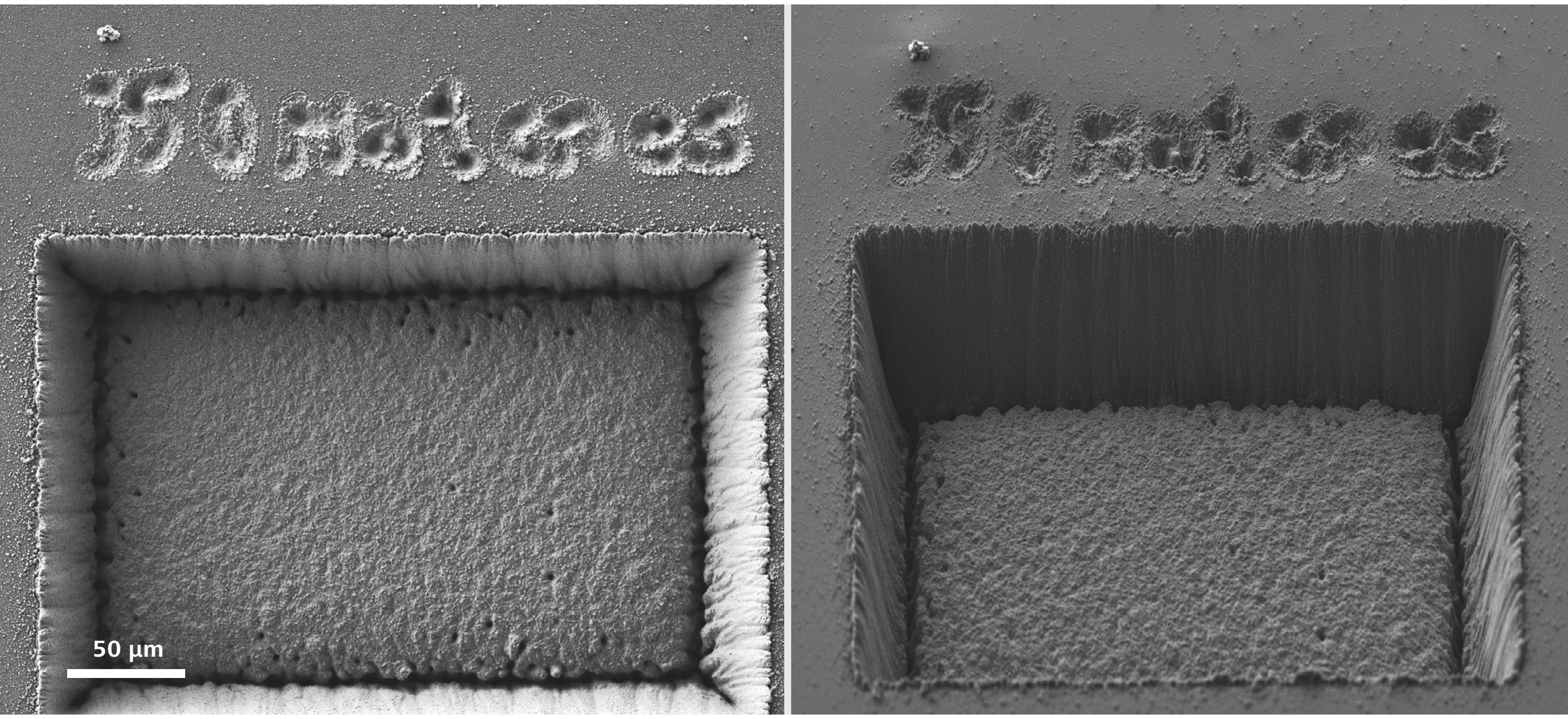
Best-Known Method

Calibration Procedure for Finding Suitable Milling Parameters on ZEISS Crossbeam laser



Seeing beyond

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A FIB-SEM (focused ion beam scanning electron microscope) has long since been an essential tool for microscopy users when intending to perform sample preparation and subsequent analyses. High throughput, seamless workflows and best resolution in 3D imaging and analysis characterize ZEISS FIB-SEMs of the Crossbeam family. The LaserFIB, a particular model of the ZEISS Crossbeam family, offers rapid massive material ablation with a green femtosecond laser, keeps the FIB-SEM chamber clean by performing laser processing in a dedicated chamber and enables site-specific preparation of deeply buried structures. The combination of laser and FIB processing enables preparing a multitude of samples e.g., cross-sections, TEM lamellae, atom probe tomography samples, pillars for microcompression testing etc.. Finding suitable parameters for efficient laser processing can be challenging because of the wide variety of laser and hatch parameters. To facilitate exactly that, the LaserFIB now comes with pre-installed recipes. This will enable users to concentrate on their science instead of spending more time than absolutely necessary to develop methods.

Introduction

Finding suitable parameters for efficient laser processing on a new material can be challenging because of the wide variety of laser and hatch parameters that can be altered. Furthermore different sets of parameters may be necessary for different applications on the same material. In this best-known method, an approach is provided to guide users through multiple steps of parameter optimization for one specific application in single-pulse operation. For each step, a single .VLF file is provided, which only requires a few modifications depending on the geometry of the used sample. By using this approach, users can easily create sets of parameters for different applications and different materials. Three possible applications and therefore sets of parameters are considered in this procedure: I) A rough trench cut into the material with an area >1 mm², where a high volume of material is removed as quickly as possible and floor surface quality is disregarded. II) A fine trench cut into the material, optimizing floor surface quality but limiting the removal rate. III) A polishing cut to fine polish a side face of a trench or edge of the sample, which can be used to reveal the underlying microstructure. All three sets of parameters have been evaluated for silicon, steel, and copper and are provided in pre-defined recipes. As an example, the following parameter optimization procedure is shown on silicon for the fine trench cut application. In addition to the above-mentioned use cases, the procedure can be used to find suitable parameters for specific combinations of milling geometry and hatching strategy in order to give a satisfactory result for any desired application.

Step 1 – Laser Power Alignment

The energy per pulse is an important parameter to alter, since it highly affects the physical processes taking place. When setting up parameter optimization it is highly recommended that users verify a suitable range of the energy per pulse first to ensure a well-defined ablation process. The energy per pulse can be altered by modifying the laser power in the laser parameter settings. The maximum power of one pulse at 100% laser power is 10 µJ. The resulting fluence, which is defined as the energy per area, is listed in Table 1 for a variety of laser power parameters, using a spot size diameter of 15 µm.

Laser power setting in %	Energy per pulse in µJ	Fluence per pulse in J/cm²
10	1	0.14
20	2	0.28
30	3	0.42
40	4	0.57
50	5	0.71
60	6	0.85
70	7	0.99
80	8	1.13
90	9	1.27
100	10	1.41

Table 1: *Energy per pulse and fluence of the laser for various laser power parameter settings.*

In order to achieve an effective ablation process, a value above the ablation threshold ϕ_{th} is necessary to control the interaction of matter and the laser beam. Spallation of the scanned material occurs in an energy range of approximately 1-10 ϕ_{th} for metals. A maximum energy-specific removal rate can be achieved within this energy range and is highly dependent on the ablated material. For example, steel (1.3401) is reported to have an ablation threshold of 0.1 J/cm² and a maximum energy-specific removal rate at a fluence of 0.5 J/cm² [T. Kramer et al., JLMN 2017, 12, 107-114].

For an empiric approach to find a suitable laser power for a new material, a pre-defined recipe can be used to mill multiple trenches in the material with a laser power setting ranging from 10 to 100%. Simply load the pre-existing file PCP_step1_laserpower.VLF into the LaserMill software and process it on a representative spot on the sample. The user still has to modify the recipe slightly to account for the height of the used sample. Note the recommended Z-offset in the LaserMill software and enter that value as Z-offset in the laser parameters in the CAD software. This has to be done for each trench separately, since each trench uses a different laser recipe. After processing, shuttle the sample under the electron beam and observe the resulting structures. Decide on a laser power setting depending on the desired application. As an example, the resulting trenches can be seen in Figure 1 for silicon. Here a laser power below 30 results in very inhomogeneous ablation or very low ablation. For a higher laser power setting, a homogeneous process can be established. Power settings ranging from 40% to 60% could be used for fine trench cutting and sidewall polishing. Settings from 70% to 100% could be used for rough trench cutting to maximize the ablated volume per pulse. After a laser power setting has been found, it is used to set up the overlap alignment in step 2.

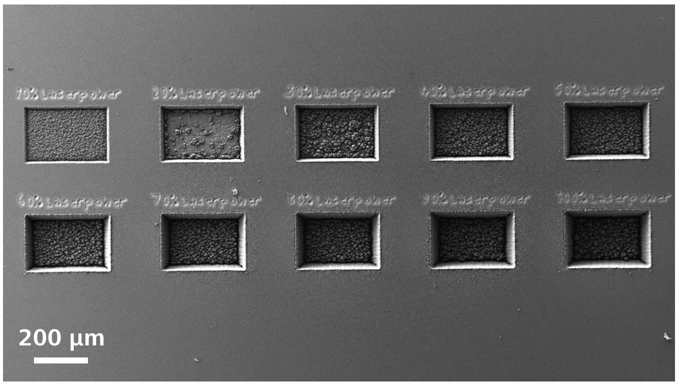

Figure 1: *Step 1 of the Parameter Calibration Procedure. Multiple trenches with different laser power settings are milled to check for effective and homogeneous ablation.*

Step 2 – Pulse Overlap Alignment

The pulse overlap controls the cumulative energy per overscan (hatch) when milling an area. The smaller the pulse overlap, the higher the number of pulses that are used to fill the area. As a result, more energy is available for ablation and more material is removed, resulting in a higher removal rate per hatch. Furthermore, the overall appearance of the trench is also affected when the pulse overlap is altered. By changing the pulse overlap, a smooth trench floor can be created or the sidewall quality increased.

The pulse overlap in line direction is controlled by the pitch between two pulses. A high pitch leads to a low pulse overlap and vice versa. Perpendicular to the line direction, the pulse overlap is controlled by the line spacing of the hatching pattern. As a result, if a symmetrical pulse overlap (and symmetrical pitch) is desired, both the laser parameters and the hatching parameters have to be adjusted to align the pulse overlap in scanning direction as well as perpendicular to it. In Table 2, commonly used pulse overlaps are calculated for the respective pitch values. This regime is mainly defined by the laser spot size of 15 µm and stipulates that the pitch be varied between 0 and 15 µm, so that two adjacent pulses still overlap.

Pitch in µm	Spot overlap in %
1	93.6
2	87.3
3	80.9
4	74.6
5	68.4
6	62.1
7	55.9
8	49.8
9	43.7
10	37.7

Table 2: *Spot overlap and corresponding pitch values for area filling.*

The pitch in line direction can be varied by controlling the ratio of scan speed and pulse repetition frequency. The pitch in µm is given by scan speed in mm/s divided by repetition rate in kHz. In order to vary the pitch between 1 and 10 µm, the pulse repetition rate is kept constant at 100 kHz and the scan speed is varied between 100 mm/s and 1000 mm/s. A predefined recipe (PCP_step2_pitch.VLF) is already pre-installed and can be loaded into LaserMill. Five trenches are predefined there with pitch values of 1 µm, 3 µm, 5 µm, 7 µm, and 10 µm. The user still has to modify the optical Z height like in step 1. This has to be done separately for all five trenches, since different pitch values require different laser recipes. The line spacing in the hatch pattern settings also has to be modified to achieve a symmetric pitch. In addition, for every trench the corresponding laser recipe has to be adjusted to the laser power value selected in step 1. An example of the applied pitch alignment is given in Figure 2 on silicon, where a laser power of 60% was chosen in step 1 to achieve a smooth floor finish. Low pitch values (~ 1 µm) can be used here for very deep trench milling and to achieve good sidewall quality. At a pitch of 3 µm, a good compromise is found between a high removal rate and low processing time for rough milling applications, but the floor quality is not sufficient. In an intermediate pitch regime (5 to 7 µm), a smooth floor surface finish is achieved. At high pitch (~ 10 µm), surface roughness increases again and the removal rate is very low, which limits the use of very high pitch values.

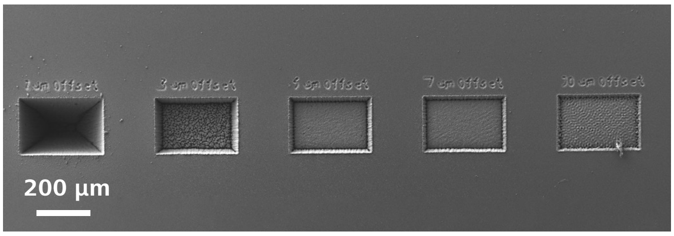

Figure 2: *Step 2 of the Parameter Calibration Procedure. Multiple trenches with different pitch values are milled into the material to check for suitable trench appearance.*

The different pitch settings are suitable for different applications. For a smooth floor finish, a higher pitch from ~ 5 to 7 µm is desired. For polishing applications, a very small pitch of ~ 1 µm can increase the sidewall quality very effectively. A pitch range of about ~ 3 µm is suitable rough trench cutting, since a high removal rate at low pitch has to be traded for lower process time at high pitch.

Step 3 – Pulse Repetition Rate Alignment

Having decided on the pulse overlap only defines the ratio of scanning velocity and pulse repetition rate but does not give a fixed, optimized value for both parameters. To account for that, both parameters are altered at a constant ratio in this third step. High repetition rates and scan speeds can be used for high ablation, whereas low values are used for polishing and further smoothing the trench floor. If both parameters have no significant effect at constant pitch, high values are more desirable since they minimize processing. In Figure 3, the pulse repetition rate alignment is shown on silicon for a laser power of 60% and a pitch of 7 µm derived from the previous alignment steps in order to maximize floor smoothness.

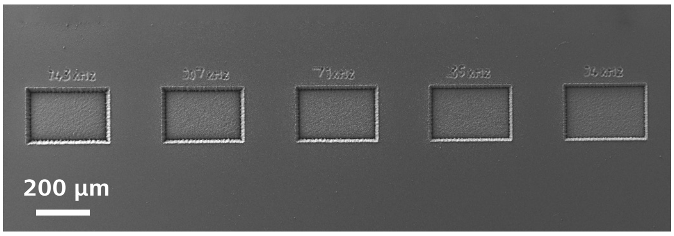

Figure 3: *Step 3 of the Parameter Calibration Procedure. Multiple trenches with different pitch values are milled into the material to check for suitable trench appearance.*

In Figure 3, the scan speed of the laser is varied left to right from 1000 mm/s to 100 mm/s, similar to step 2. Contrary to step 2, the pulse repetition rate is changed, as well, to achieve a constant pitch of 7 µm for each trench. The resulting frequency value in kHz can be calculated by dividing the scan speed in mm/s by the pitch in µm. This yields repetition rates from 143 kHz to 14.3 kHz for Figure 3. The higher frequency of the laser results in a higher depth of the trench. The overall appearance of the trenches does not vary significantly, and the higher frequency can be used to reduce the processing time as much as possible. For the chosen trench, determine the depth of the trench and divide the recorded depth by the number of hatches executed. This value will be used as the "removal rate per layer" and has to be entered in the hatch parameters. When step 3 is finished, the milling parameters have been successfully set up and can be applied. The optimized sets of parameters should be independent of the size of the processed area and can be used on a larger area right away. If a specific depth of the milled laser trench needs to be achieved, the optional depth alignment step can be executed.

Optional: Step 4 – Depth Alignment

To reliably mill trenches with a pre-defined depth, an additional depth alignment procedure can be carried out. Multiple trenches are milled into the material using the same laser and hatch parameters developed at steps 1 to 3. The trenches only feature different numbers of hatches, which results in a higher trench depth for a higher number of hatches. By loading the pre-defined recipe PCP_step4_depth.VLF, eight trenches ranging from 25 to 200 hatches are milled into the material. The user has to change the laser parameters (laser power, scan speed, frequency) and the hatch parameters (line spacing and removal rate per layer) to the values resulting from steps 1 to 3. In Figure 4, the depth alignment is shown on silicon for a fine milling application.

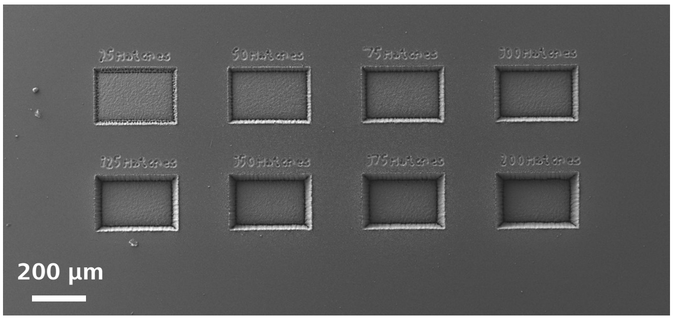

Figure 4: *Optional step 4 of the Parameter Calibration Procedure. Multiple trenches with different numbers of hatches are milled into the material to achieve different depths using the same set of milling parameters.*

The depth of each trench is determined and noted with the respective number of hatchings. These values, as well as the "removal rate per layer", are the variables of the depth alignment method, which is executed in a python script. The result from the calculation will give the user a precise number of hatches that is required to achieve a desired depth. Note that the range of this depth alignment is dependent on the maximum depth of the inputs.

Milling Parameters for Silicon, Copper and Steel

The resulting milling parameters from parameter optimization on silicon, copper, and steel are summarized in Table 3 where sets of parameters are given for rough cut, fine cut, and polishing applications. The sets of parameters are pre-installed in three recipes and can be used right away: Silicon.VLF, Steel.VLF and Copper.VLF.

Note: Please be advised that a specific steel/copper alloy was used for the parameter optimization. The shown parameters should not be considered an optimized set of parameters for any steel/copper alloy but may be referred to as a starting point for further adjustment.

Material	Application	Laser power in %	Scan speed in mm/s	Frequency in kHz	Pitch in µm	Line spacing in µm	Removal rate per layer in µm	Hatching strategy
Silicon	Fine cut	50	107	107	7	7	1	Rotating
	Rough cut	80	300	300	3	3	5	Rotating
	Polishing	30	100	100	1	1	0	Approaching
Copper	Fine cut	80	500	100	5	5	1	Rotating
	Rough cut	100	900	450	2	2	2	Rotating
	Polishing	60	150	150	1	1	0	Approaching
Steel	Fine cut	70	250	50	5	5	1	Rotating
	Rough cut	100	900	300	3	3	5	Rotating
	Polishing	50	125	125	1	1	0	Approaching

Table 3: *Sets of milling parameters on silicon, copper, and steel for different applications.*

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