

# Selective etching of 10 MHz repetition rate fs-laser inscribed tracks in YAG

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**Abstract.** We investigated fs-laser structuring of YAG crystals at high writing velocities up to 100 mm/s using a commercial 10 MHz fs-laser system supplied by Coherent Inc. and selective etching of these structures for fabrication of ultrahigh aspect ratio microchannels. Usage of a diluted acid mixture of 22% H<sub>3</sub>PO<sub>4</sub> and 24% H<sub>2</sub>SO<sub>4</sub> accelerated the etching process significantly to an etching parameter  $D$  of 11.2  $\mu\text{m}^2/\text{s}$ , which is three times higher than previously reported. Additionally, the selectivity of the etching process was increased by an order of magnitude.

## 1 Introduction

Selective etching of fs-laser inscribed structures has become a huge research field. Three-dimensional hollow micro- and sub-microstructures with aspect ratios of length to diameter in excess of 8000 [1] find applications in e.g. micro- and optofluidics, microsensors, photonics, and integrated lab-on-a-chip applications [2]. For glasses the process has been extensively studied and even finds industrial applications [3], but it is also applied for crystals such as sapphire [4], YAG [5], quartz [6], and CaF<sub>2</sub> [7].

The fabrication time for complex hollow structures is mainly limited by the selective etching rate, especially in crystals [8]. Nevertheless, also the fs-laser writing velocity can be a limiting factor [9] considering that lots of samples could be etched at the same time, while writing one single track requires one writing laser beam for each track and complex structures easily consist of a huge amount of written tracks.

For this reason, we systematically investigated the fs-laser structuring process using a 10 MHz-pulse repetition rate laser system. Due to the high repetition rate, a spatial pulse overlap is warranted also at very high writing velocities. In this way we reach up to 100 mm/s writing velocity, limited only by the maximum translation speed of the used translation stages. Furthermore, we investigated the selective etching behavior of the MHz-writing rate inscribed structures in YAG for the first time. Using the diluted acid mixture 22% H<sub>3</sub>PO<sub>4</sub> 24% H<sub>2</sub>SO<sub>4</sub> we were able to accelerate the etching process compared to the undiluted mixture and to increase the etching parameter  $D$  by a factor of 3 to 11.2  $\mu\text{m}^2/\text{s}$ , while increasing also the selectivity by an order of magnitude. Using a precision dicing saw we excavated the channel walls to measure the channel wall roughness for the first

time. A low roughness is crucial for photonic applications such as etched waveguide structures [1,10], gratings and lenses or resonators, what is known from waveguides fabricated using lithography [11].

## 2 Experimental procedure

For the fs-laser inscription we used a 10 MHz repetition rate *Fidelity HP High Energy* Yb-doped fiber laser oscillator supplied by Coherent Inc. The laser had a maximum output power of 10 W, 140 fs pulse duration, a center wavelength of 1040 nm, and a nearly diffraction limited beam quality of  $M^2 < 1.3$ . The beam was focused 364  $\mu\text{m}$  deep into the polished surfaces of YAG crystals using focusing lenses of an  $NA$  of 0.50 and 0.55 (3.1 mm and 4.5 mm focal length, respectively). The laser beam polarization was chosen either parallel ( $\pi$ ) or perpendicular ( $\sigma$ ) to the writing direction (along the x-axis cf. Fig. 1 a)) which was perpendicular to the structuring laser beam (z-direction). Tracks were written along the [111] and [100] direction to investigate the influence of the crystal lattice orientation. For this purpose, the samples were translated on *Aerotech ABL1000* stages with velocities between 1 - 100 mm/s at average inscription laser powers of 0.54 - 1.85 W. More details on the fs-laser structuring setup can be found in [8].

We investigated the etching process using different etching agents at different temperatures including 43% H<sub>3</sub>PO<sub>4</sub> 48% H<sub>2</sub>SO<sub>4</sub> at 83°C, 90°C, and 105°C and 22% H<sub>3</sub>PO<sub>4</sub> 24% H<sub>2</sub>SO<sub>4</sub> at 83°C with respect to the characteristic etching parameter  $D$  and the selectivity  $S$  as defined in [8]. For the microscopic investigations, we used a *Keyence VHX7000* digital microscope.

To access the channel wall roughness, we cut slices containing one or two channels out of the samples with

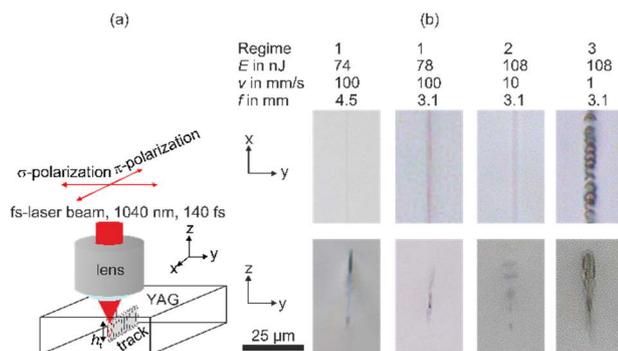
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several channels inscribed, using a precision dicing saw *Disco D322*, first. The cuts were performed along the  $xz$ -plane. The channels were accessed from  $y$ -direction by increasing the cutting depth perpendicular to the  $xz$ -plane in 10 - 20  $\mu\text{m}$  steps until the top of the channel was removed. In this way, we gained access to a direct measurement of the sidewall roughness with a laser-scanning microscope *Keyence VK-X*. The surface roughness was measured using the microscope software. Since the effort for this cutting procedure is very high, we limited the study of the channel wall roughness to selected processing parameters including the influence of the writing direction ([100] or [111]), the etching temperature, the focal length of the inscription beam as well as the etching agent concentration.

### 3 Results

Depending on the inscription pulse energy, we observed three regimes of material modification during inscription with 10 MHz repetition rate (cf. Fig. 1 b)). Pulse energies above 100 nJ lead to cracks, while those below 60 nJ did not lead to continuous track inscription. In the second regime the focus is split in vertical direction (cf. Fig. 1 b), regime 2). This vertical segmentation could result from self-focusing effects [1]. Third regime structures were strongly broadened by heat accumulation [12].

The most suitable regime (regime 1 in Fig. 1 b)) occurred at writing velocities of 30 to 100 mm/s and pulse energies between 60 and 100 nJ. These tracks were smooth and well confined, 15 to 40  $\mu\text{m}$  in height and only 2 to 3  $\mu\text{m}$  in width, similar to those observed at 1 kHz inscription frequency, and we regard this regime to be the only suitable scheme for selective etching of smooth hollow channels.

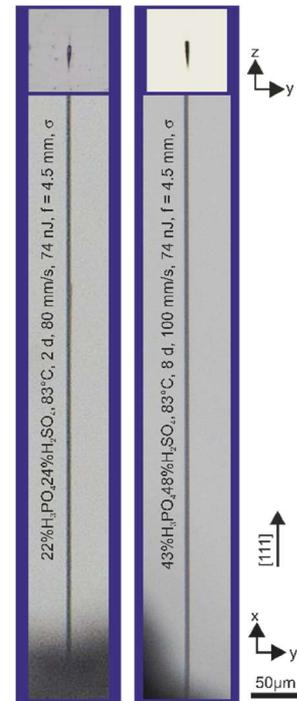


**Fig. 1.** Light microscope images of fs-laser written tracks inscribed in [111]-direction with  $\pi$ -polarization and the  $f=3.1$  mm lens, and  $\sigma$ -polarization with the  $f=4.5$  mm lens.

The etching within these tracks was highly selective for both 43% $\text{H}_3\text{PO}_4$  48% $\text{H}_2\text{SO}_4$  and 22% $\text{H}_3\text{PO}_4$  24% $\text{H}_2\text{SO}_4$  at 83°C and decreasing for higher temperatures. Due to this high selectivity, the diameter of the channels is mainly determined by the dimensions of the fs-laser inscribed material modification (cf. Fig. 2).

With regime 1 structures selectivity values in excess of 5000 were reached by etching tracks written in  $\sigma$ -polarization with a lens of 4.5 mm focal length and the diluted acid mixture at 83°C. The writing direction did not

influence the selectivity significantly, neither did the pulse energy and writing velocity if chosen within the parameter range for regime 1. Focusing with a lens of 4.5 mm focal length though, leads to a higher selectivity and a larger etching parameter than using a lens of 3.1 mm focal length. With the 43% $\text{H}_3\text{PO}_4$  48% $\text{H}_2\text{SO}_4$  etchant, the selectivity was lower and no influence of polarization or writing direction was found.



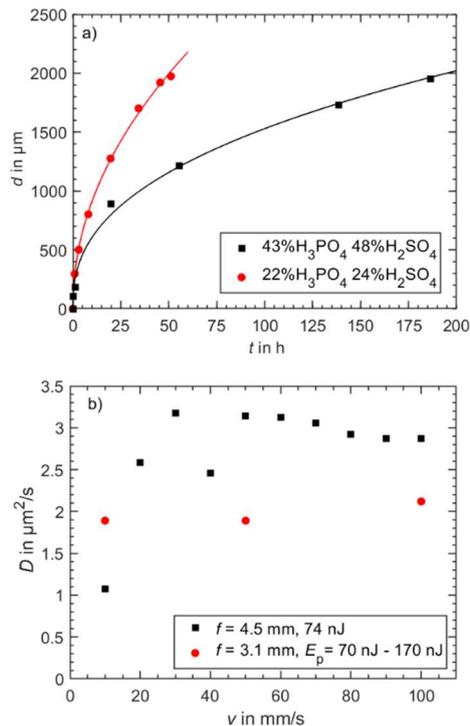
**Fig. 2.** Light microscope images of selectively etched channels. Writing direction and etching parameters are denoted in the image.

The etching depth  $d$  showed a dependence on time  $t$  like

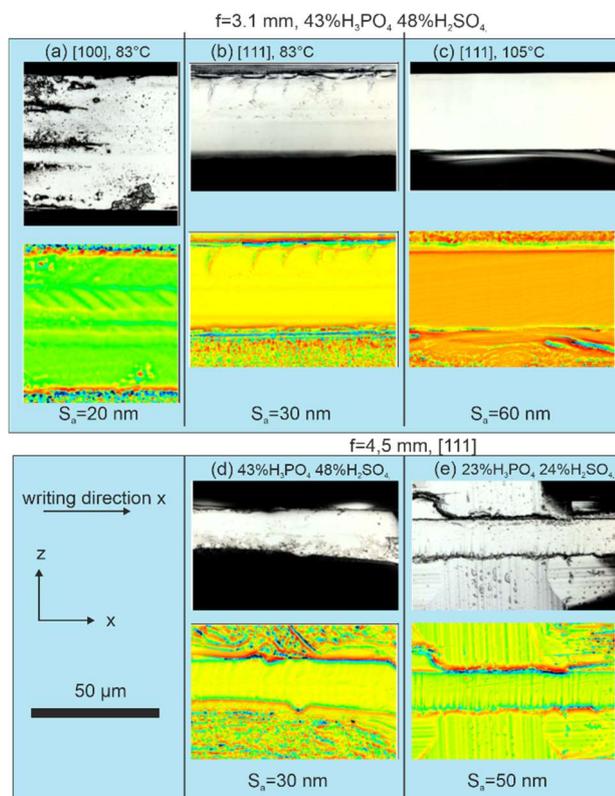
$$d = (2 \cdot D \cdot t)^{1/2}. \quad (1)$$

The fitting parameter  $D$  characterizes the selective etching progress. Figure 3 a) shows the etched depth vs. time for both investigated etching agents at 83°C. Different than for tracks written at 1 kHz [8] no significant influence of the writing velocity on the etching velocity is observed (cf. Fig. 3 b)), enabling to use unprecedented inscription writing velocities of up to 100 mm/s. This is only a factor of 2 below the fastest fs-laser writing velocity in glasses [9] and only limited by the translation velocity of the stages used in this experiment.

The largest etching parameter value of  $D = 11.2 \mu\text{m}^2/\text{s}$  was found for tracks inscribed with a focusing lens of 4.5 mm focal length,  $\sigma$ -polarization along the [111] direction and pulse energies between 60 and 100 nJ etching with 22% $\text{H}_3\text{PO}_4$  24% $\text{H}_2\text{SO}_4$  at 83°C (red curve in Fig. 3 a)). These also showed the highest selectivity. Extrapolation of the fit yields a total etching time of 13 days required for etching a 1 cm long



**Fig. 3.** a) Etched depth  $d$  vs. etching time  $t$  for both etching agents and first writing regime tracks and fitting curves. b) Fitting parameter  $D$  vs. writing velocity  $v$  for tracks etched in 43% $H_3PO_4$  48% $H_2SO_4$  at 83°C, the writing parameters are denoted in the figure.



**Fig. 4.** Laser scanning microscope images of the opened selectively etched channels of regime 1 inscribed at 10 MHz. The different processing parameters are denoted in each picture.

channel from two sides. This is a factor of 3 shorter than under the previous fastest etching conditions (43% $H_3PO_4$  48% $H_2SO_4$  at 105°C, with a selectivity < 1000).

Fig. 4 shows microscope images of the channels opened to determine the side-wall roughness. We did not observe any significant influence of the writing direction on the surface roughness of the channels. The channel cross section a) seems to be larger compared to b) - e) because of a rhombic cross section of the channel etched along [100] instead of the nearly rectangular channels obtained for [111] direction. We obtained an average surface roughness  $S_a = 27 \pm 3$  nm for etching with 43% $H_3PO_4$  48% $H_2SO_4$  at 83°C, independent on the focusing conditions (cf. Fig. 4 b) and d)), though the height of the channels written with the lens of  $f = 3.1$  mm is larger. Higher etching temperatures as well as a diluted acid increase the average surface roughness to  $S_a = 55 \pm 4$  nm. Overall, the surface roughness is in the range of  $\lambda/20$ , what is typical for laser polish.

Nevertheless, compared to surface roughness values obtained using lithographic methods or chemical mechanical polishing ( $S_a < 1$  nm [11]) the obtained roughness values are high. But, since the processing parameters were not yet investigated systematically with respect to reducing the surface roughness, it is likely that parameters yielding smoother channel surfaces exist. Overall, since the surface roughness is already comparable to laser polish, this is a promising result for future optical applications of selective etching.

## 4 Conclusion

For the first time we used a 10 MHz repetition rate laser system for fs-laser structuring of YAG. We achieved record-high writing velocities of up to 100 mm/s and identified inscription properties suitable for successive selective etching of the inscribed tracks. Using a diluted mixture of 22% $H_3PO_4$  24% $H_2SO_4$  at a temperature of 83°C three times improved etching parameters of up to  $D = 11.2 \mu m^2/s$  were achieved, enabling etching of 1 cm long microchannels in less than 14 days. We found the surface roughness of the channel walls to be below 60 nm. Our results represent a significant improvement towards future industrial applications of selective etching of fs-laser written structures in YAG concerning the enhanced processing speed. The obtained roughness parameters show the applicability for photonic applications.

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