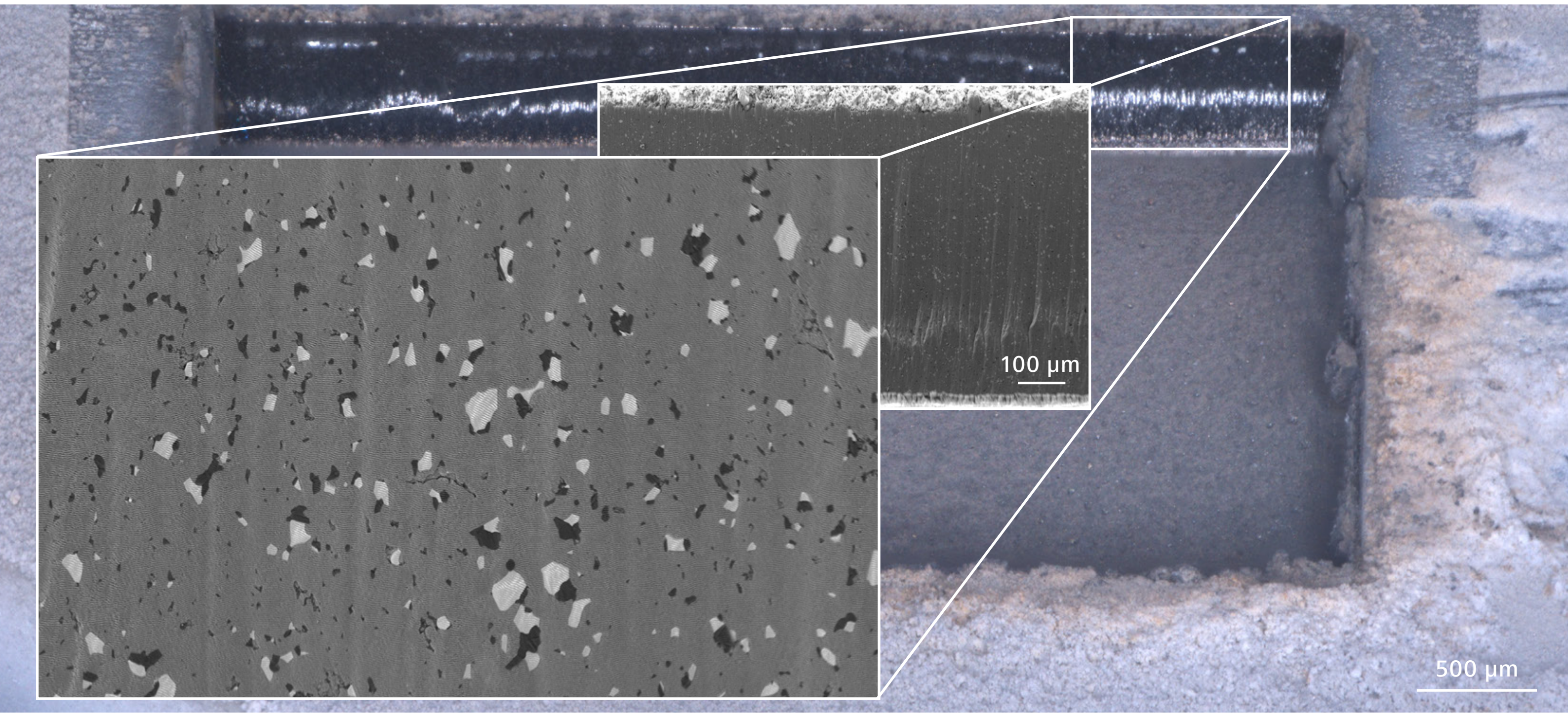
Application Note

# Ceramics Pre-preparation for FIB-investigation Using ZEISS Crossbeam laser




Seeing beyond

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Date: February 2021

**The introduction of the LaserFIB, a femtosecond laser on ZEISS Crossbeam has shown great advantages in large area sample preparation for FIB/SEM analyses with minimal to no heat affected zone in the sample material. Recent application examples covered metal and alloy samples as well as microelectronic components with the task to access deeply buried features and to cross-section larger areas for statistically significant analyses. The presented work will outline that the femtosecond laser is also suited for preparation and investigation of various ceramic materials. On the example of technical ceramics including Zirconia, Silicon Nitride, Forsterite, PZT and a SiC-$ZrB_2$ compound, the determination of depth milling rates with up to 28 µm/s, large area milling of around 10 $mm^2$ and the final surface quality revealing the true microstructure are shown. Additionally, a basic recipe to prepare technical ceramics with ZEISS Crossbeam laser is given.**

**Introduction**

Focused ion beam equipped scanning electron microscopes (FIB-SEM) are widely used in the field of materials science as well as quality control. Not only as a highly sophisticated tool for TEM-sample preparation but also as a versatile instrument for materials characterization. A proven benefit of FIB preparation is location-specific cross-sectioning while maintaining a virtually deformation-free microstructure. With typical FIB beam diameters ranging from about 5 nm to several micrometers, these systems are primarily used to section regions of a few tens of microns. It is still possible to prepare larger cross-sections that extend to several hundred micrometers in length and depth, but this has shown to be a time intensive method.

To bridge the gap between small-scale FIB preparation and large-scale mechanical sample preparation by grinding and polishing, and to avoid issues with altered microstructure caused by large heat affected zones when using nanosecond or even continuous wave lasers, ZEISS Crossbeam has been equipped with a femtosecond (fs) laser system. The use of a femtosecond laser facilitates extremely fast sample machining and surface preparation of deeply buried features as well as large areas. Additionally, due to the pseudo athermal ablation process, the surface quality often reveals the true microstructure directly on the laser cut cross-section without further (FIB) polishing.


***Figure 1*** *ZEISS Crossbeam equipped with a femtosecond laser.*

As shown in figure 1 the fs-laser is installed on a separate airlock-chamber and is therefore separated from the SEM main chamber which, with respect to ablating large quantities of different materials, provides huge advantages regarding cleanliness of the chamber and maintenance intervals.

The fs-laser has already been tested on metals and alloys revealing the microstructure and showing that even EBSD measurements are possible directly on the laser cut [1].For e.g. microelectronic components it allows to access deeply buried features for further FIB polishing and analyses [2]. The present work extends the material range addressable by the fs-laser to various technical ceramics, gives milling rates and provides a kind of universal recipe as guideline for machining other ceramics.

## Experimental Setup, Results and Discussion

Laser milling experiments were conducted on five different technical ceramics to cover a wide range of materials. Flat samples of Zirconia ($ZrO_2$), Silicon Nitride ($Si_3N_4$), Forsterite ($Mg_2[SiO_4]$), Lead Zirconate Titanate ($Pb[Zr_xTi_{1-x}]O_3$ $(0 \leq x \leq 1)$) and a compound of SiC-$ZrB_2$ were used, all featuring ground surfaces.

When approaching a new sample material, the first step is to find suitable process parameters for laser milling with as little surface damage as possible. Parameters that can be adjusted and have the most influence on milling rate and surface quality have shown to be laser power [%], scan speed [mm/s] and pulse frequency [kHz]. Further parameters are hatch distance [µm] and hatch rotation [°] (Fig. 2). Extensive previous tests on different material types have shown that a hatch distance of 4 µm and a hatch rotation of 19° per hatch, as well as a frequency/ scanspeed combination leading to ca. 50% pulse overlap (Fig. 3) result in the highest milling rates. The best sidewall surface quality however can be achieved with no hatch rotation and the laser tracks parallel to the cross-section face. Frequency, scanspeed and power have to be fine-tuned for each material.

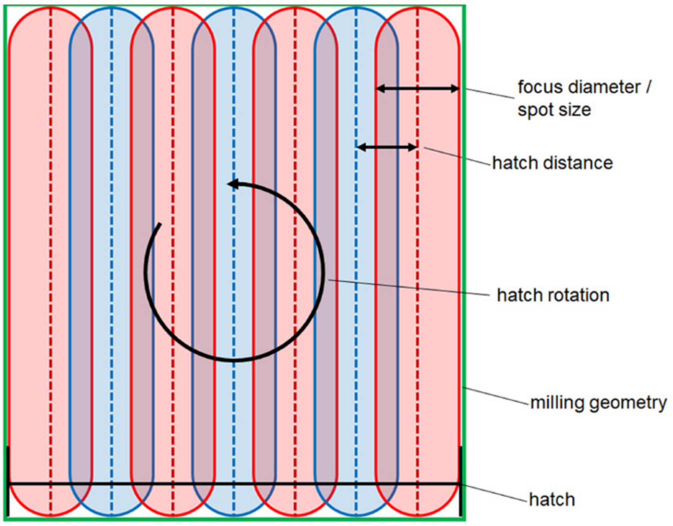


***Figure 2*** *Adjustable hatch parameters in the laser milling software.*

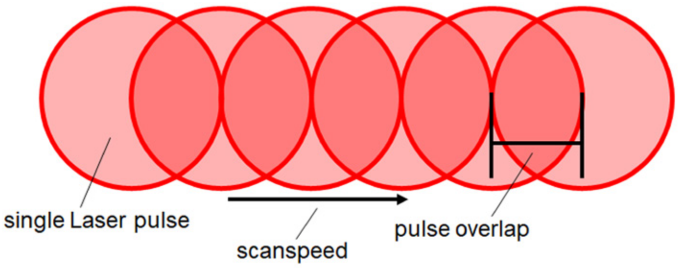


***Figure 3*** *Parameter combination of frequency and scanspeed leading to a specific pulse overlap, best milling rates with ca. 50% overlap.*

To estimate expected milling times for different tasks on different ceramic materials it is crucial to know the milling rate for specific parameter combinations. For testing the five ceramic types presented in this work, power, hatch distance and hatch rotation were kept constant, while the hatch number was set to 50 repetitions. This lead to a decent milling depth and thus reliable measurements. The frequency was varied from 10 – 100 kHz in 10 kHz increments. Scanspeed was changed accordingly to achieve 50% pulse overlap. All parameters are shown in Table 1.

| | |
|---|---|
| **Power [%]** | 100 |
| **Focus diameter [µm]** | 12 |
| **Hatch distance [µm]** | 4 |
| **Hatch number** | 50 |
| **Hatch rotation [°]** | 19 |

| Test No. | Frequency [kHz] | Scanspeed [mm/s] | Milling time [s] |
|---|---|---|---|
| **1** | 10 | 50 | 65.0 |
| **2** | 20 | 100 | 34.0 |
| **3** | 30 | 150 | 23.0 |
| **4** | 40 | 200 | 18.0 |
| **5** | 50 | 250 | 15.4 |
| **6** | 60 | 300 | 13.4 |
| **7** | 70 | 350 | 11.8 |
| **8** | 80 | 400 | 10.7 |
| **9** | 90 | 450 | 9.7 |
| **10** | 100 | 500 | 8.1 |

***Table 1*** *Chosen laser parameter set for initial milling rate tests. Laser pulse frequency and scanspeed were varied in ten iterations.*

For each parameter set a 500 × 500 µm square was milled into each of the samples and the milling time as well as the resulting milling depth was recorded. Resulting milling times for each parameter set are shown in Table 1, too. Figure 4 exemplarily shows the milled trenches in the Silicon Nitride sample. All trenches showed almost the same depth and good sidewall quality. From the measured milling depth and the hatch number the milling rate in µm/hatch can be calculated, which is another important parameter when milling large volumes. The µm/hatch value is used to change the laser focus after each hatch, which results in steeper sidewalls.

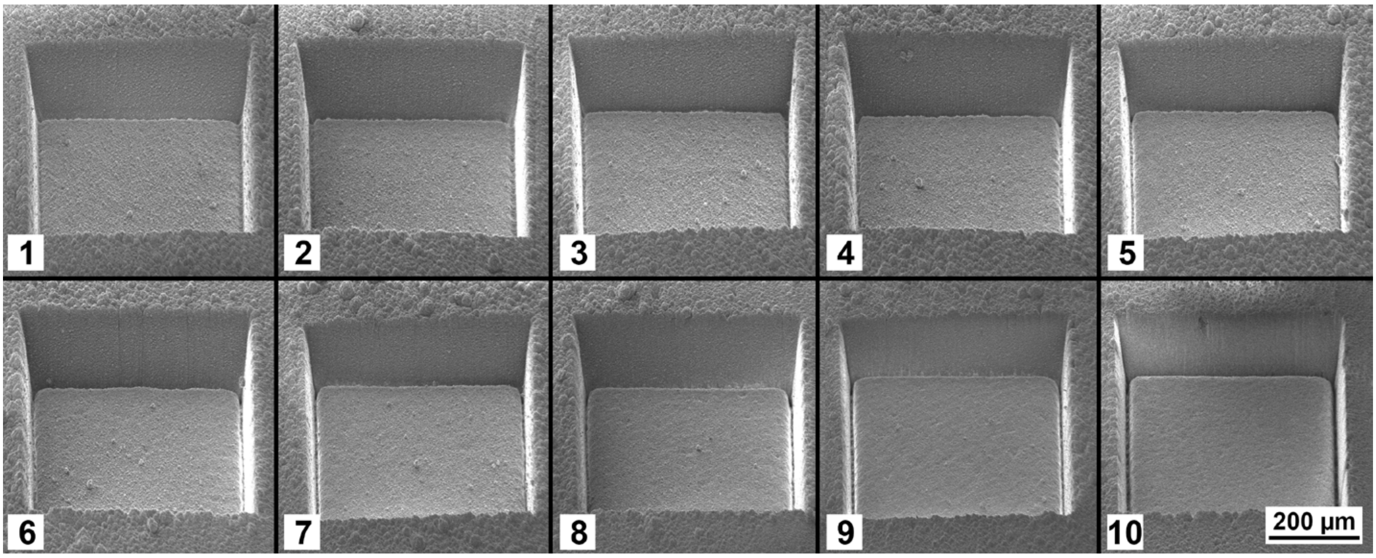


*Figure 4* *Silicon Nitride, milled trenches to determine milling rates, homogeneous depth, good sidewall quality; SEM, SE images.*

To get an idea about what milling times can be expected when milling to a certain depth, the depth milling rate in µm/s is calculated from the measured trench depth and the time needed for each individual milling object. By multiplying with the area of the milled shapes, a milling rate in mm³/h can be calculated. Figure 5 shows the determined milling rates for all five ceramics and for different parameter sets. With 100 kHz frequency and 500 mm/s scanspeed for example, milling rates up to 26 mm³/h can be achieved for Forsterite. The SiC-$ZrB_2$ compound, however, is milled with 13 mm³/h only using identical parameters. These results indicate a significant influence of the material on the milling rate. It can be expected that hard ceramics like carbide compounds take significantly longer to mill than softer materials like Forsterite.

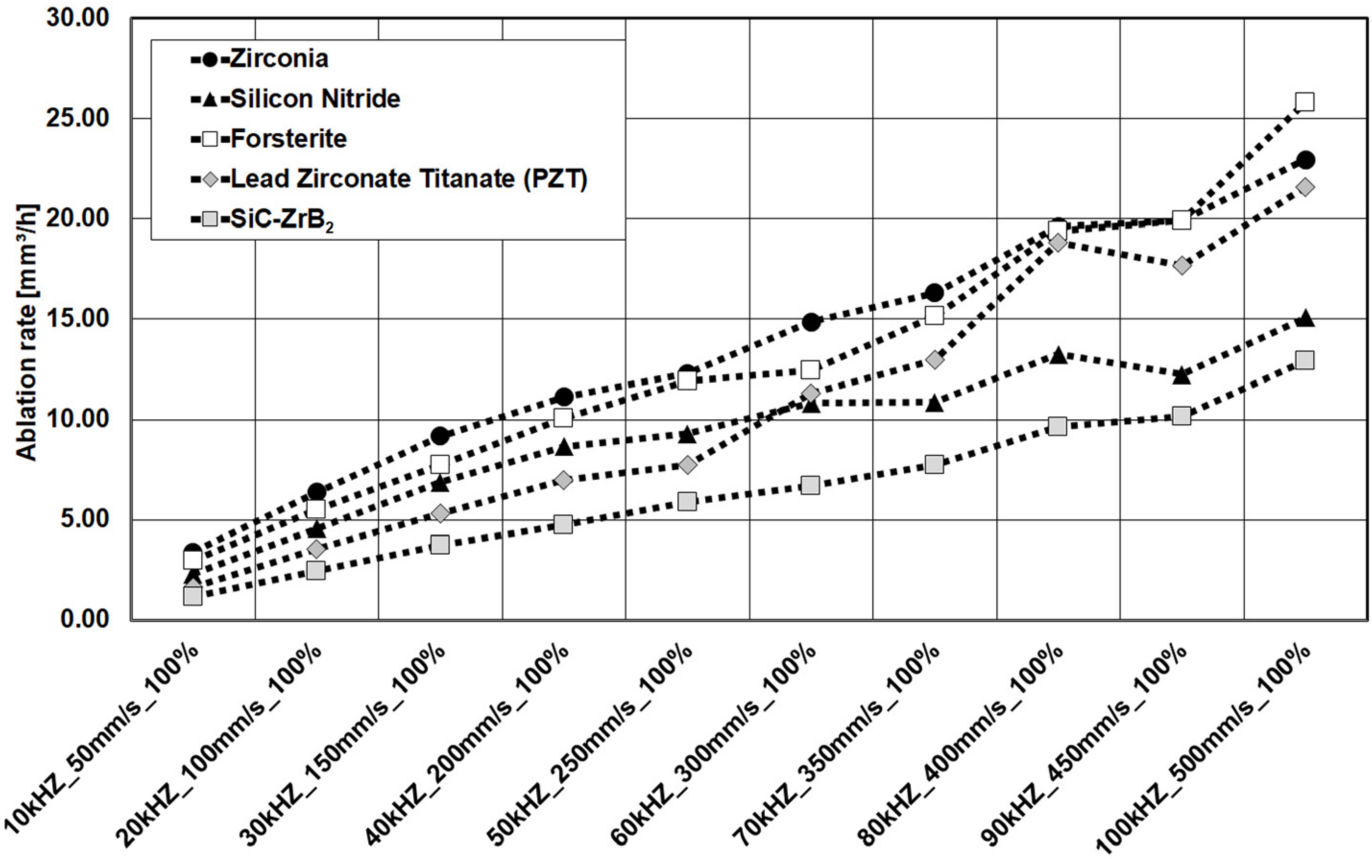


*Figure 5* *Milling rates for different ceramics and different laser parameter sets.*

High milling rates (Fig. 5) and a good sidewall and milling surface quality (Fig. 4) for the parameter set 100 kHz and 500 mm/s make this set the preferred one as a standard recipe for technical ceramics and a good starting point for individual recipe development. Table 2 lists the parameter details of the developed recipe as well as the mean milling rates i.e. refocus values for each of the tested ceramics.

| Parameter set | | Refocus value [µm/hatch] | |
|---|---|---|---|
| **Power [%]** | 100 | Zirconia | 4.5 |
| **Hatch distance [µm]** | 4 | Silicon Nitride | 3.2 |
| **Hatch rotation [°]** | 19 | Forsterite | 4.2 |
| **Frequency [kHz]** | 100 | Lead Zirconate Titanate | 3.2 |
| **Scanspeed [mm/s]** | 500 | $SiC\text{-}ZrB_2$ | 2.0 |

***Table 2*** *Standard preparation recipe and refocus values for specific ceramics.*

These values were used to mill specific geometries in each ceramic sample to either access deeply buried features or reveal the microstructure on a larger scale than possible with FIB preparation alone. To ensure that potential redeposition of ablated material does not affect the prepared surface, a U-shaped pattern is used to create a free-standing final cross-section face of interest. Figure 6 shows the preparation results for Zirconia, Silicon Nitride, Forsterite and PZT. All samples show a smooth surface and steep sidewalls, ideal for further preparation and investigation. To also reveal the microstructure of laser milled samples, a fine polishing laser step should be applied.

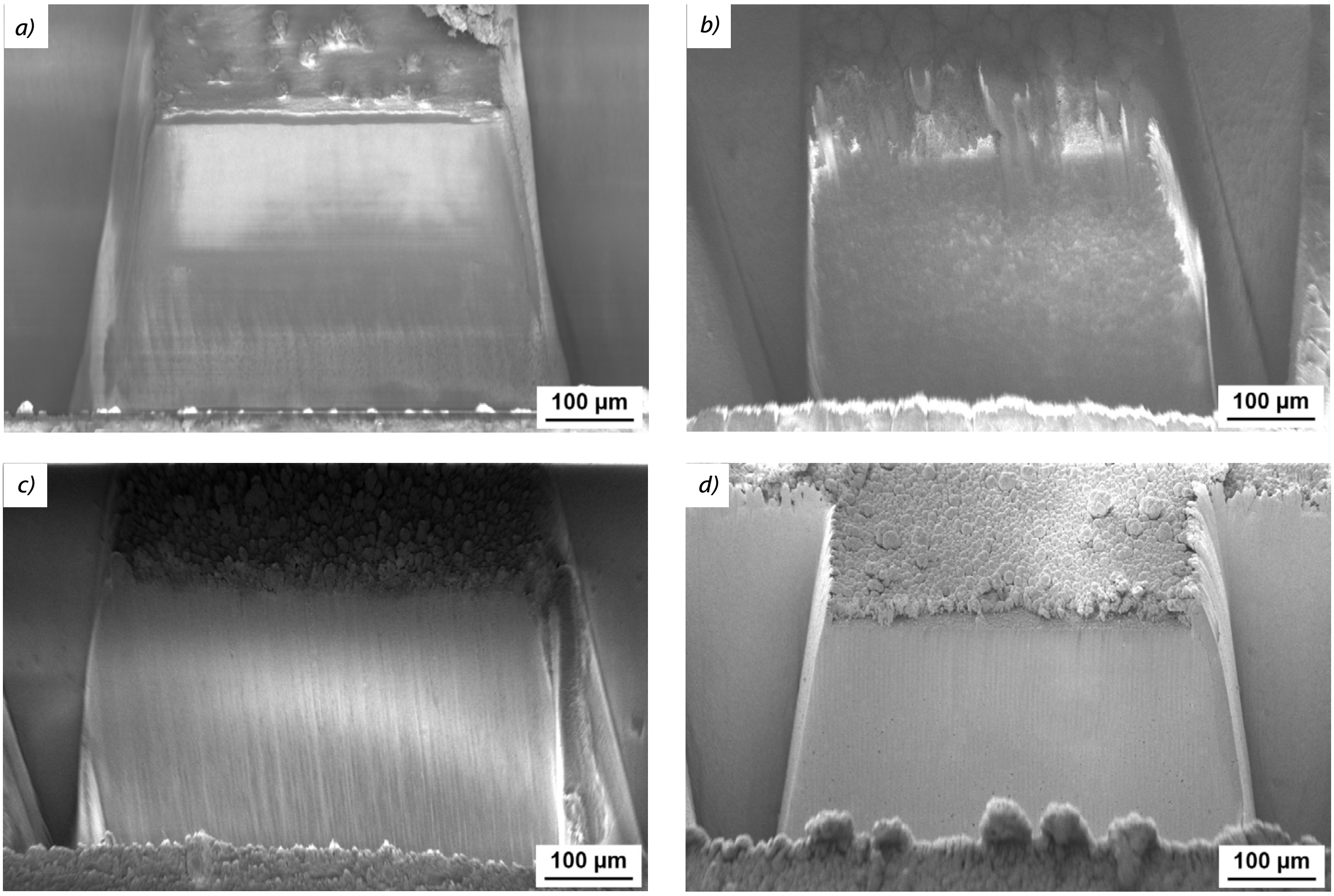


***Figure 6*** *Milling and surface results for a) Zirconia, b) Silicon Nitride, c) Forsterite and d) PZT after rough milling with 100 kHz and 500 mm/s, smooth surface quality for all samples; SEM, SE images.*

The full potential of the LaserFIB however can be unlocked when large areas or large volumes are ablated. To illustrate this capability, a 5 × 2 mm trench with a depth of ca. 700 µm was milled in the SiC-$ZrB_2$ compound sample.

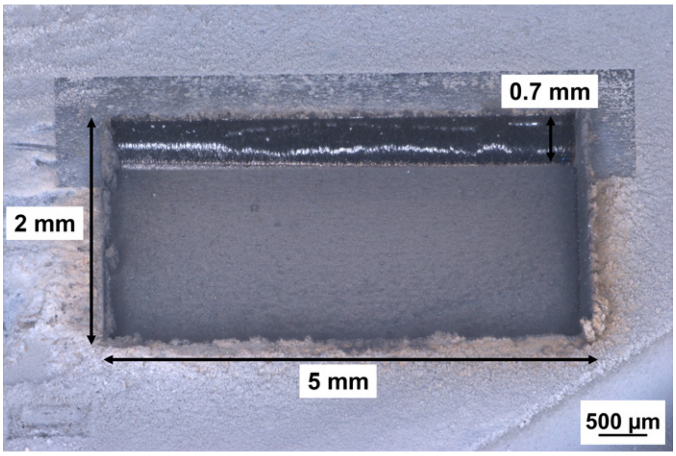


***Figure 7*** *Light microscopic view of large laser milled trench in SiC-$ZrB_2$ ceramic, 5 × 2 × 0.7 mm*

Figure 7 shows a light microscopic image of the milled trench. Rough milling of 5 × 2 mm was carried out with the standard recipe shown in Table 2 and 400 hatches in total. In addition, a 4.8 × 0.2 mm fine polishing step with 20 kHz and 120 mm/s and no hatch rotation was done at the cross-section face. The total milling took around 80 minutes and lead to a depth of 700 µm.

Figure 8 shows the resulting surface in high magnification. Besides the residual LIPSS (laser induced periodic surface structures), which currently cannot be avoided when applying femtosecond laser pulses to a sample, the individual phases of the ceramic compound can be seen and clearly distinguished from each other. The bright grains in Fig. 8 represent the $ZrB_2$ phase surrounded by the gray SiC matrix. The black phase represents pure Si. In such a state, the laser milled surface is suited for further investigation directly on the laser cut or for specifying regions of interest for FIB post-polishing.

**Conclusion**

The presented work shows that ZEISS Crossbeam laser is a very suitable tool to rapidly prepare large cross-sections in different ceramics without major defects or heat dissipation into the cross-section surface. The investigated technical ceramics cover a wide range of state-of-the-art ceramic materials and thus it can be concluded that the fs-laser is capable of preparing all sorts of ceramics in an easy, quick and reproducible way. It could furthermore be shown that for ceramics a universal recipe for rough milling is applicable and parameter tuning is only needed for fine polishing and revealing the true microstructure. With this work a basis for accessing deeply buried features for further FIB polishing and investigation in ceramic components is created.

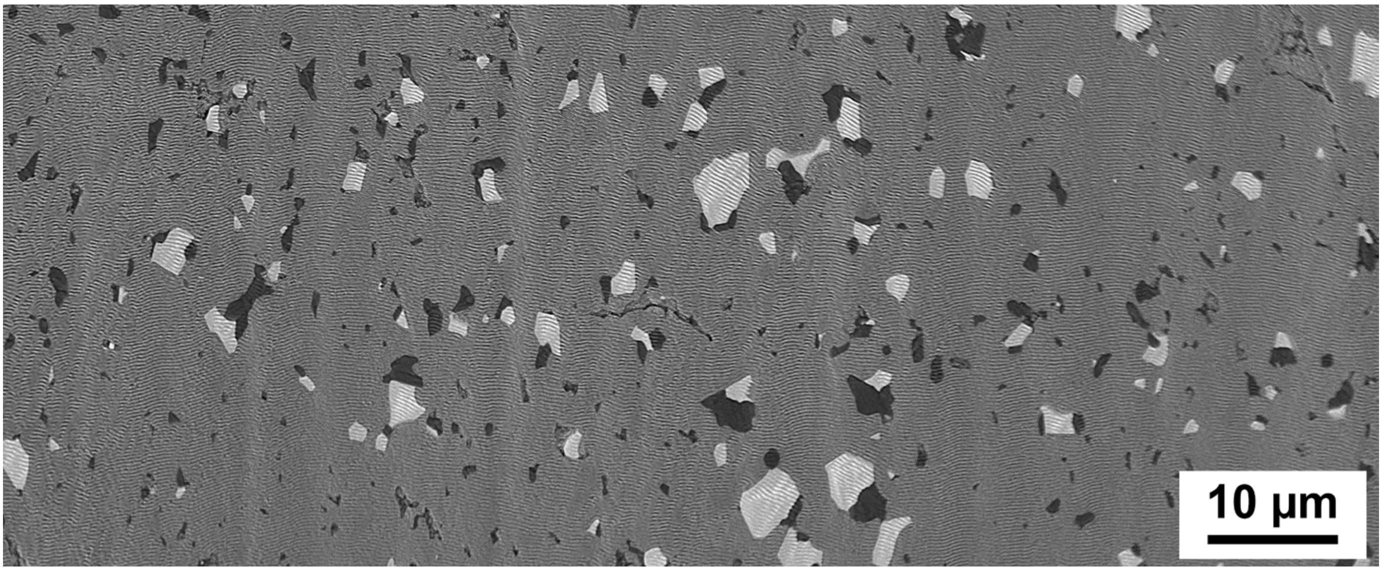


***Figure 8*** *Surface quality of laser milled SiC-$ZrB_2$ ceramic, LIPSS clearly visible; SEM, Inlens-SE image.*

**References:**

[1] T. Schubert et al., Rapid Sample Preparation for EBSD-Analysis Enabled by the LaserFIB, ZEISS Application Note (2020), available online. https://zeiss.widen.net/s/xxwptzwnl6/en_wp_crossbeam_ebsd_laserfib

[2] Tordoff, B., Hartfield, C., Holwell, A.J. et al. The LaserFIB: new application opportunities combining a high-performance FIB-SEM with femtosecond laser processing in an integrated second chamber. Appl. Microsc. 50, 24 (2020). https://doi.org/10.1186/s42649-020-00044-5

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