Study of micro-structured master fabrication on Nickel alloy by DPSS laser ablation technique

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Abstract
A direct write laser ablation process to create a micro-structure as master based on Ni alloy has been developed. The q-switched Nd:YVO_4 laser source has been applied on a Ni alloy polished surface to fabricate a well defined microstructure without litography-based technologies. The effects of different processing parameters such as laser fluence, q-switch working frequency and scan rate have been examined in high vacuum conditions (10^{-7} Torr). Both morphological and structural surface modifications of Ni alloy have been investigated by Field Effect Scanning Electron Microscopy, EDX and microprofilometer 2D. The fabrication of some masters for glass hot embossing in microfluidics application has been performed. This study aims the understanding of nanosecond pulse Nd:YVO_4 availability in Ni alloy material direct master imprinting.

Keywords: Direct Imprinting Process, Laser Engraving, Laser Ablation, Ni alloy, Zemax simulation**.**

1. Introduction
The technology for fast prototyping and master microstructures fabrication is becoming more and more important due to the increase in the use of micro-optical components in optoelectronics, optical computing and microfluidic systems [1]. The most widely known and employed microstructure fabrication techniques make use of lithography [2, 3] (photolithography, electron-beam lithography, etc.), which works well for patterning but requires time-consuming processes, expensive materials (resists, solvents and developers etc.) and elaborated equipments [4]. Direct laser imprinting lithography is an efficient, low-cost method with high resolution for master fabrication to be used in mass replication of micro- to nano-structures [5-7]. The critical issue of this technology is the laser set-up for high resolution engraving of the master. There are three types of materials commonly used: metal, silica or quartz, and polymers [8]. According to the principle of imprinting, metal is the most suitable material to be used as a master, and, because of the antisticking properties, Nickel result to be the most suitable candidate. This is due to its strong wearing to pressure under high temperatures. It can also be used repeatedly. Polymers have a low thermal conductivity and a high photochemical sensitivity. This fact limits wider applications. Silicon masters have serious drawbacks such as misalignment, tilting and brittleness [2]. Masters are typically fabricated with quartz by using an electron beam writer and an etching system [9]. This method promises a high fidelity of the produced masters, but it is very expensive and high time-consuming for large surfaces. The techniques for metal master manufacturing are mainly based on different types of lithography [10] which are extremely challenging and cost-inefficient.
In order to avoid those problems, micro channels were fabricated on glass by imprinting lithography. This technique was carried out in previous studies and good quality of channels was transferred from a Ni alloy master into soda lime glass [4].
In this paper, a novel technique, using nanoseconds DPSS laser direct writing on Ni alloy for master fabrication, is studied. In fact the laser is a flexible and effective tool with a low operating cost [11]. Laser radiation can be focused to micron-sized spots. By controlling the laser beam position through a galvanometric head, a wide variety of low cost geometries can be obtained [12-13]. Limited heat transport to minimize thermal degradation of the material outside the production zone is achieved by employing nanosecond pulse width. Short pulse widths,

under the right conditions, can promote the spallation process that minimizes the volume of molten material produced and the subsequent loss of geometry control because of its displacement. Moreover, the short pulse duration leads to high peak power, and therefore a very small heat-affected zone (HAZ) can be obtained.[14]
Nanosecond laser, due to its low cost, could be employed for a large scale of microfluidics master production. Different lasers, such as picoseconds and femtosecond,s allow a better control of the engraving and, in general, of process. Therefore undesired effects (such as debris creation) could be more easily avoid. Unfortunately the use of such lasers would increase dramatically the budget for large production.

2. Experimental

2.1 DPSS laser ablation system set up

The laser processing system employed for ablation of Ni alloy is composed by:

- Load lock chamber in which Ni alloy samples are loaded from the main process chamber (fig. 1-B and 1-A respectively)
- DPSS laser system model MLQ20 produced by Microla Optoelectronics (fig. 1-D and 1-E)
- Galvanometric scanner head (fig. 1-C)

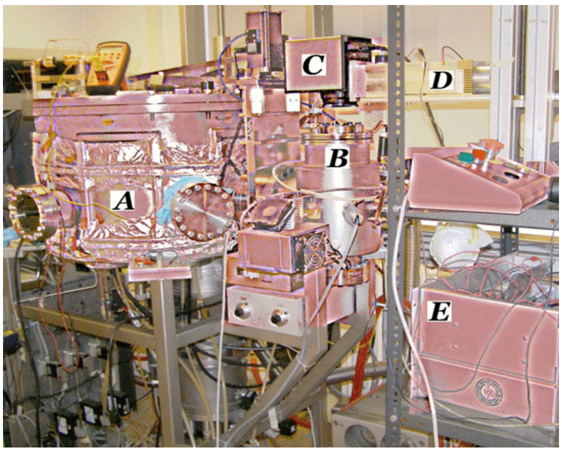

FIGURE 1: *Image of* laser processing system setup

In order to produce a surface microstructuration on Ni alloy, a DPPS laser system made by Microla Optoelectronics (MLQ-20 series) was used. This DPSS laser system has an end-pump configuration. Pumping system gives a maximum pumping power of 45W with a 808nm wavelength. An optical fibre with a core of 600 µm transports the pumping signal into the resonator. The active medium is a Nd:YVO_4 crystal doped 0.3% Nd.
This laser has an output wavelength of 1064nm linearly polarized with a maximum power density of 1300 W/cm^2 in continuous wave mode. It can be used also in q-switching mode with a 1-200 kHz range of frequencies. The shortest pulse width is 8ns (fig. 2).

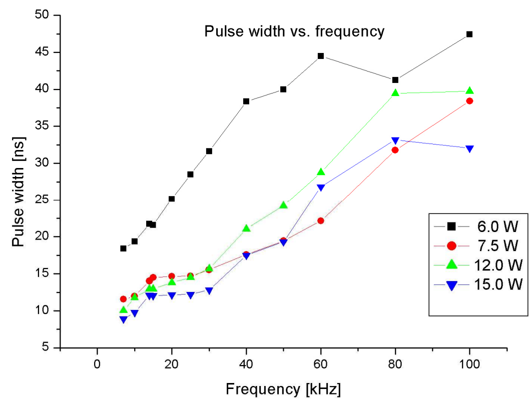

FIGURE 2*: MLQ20 pulse width vs frequency*

A galvanometric head have been used to drive laser beam towards Ni alloy substrate. In order to get into focus the laser beam on the Ni alloy substrate, a telecentric 100 mm focal θ-lens have been used.
Laser beam dimension in different points of optical path was estimated by using optical simulation software (Zemax); by using this tool it was expected to have a laser beam width of 13,3µm in the focus point (figure 3).

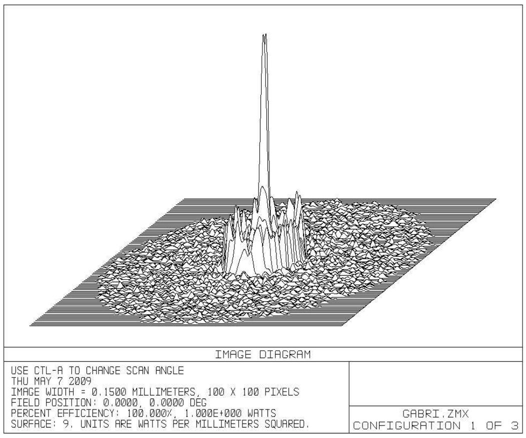

FIGURE 3: *Zemax simulation of the laser beam at the focus point with a 100 mm θ-lenses*

During laser ablation process, the Ni alloy samples have been loaded in the ultra-high vacuum (UHV) process chamber equipped with a deep-UV (DUV) silica glass window. The base vacuum level chosen for this experiment was ≈1×10^{-7} Torr,

2.2 Preparation of Ni alloy samples

Ni alloys have been prepared at Materials Science and Chemical Engineering Department of Politecnico di Torino. Studies on chemical compatibility between different metals (alloys) and soda lime glass were carried out in a previous work [4]. Further to this study, and in order to fabricate a suitable master, the Ni alloy with the composition of the list in table 1 was chosen.

Cr	Ni	Mn	Si	Mo	Cu	Fe	P
22.7	60.69	0.15	0.03	15.5	0.02	0.5	0.005

TABLE 1: *composition of Ni alloy*

The Ni alloys have been cut into 20mm×20mm slices by using Struser Sectom-10 cutting machine and grinded and polished with Logitech PM5 polishing machine.

3. Results and discussion

The analysis of laser parameters in ablation process has been performed on several 4x4mm^2 squares samples in order to check the best set-up. The influence of q-switch repetition rate, scan rate and speed of laser ablation process have been studied.

During a laser ablation process in nanoseconds range pulse-width, the material is partially evaporated while the rest of material settles on the surface forming debris. In this work a particular effort has been focused on the laser parameters set-up in order to maximize the ablation effect and minimize both the presence of debris and molten zone in laser microstructuration of Ni alloy .

While the interaction between laser beam and Ni alloy was studying, the optical power have been fixed at 15,1 W and the speed of the galvanometric head was set on 20 cm/s. This speed have been chosen because of the distance between two consecutive spots is comparable with the dimension of the laser spot. And thus, for a speed of 20 cm/s the distance between two spots engraved is 20µm at a frequency of 10kHz, 6.6 µm at 30 kHz, 4.4 µm at 45kHz, 3,3 µm at 60kHz, and 2.2 µm at 90 kHz.

3.1 2D Microprofilometer analysis

A Tencor Microprofilometer 2D have been employed for the morphological characterization of the processed samples in order to study the influence of the laser parameters on channel depth.

In figure 4 the dependence of channel depth from scan rate at different frequency values is shown.

According to Zemax simulation, in the focus point the laser beam diameter is 13,6 µm. From this analysis we found the deepest channel at 30 kHz of repetition rate.

By increasing frequency, the distance between laser spots decreases. Unfortunately, at the same time pulse width decreases as well (see characterization of MLQ-20 laser in figure 2) and so peak energy per pulse

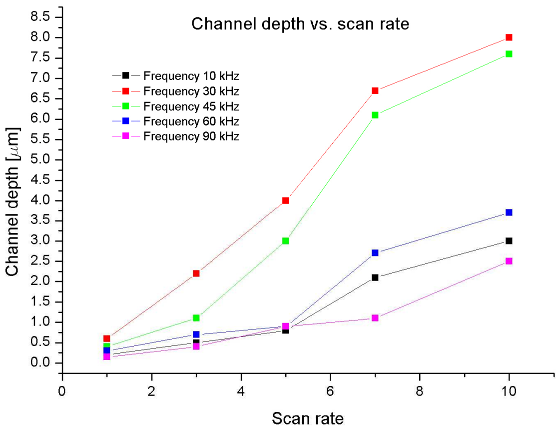

***FIGURE 4**: channel depth vs scan rate at different frequencies*

By using the results of this characterization the frequency of 30 kHz have been was found to be the most suitable one.

By using the above mentioned laser parameters debris have been investigated as high as 10% of channel's depth during laser ablation process felled near the edge of the channel (figure 5). This percentage is the lowest obtained in this processes. When the frequency decrease at 10 kHz, debris as high as 25-35% of channel's depth have been found. By using higher frequency (45 kHz, 60 kHz and 90 kHz) values, percentages of debris between 15-25% have been found.

In figure 6 a complete characterization of channel's depth using the previous conditions is shown. By using this parameters we estimated a 430 nm/pulse etch rate.

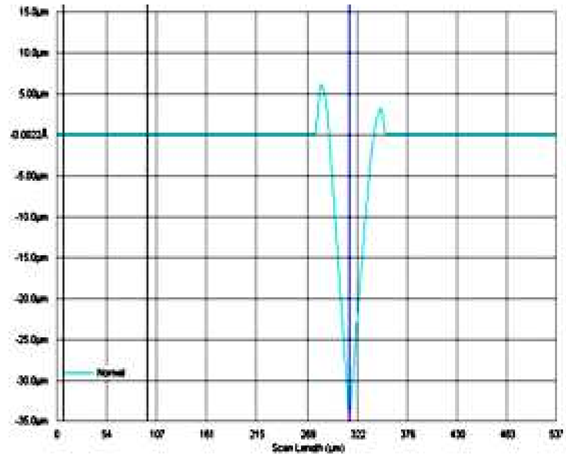

***FIGURE 5:** profilometer image channel in samples fabricated at a frequency of 30 kHz*

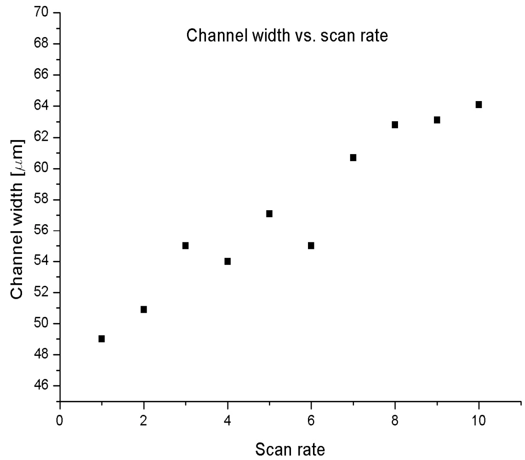

***FIGURE 6**: channel depth vs scan rate at 30 kHz frequency*

3.2 FESEM analysis

A Carl Zeiss Supra 40 Field Emission Scanning microscope was employed for the morphological characterization of the processed samples. By using Fesem we found the influence of laser parameters on channel dimension. In figure 7 the dependence of channel's depth by scan rate with best condition found with microprofilometer characterization is shown.

Fesem analysis shows the debris presence in all etched samples. The formation of this debris at the channel's edges is due to the effect of the recoil pressure on the melted Ni alloy of a plasma induced by the laser. When ablating the material, part of the energy of every laser pulse is spent to sublimate the substrate, while the other part is spent to induce and maintain a plasma where ablation occurs. This plasma exercises a pressure on the layer of melted Ni alloy higher than its surface tension, and therefore the melted material is pushed towards the edges of the channel, where it solidifies.[15]
From Fesem analysis is possible to observe that percentage of debris is lower for sample fabricated at 30kHz and 45kHz than for samples fabricated at 10 kHz, 60kHz and 90 kHz (fig. 8-11).

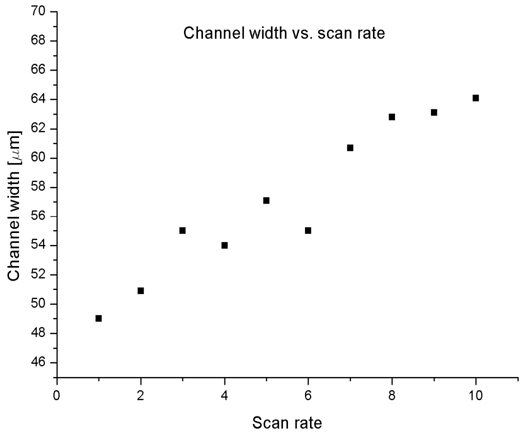

***FIGURE 7**: Channel depth vs scan rate*

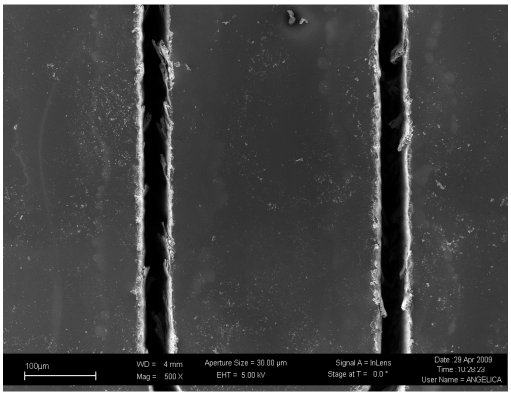

***FIGURE 8**: FESEM image of channels at 30 kHz*

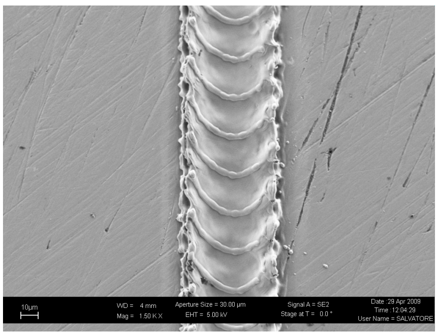

***FIGURE 9**: FESEM image of channel fabricated at 10 kHz*

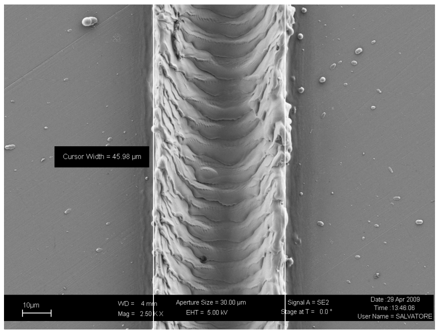

***FIGURE 10**: FESEM image of channel fabricated at 45 kHz*

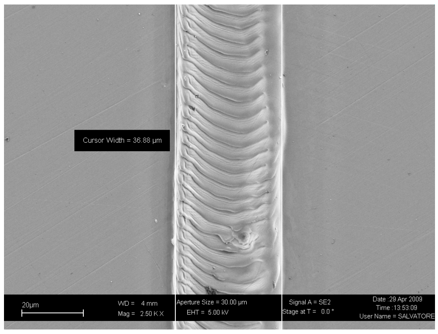

***FIGURE 11**: FESEM image of channel fabricated at 60 kHz*

An Energy Dispersive X-ray analysis (EDX) have been made to analyze composition of channel in Ni alloy processed. This analysis shows (figure 12) that in ablated channel oxygen is absent (samples were fabricated UHV condition, 10^{-7} Torr). Moreover EDX analysis shows that debris is formed by a mixture of resolidificated Ni and the other components of the alloy.

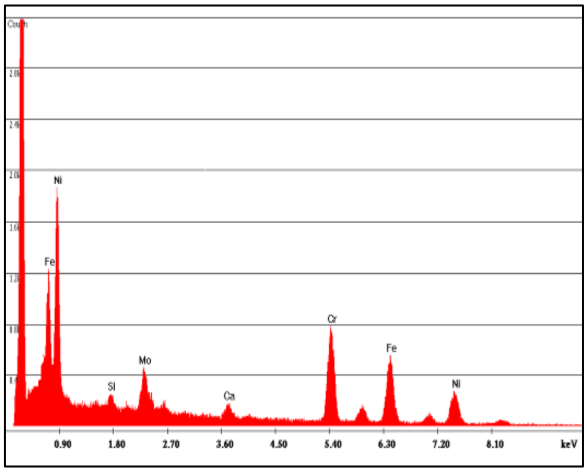

***FIGURE 12**: EDX analysis of ablated sample*

3.3 Fabrication of master prototype for microfluidics

By employing the parameters showed in the previous section (frequency 30 kHz, speed 20 cm/s), different masters prototypes have been fabricated

Masters in Ni have been adopted in order to make polymeric microfluidics devices for biomedical purposes (in particular focused on the detection of genetic mutations).

In figures 13 and 14 images of a part of a master fabricated by laser ablation (with a channel depth of about 50 μm) for microfluidics application are shown. Their profile is shown in figure 15.

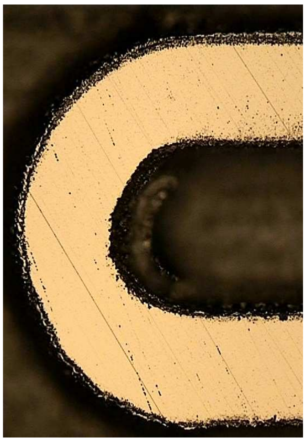

FIGURE 13: 10x optical microscope image of a part of a master for microfluidics fabricated through laser ablation (channel depth ≈50 μm)

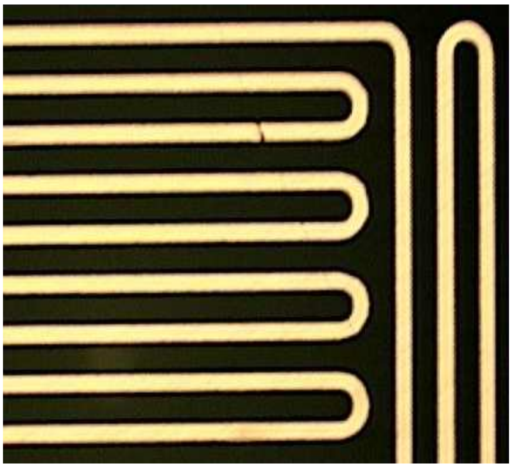

FIGURE 14: 2x optical microscope image of a part of a master for microfluidics fabricated through laser ablation

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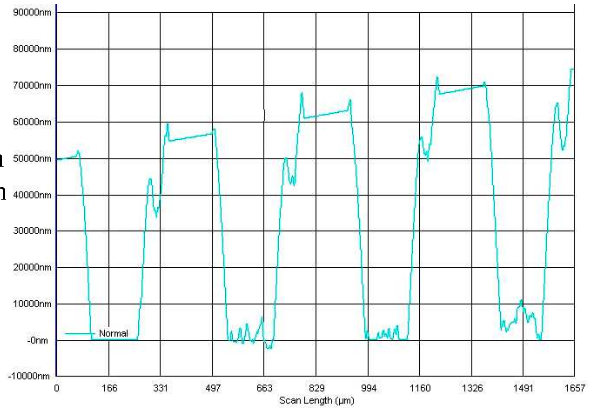

FIGURE 15: Microprofilometer profile of a master for microfluidics fabricated through laser ablation

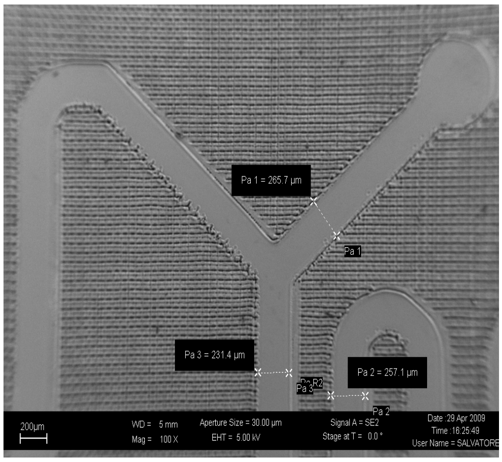

FIGURE 16: Fesem image of part of a master for microfluidics fabricated through laser ablation

4. Conclusion

The aim of this work was to analyze the nanosecond DPSS laser ablation process on a Ni alloy in ultra-high vacuum conditions. Different series of ablated samples have been prepared and characterised, with different conditions concerning q-switch working frequency and scan rate. By using Zemax simulations, laser beam dimension at the focus point have been analyzed. Fesem and 2D microprofilometer analysis allowed to characterize laser-material interaction. EDX analysis allowed to make an accurate analysis of the composition of ablated channel.

Further to those analysis, laser parameters which guaranteed highest ablation rate with the lowest debris formation have been found.

DPSS laser ablation seems to be a promising and low cost method for micro structured master fabrication

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