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X-PIC – Deliverable

FLM scaling down technological limits

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[1] R = Report, P = Prototype, D = Demonstrator, ORDP = Open Research Data Pilot, O = Other, W = Websites, patents filling, etc.

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Version	Date	Description
Version 1	31/03/2022	Version 1

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Summary

An essential goal of XPIC project is the development of integrated optical circuits in the eXtreme UltraViolet (XUV) exploiting empty channels in fused silica. In fact, this material presents a refractive index lower than one in that spectral region, making it possible to obtain low losses optical waveguides in vacuum. One essential characteristic of those waveguides are their small dimensions, indeed as the radiation wavelength is in the few tens of nanometers range, the empty channels profile should also be in the nanometric scale.

The objective of this derivable is to present the results regarding the exploitation of Femtosecond laser micromachining as a manufacturing technique for nanometer size channels in fused silica. With a good combination of laser parameters, focusing optics and sample translation speed we have been able to obtain channels with profile dimensions of some hundred nanometer in the transversal direction. These results demonstrate the feasibility of this technique for the scope of XPIC project and thus paves the way for future integrated circuits in fused silica for confinement and control of XUV radiation.

1. Experimental background: Femtosecond laser micromachining followed by chemical etching

We will exploit the femtosecond-laser-micromachining followed by chemical etching (FLICE) technique to fabricate nanometric empty channels in fused silica glass substrates. It is a two-step process:

1. First the sample is irradiated by a focused femtosecond laser beam.
2. Subsequently the irradiated region is removed by a wet-etching agent.

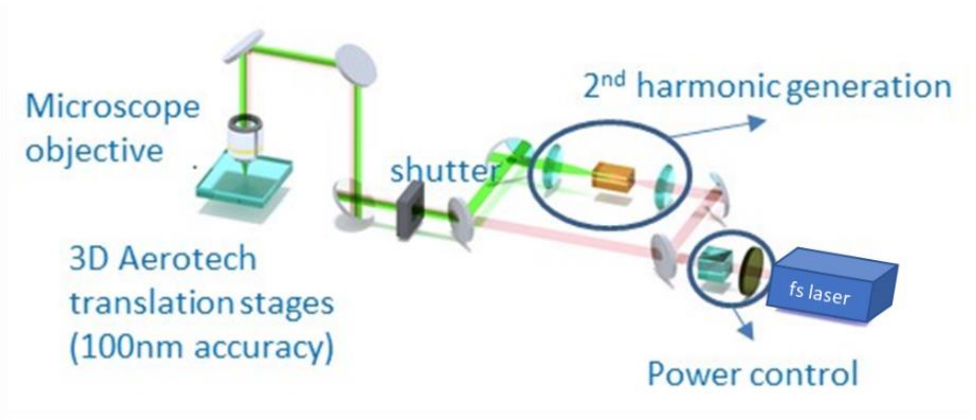

Figure 1. Femtosecond laser micromachining set-up

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A typical femtosecond laser fabrication setup is shown in Figure 1. The femtosecond laser is focused inside the material by a microscope objective. The high intensity confined into the laser focus induces non-linear effects into the material that will determine a permanent modification in that focal region [1]. Moving the sample with respect to the laser beam will allow to create three dimension modified regions embedded in the glass.

Depending on the energy delivered to the material the characteristics of the modification varies. Indeed, for intensities in the range of $I_p \approx 10^{12}$ W/cm^2 local density modifications are generated, in a nanometer scale periodical fashion that induces an increase in the material local etching rate. This effect is known as "nanograting" generation [2], and their spatial distribution is directly correlated with the relative orientation of beam polarization and sample translation (see Fig.2). So, in order to obtain the highest etching rate as possible it is mandatory to translate the sample perpendicularly to the fs laser beam polarization.

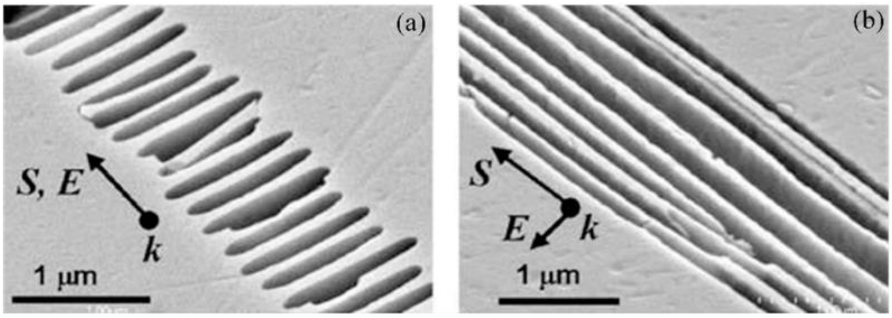

Figure 2. Polarization-selective etching enabled by the nanograting formation. Top view (XY-sections) SEM images of nanograting structures formed along the writing direction with laser polarization (a) parallel and (b) perpendicular to the writing direction. The samples were etched for 20min in 0.5% HF [3]

After the irradiation step the sample in immersed in an etchant solution in order to remove the modified region and construct empty channels buried inside the fused silica substrate. There are different etchants that might be used to create these channels, like hydrofluoric acid (HF), Potassium hydroxide (KOH) or Sodium Hydroxide (NaOH). HF gives the fastest etching rate (almost 20 times higher than the non-irradiated material) but it also has the smaller selectivity thus being not the best solution if looking for small and precise channels. KOH and NaOH allows for highly accurate channel production, but they are known to have slow etching rates, thus slowing down the manufacturing process.

Spherical aberrations

Independently of the selected regime, when performing 3D machining inside a bulk material, depth dependent spherical aberrations must be considered [3,4]. They mainly derive from the refraction of light rays at the sample surface, due to the refractive index mismatch between air and glass.

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Refraction has a nonlinear dependence on the incidence angle of the rays (Snell's law) thus deforming the intensity distribution in the focal region which results in an elongated voxel along the laser propagation direction. Spherical aberrations are strongly dependent on the numerical aperture of the objective used and on the focusing depth, which constitutes a critical issue for the homogeneity of the modified region.

2. Explored parameters

Our objective is to explore the smallest feature size of the channels fabricated by the FLICE technique, while maintaining a reasonable etching rate. We are looking for long structures (few mm in length) with profiles under the micrometer range which constitutes a challenging issue.

The first parameter that determines the irradiated track dimension is the focusing objective, the higher numerical aperture (NA) it has, the smaller is the laser spot we will obtain (voxel) and, therefore, the smaller the profile of the modified region. We decided to use an oil-immersion 100x microscope objective, with 1.4 NA (Figure 3). This microscope objective has a limited working distance of 0.170 mm, so we will irradiate our tracks next to the sample top surface. As we are looking for channels profile under 0.001mm this fabrication depth does not constitute a problem.

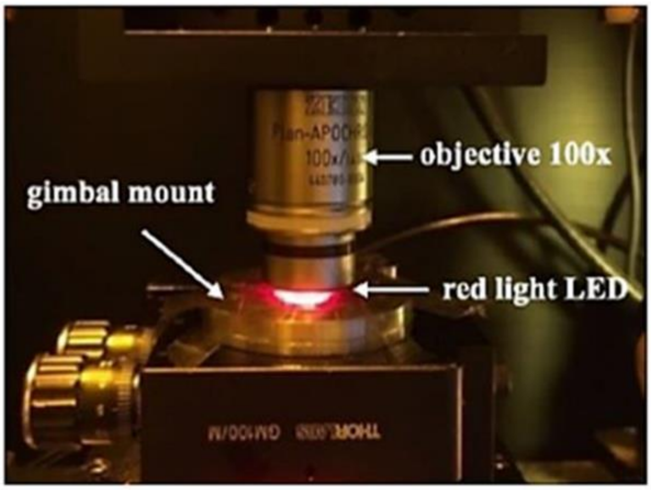

Figure 3. picture of the microscope objective and glass sample during fs laser irradiation. The gimbal is used for sample aligment. The red led light iluminates the sample for machine vision.

For the irradiation we will use the fundamental wavelength of the fs laser, the fused silica glass sample is mounted onto a high-resolution 3D movement system (Aerotech, ANT, Hampshire, UK) and moved with respect to the laser beam following a straight direction perpendicularly to the laser polarization. Each irradiated line is repeated four times in order to make have some statistics. We used two different fs laser; we started our research with the FemtoREGEN, High Q Laser GmbH,

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1040 nm wavelength that broke up after the first experiments, so we moved to the Satsuma HP laser (Amplitude) that was already present in our labs.

After the irradiation the samples have to be polished on the edges to ensure that the irradiated regions are exposed and afterwards they are chemically etched to finish the empty channel fabrication. For this selective etching we choose to use KOH aqueous solutions with 1M concentration keep at 90°C for 6 hours.

Once the samples are chemically etched they are checked under an optical microscope to measure the empty length and calculate the etching rate. To check and measure the profile of the micro or nano channels we use a scanning electron microscope due to their small dimensions (< 1µm).

The fabrication parameters that we can tailor to find the minimum feature size that is possible to obtain with FLICE are:

- **Laser repetition rate**
- **Pulse energy**
- **Sample scanning speed**

2.1 Optimization of etching rate

In a first rough parameter exploration (High Q laser) we found that with KOH solution etching rates vary from a few µm/h to some hundred µm/h. So we firstly we decided to find the best combination of repetition rate, pulse energy and translation speed to have a good etching rate. Repetition rates (RR) higher than 200 kHz resulted in low etching rates (lower than 10 µm/h). For RR lower than 200kHz, and low pulse energies the etching rates become higher and the etched tracks have an elliptical profile with a few micrometer side. In table 1 are reported the parameter combinations that give us the best etching rates, each value is the media of four experiments. As it can be seen the higher etching rates are always found for 0.1mm/s translation speed and for R.R. higher than 20kHz, being the highest value ~200 µm/h. Although the etching rates for 20Kkz R.R. are always high, the dimensions of the etched tracks are also pretty big so we decided to make a first detailed parameter tunning for higher R.R.

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Repetition rate 200 kHz		Pulse energy (nJ)			
		98	88	73	59
Translation speed (mm/s)	0,1	159	167	135	/
	0,5	63	40	133	/
	1	24	98	/	/
	5	183	165	/	/

Repetition rate 100 kHz		Pulse energy (nJ)			
		117	88	78	68
Translation speed (mm/s)	0,1	166	84	168	/
	0,5	78	60	/	/
	1	7	30	/	/
	5	140	/	/	/

Repetition rate 20 kHz		Pulse energy (nJ)			
		150	125	100	75
Translation speed (mm/s)	0,1	148	71	107	/
	0,5	214	194	208	/
	1	168	121	/	/
	5	178	183	/	/

Repetition rate 10 kHz		Pulse energy (nJ)			
		150	125	90	60
Translation speed (mm/s)	0,1	10	34	/	/
	0,5	159	/	/	/
	1	/	/	/	/
	5	/	/	/	/

Table 1. Irradiated fused silica etching rates (µm/h) for different laser repetition rates, pulse energies and sample translation speeds.

Once we know the expected etching rates, we focus on a fixed laser repetition rate of 100kHz, that gives the best etching rate / track dimension ratio (laser Satsuma HP). We expect to obtain a smaller channel if we use low pulse energies (we deposit less energy into the material), so we have to find the energy threshold to have a modification.

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Pulse energy [nJ]	Etching Rate [µm/h]	Pulse energy [nJ]	Etching Rate [µm/h]
75	198	64	170
74	184	63	188
73	195	62	181
72	208	61	178
71	205	60	/
70	196	59	/
69	199	58	/
68	198	57	/
67	182	56	/
66	182	55	/
65	177	54	/

Table 2. Modification threshold at laser Rep. Rate of 100kHz and scanning speed of 0.1mm/s

The results of are reported in Table 2. We notice that the obtained values are slightly different than the previous ones, we believe that this is due to the different laser system as it has a different wavelength but more important a shorter pulse length. Nevertheless with this new parameter scan we can conclude that at these low energies the etching rate is maintained at a rather good value around 180 µm/h.

With these parameter combination we have, to the best of our knowledge, the thinnest channels ever fabricated in fused silica with the FLICE technique, arriving in the transversal direction to some hundred of nanometers. These channels are always highly elliptical, being longer than 1 µm in the laser propagation direction. The ellipticity decreases with the pulse energy (see Figure 4) but it never reaches values under 3.

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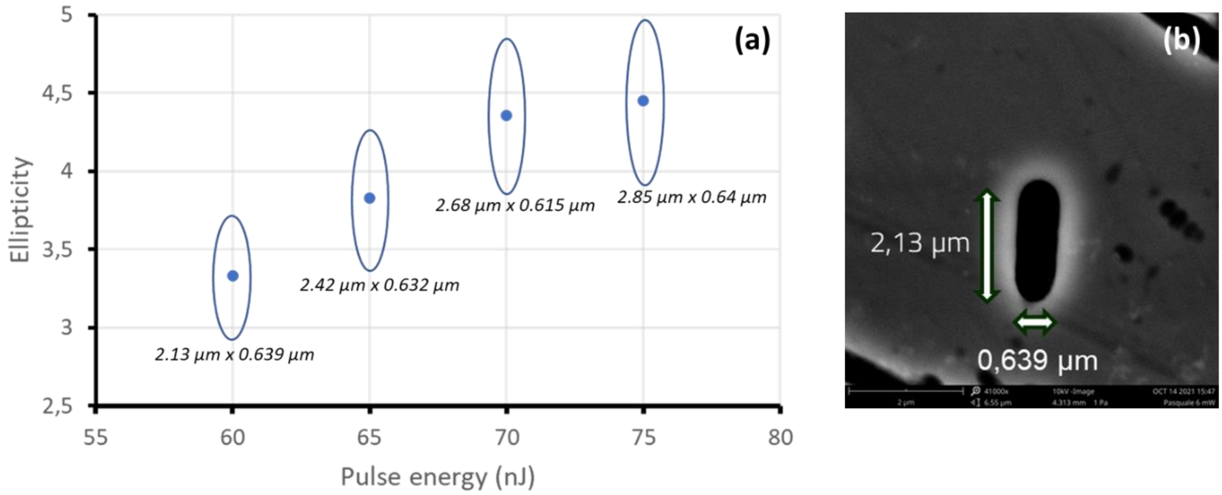

Figure 4. (a) Track ellipticity behavior with pulse energy, (b) Scanning electron microscope of the track written at 60 nJ.

2.2 Exploring lower repetition rates (low etching rates)

After arriving to our limit with 200kHz repetition rate we also explored the low etching rate regimes, to be sure that they don't give smaller features. The main problem in this regime is that we obtain extremely elliptical features, most probably due to nonlinear effects as self-focusing. To reduce their length in the laser propagation direction we decided to explore the possibility of using a longer pulse duration. In figure 5 are reported some representative examples at 10 kHz, using the same pulse energy (1µJ) and translation speed of 0.5 mm/s, with pulse durations of 300fs, 460 fs and 520 fs.

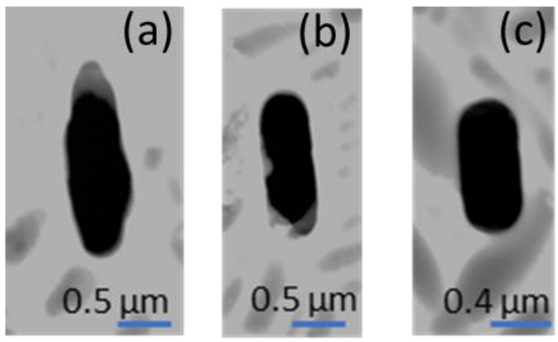

Figure 5. Scanning electron microscope images of the tracks at 10kHz with different laser pulse length of (a) 300fs, (b) 460 fs and (c) 920 fs.

In table 3 are reported the etching rates and dimension of the tracks. As expected, the etching rates are low (< 60 µm/h) thus slowing the overall fabrication process. Regardless of this slow etching rate we obtain amazing track widths in this regime, reaching values under 500nm. Unfortunately we

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are also increasing the dimension in the longitudinal direction thus increasing the ellipticity of our channels.

Pulse duration (fs)	Pulse energy [nJ]	Etching rate [μm/h]	Longitudinal length [μm]	Tansversal length [μm]	ellipticity
300	100	56	2,65	~0,500	5,3
460	100	19	2,17	0,400	5,4
920	100	9,5	2,52	0,380	6,6

Table 3. Details of the 10kHz tracks, fabricated with translation speed of 0.5 mm/s

This result constitutes a breakthrough of the micrometer limit of the FLICE technique in fused silica glass, thus demonstrating that it is possible to arrive to the nanometer scale and thus to fabricate XUV optical waveguides in fused silica with this technique, for XPIC objectives.

In the next future to obtain single mode waveguide we will need to reduce the channels ellipticity, that we believe is mainly a consequence of spherical aberrations due to refractive index mismatch between the immersion oil (1.53) and fused silica (1.45). To this purpose we will explore three different strategies:

- Fabricating the tacks next to the sample top surface as we know that the effect of spherical aberrations in focal point length is proportional to the focusing depth.
- Working with a microscope objective with an immersion fluid like silicone or glycerol that have a refractive index similar to fused silica (we are already buying this objective).
- Inserting an active optical element, like Spatial light modulator phase mask, before the microscope objective in order to compensate for these aberrations (instrument already presentbin our labs).

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