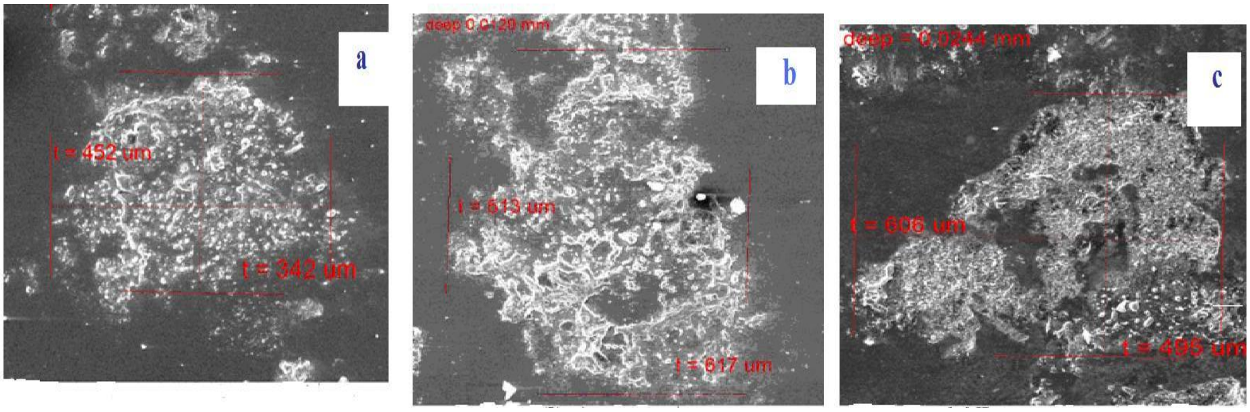

Fig (7): SEM amages of Fe plasma deposited on agate substrate, showing the foil thicknesses influence on the deposition quality. The corresponded thickness for each image was;
(a): 1µm, (b): 2µm and (c): 3µm.

Figure (7) proves the achievement of the homogeneious deposition using the same optimum parameters for the agate substrate as well as for copper substrate. Figure (7) and table (3) show the homogeneous deposition for all experimental set with low crater depth (i.e. in the range of 0.01 mm scale). Here, a good enhancement in the film homogeneity was noticed beside better adhesion than before. This means that the use of the agate as a substrate gave better results than the copper.

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Table (3): Enhancing the Deposition of Fe on the Agate Substrate by changing the Foil Thickness.

Foil thickness (µm)	Deposition diameter (µm)	Crater depth (mm)	homogeneity	Remarks/adhesion
1	397	0.053	Good	Good
2	615	0.0120	Good	Exeleant
3	550.5	0.0244	Good	Good

It is clear that the deposition was good and homogeneous on the agate, therefore the used values (laser and target parameters) were optimum not only for copper substrate, but valued also for agate substrate.

The SEM and EDAX images showed the homogeneous structure of the films using the optimum values of the deposition. All the above deposition patterns have a well-adhering features. It is important to interpret these results and find how the ejected material temperature can affect the homogeneity of the deposited film.

The dependence of the deposition on the plasma temperature come from the fact that the laser pulse heats the interface of the foil, resulting in a melt front that propagates in the direction of propagation of the incident laser beam, at that time the material at the interface is superheated beyond its boiling point and the resulting vapor-induced pressure propels material forward toward the substrate [10].

From the analysis of the obtained results mentioned above, it can be concluded that LIFT is an effective ablation process that takes place during the energy transfer from the electronic system to the ions as a result of heat effect leading to the formation of a shock pressure at the foil-substrate interface.

Based on the above considerations, the observed differences in the morphology and crater depth of the deposition feature in the LIFT experiments can be interpreted. All changes in the system occur during the heat transition. The concurrent observation that the morphology and size of the transferred films, which were changed within laser intensity scale, was a strong indication that electron-phonon interactions and the subsequent heating of the lattice was the major mechanism responsible for the observed morphological changes. So the ablated and deposited areas were subject to the heating effect resulted a distorted morphology [11].

One of the characteristic plume shapes of the ablated material was that the plume of high temperature yielded high acceleration in the direction of the smallest particles dimension of the plasma. Effect of this preferential acceleration was on the angular dependence of the flux of particles. This can cause problems for the uniformity of thin films.

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Another problem for the creation of uniform films was the presence of droplets or nano-clusters. Splashing can occur when high pressure regions appear near the molten surface of the target, sending some particles out with enough energy to reach the substrate.

Adhesion of the deposited films was very good. It was not possible to remove it from the substrate even when applying a sticking tape (the so-called "tape test" which is simple but largely used to test the adhesion of deposited films). This result seems encouraging about LIFT technique as a possible approach satisfying the needs of industrial applications.

4. CONCLUSIONS

From the obtained results one can conclude that:

1. The LIFT threshold was examined for the used iron foils, it was found to be, more or less, around 1.5×10^{13}W/cm^2.
2. The optimum parameters for achieving homogeneous film were 2 μm target thickness, 2×10^{13} W/cm^2 laser intensity, target-substrate in-contact and targets out of focal point of the lens (defocusing).
3. Very good films adhesion was achieved with the optimum parameters.
4. Using agate as substrate led to better results than using substrate of copper.

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