Femtosecond Laser Processing of Membranes for Sensor Devices on different Bulk Materials

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Abstract. *We demonstrate that diaphragms for sensor applications can be fabricated by laser ablation in a variety of substrates such as ceramics, glass, sapphire or SiC. However, ablation can cause pinholes in membranes made of SiC, Si and metals. Our experiments indicate that pinhole defects in the ablated membranes are affected by ripple structures related to the polarization of the laser. From our simulation results on light propagation in Laser-Induced Periodic Surface Structures (LIPSS) we find out that they are acting as a slot waveguide in SiC material. The results further show that field intensity is enhanced inside LIPSS and spreads out at surface distortions promoting the formation of pinholes. The membrane corner area is most vulnerable for pinhole formation. Pinholes funnel laser radiation into the bulk material causing structural damage and stress in the membrane. We show that a polarization flipping technique inhibits the formation of pin holes caused by LIPSS.*

Keywords

AlGaN/GaN HEMT, diaphragms, laser ablation, LIPSS, SiC MEMS, slot waveguide.

1. Introduction

Membranes are widely used as a structural unit for various sensor applications, MEMS, micro devices used in biology, medicine or life science [1] and [2]. Reactive Ion Etching (RIE) is an established and precise technology to produce them in materials (mainly Silicon) commonly used in micro technology. Only a few results were published in the field of bulk micromachining of wide band gap materials to fabricate 3D micromechanical structures. The widely used approaches employed laser tools working mostly with pulses in ns range, optionally in fs pulse duration [3], [4] and [5]. We showed that femtosecond laser ablation is a promising technology to fabricate micromechanical membrane structures in SiC, borosilicate glass, sapphire, Al_2O_3 and zirconium ceramic substrates [6] and [7]. Our main focus was on SiC, which can be machined by laser ablation faster than by RIE. On a 350 μm thick 4H-SiC substrate, we achieved by laser ablation an array of 275 μm deep and 1000 μm to 3000 μm in diameter blind holes without damaging the 2 μm GaN/AlGaN heterostructure layer grown on its opposite side. Recently we investigated a combination of ablation and RIE to produce thinner membranes without back side damage [6]. It is possible to use a substrate pre structured by ablation in a maskless RIE procedure when membrane fabrication and substrate thinning is intended.

The materials we tested (including diamond) revealed Laser-Induced Periodic Surface Structures (LIPSS) similar as investigated in [8]. On the other hand, we observed pinhole defects only in metals or high refractive index SiC and Si [7]. Comparing experimental and simulation results, we found that LIPSS are acting as slot waveguides in high refractive materials and can trigger the formation of pinholes. In the glass the formation of crater structures and spikes was explained previously by a different electronic damage mechanism due to nonlinear absorption of multiple incident ultrashort laser pulses [9]. The generation and orientation of LIPSS in different materials during fs-laser ablation using linear polarized light were extensively studied in [8]. Moreover, in materials we investigated, LIPSS were created perpendicular to the direction of the laser polarization. Such an angular orientated configuration is most suitable for a good performance of slot waveguides [10]. Consequently, steady rotation or frequent angular flipping of the polarization is an effective measure firstly to avoid a pronounced formation of LIPSS and secondly to reduce the performance of existing LIPSS in their waveguide function. The slot waveguide approach does not explain the formation of LIPSS and small pinholes some hundreds of nanometer in diameter. However, our qualitative simulation results in [10] pointed towards an existing feedback mechanism promoting the growth from nanometer size to typically 2 to 5 micrometer by interconnecting with adjacent bores.

In this paper, we report on consequences for the diaphragm surface texture caused by the structural stress induced from the radiation funnelling effects of the pinholes in the corner zone of the structures. We give a brief possible reason why the formation of High Spatial Frequency LIPSS (HSFL) is often observed at the edges of pinholes and scratches. In a comparison of two different substrate materials, we demonstrate the relevance of back reflections from a thin membrane for the ablation quality limits.

2. Experiments

For laser ablation, we used a Spectra Physics SPIRIT delivering 350 fs pulses at 200 kHz with an average power output of 4000 mW at 1040 nm and 1600 mW at 520 nm. This laser is part of the laser work station microSTRUCTvario from 3D-MICROMAC and the used 100 mm focal length scanner optics provides a focal spot diameter of about 13 µm, and 25 µm at 520 nm and 1040 nm wavelength, respectively. In all experiments we used 1000 mm·s^{-1} scan speed and 5 µm hatch distance between lines in the scan mesh.

To evaluate the influence of pinholes and structural stress on the surface texture and membrane quality the scan pattern was rotated by 15° increments between consecutive scans at a constant polarization direction parallel to the x-scan axis. Alternatively, the specimen was rotated. Rotating the sample automatically changes the polarization and the spatial orientation of the scan-pattern relative to the sample continuously. For practical use, the specimen rotation is only suitable for circular structures but not feasible at a general design pattern. Therefore, we conducted experiments with 90° flipping of the polarization after several consecutive scans by frequently inserting and removing a half-wave plate into the beam path of the laser setup (Fig. 1). This method preserves a constant spatial scan-pattern on the substrate, only the polarization direction is altered in 90° increments. We compared the three methods with our standard ablation procedure which is xy-scanning at constant special scan-pattern orientation and constant polarization direction. This offered a possibility to distinguish whether the scan pattern rotation or the polarization change is the dominant parameter for a proper surface quality of the obtained diaphragm.

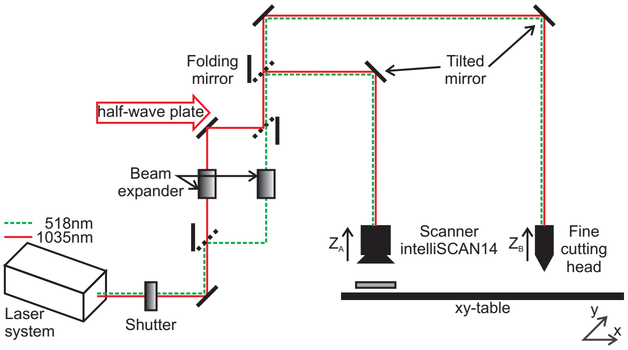

Fig. 1: Schematic of laser setup, to supress pinhole formation in the membrane a half-wave plate was used to flip the polarization 90° after several consecutive scans.

3. Results and Discussion

3.1. HSFL Formation at Pinhole Edges

In SiC we often observed HSFL structures at the edges of pinholes, deep scratches and slopes. In such locations LIPSS show the tendency to split into structures with a periodicity of only a fraction of the used laser wavelength. Figure 2 depicts the edge of a pinhole in SiC. The horizontal area of the membrane shows LIPSS

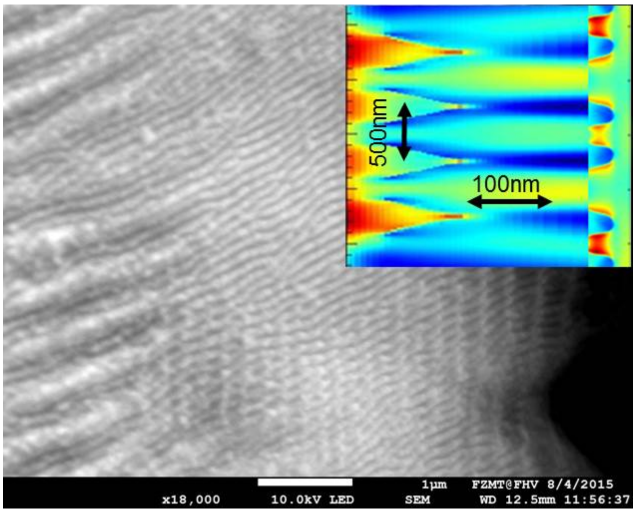

Fig. 2: At the edges of bores, pinholes or scratches we observed often a transition from LIPSS into HSFL. The insert shows how the light field below the ridges of the LIPSS can couple to the groves of HSFL structures. The splitting is initiated at the edge and continues down the wall of the pinhole.

separated in about 500 nm increments. The transition from LIPSS to HSFL is visible at the intersection of the horizontal bottom of the membrane and the slope section of the pinhole.

The calculated intensity distribution in LIPSS with 500 nm periodicity can be seen on the left side of the insert in Fig. 2. Calculating the light distribution of a periodic structure at 250 nm spatial increments on the right side of the insert revealed that the zones with higher radiation match exactly with the light distribution of higher intensity from the 500 nm LIPSS inside the bulk material. We assumed for all simulations a horizontal plane and a vertically orientated laser beam for the ablation. The calculated distribution of the laser intensity deeper inside the bulk material cannot interfere with the radiation inside the HSFL as long as both are in the same plane. However, apparently there is a chance to get sufficient intensity overlap at the edge of a pinhole or laser structured bore (Fig. 3 and Fig. 3 upper right insert, respectively). In this locally confined area the inner bulk radiation field from the LIPSS surfaces at the side wall where it can positively interfere with the light distribution of the HSFL structure. The small radius of a contour or pinhole edge and the high refractive index of SiC make such an edge to a lens of very short focal length concentrating laser light intensity sufficient high for the LIPSS to HSFL transition. We visualized this concentration effect in the lower right insert of Fig. 3. The lens effect of the pinhole edge causes a permanent change of the refractive index in an acrylic sample causing intensity related colour changes when observed between polarizers. In addition, the sidewall of a bore or pinhole is not perpendicular due to the Gaussian intensity distribution of a laser beam. Light refraction at the sloped sidewall directs further laser radiation into the bulk material as can be seen in the upper right insert of Fig. 3. The intensity of the light entering the bulk via the side wall was at least intense enough to create darker colour centres in a glass substrate, which we used to visualize leaking radiation from the ablation zone.

We observed that the pinhole formation starts predominately at scratches and corners. One reason can be seen in already mentioned focusing effects of sharp edges, a second in the spatial spreading of laser intensity at distortions and interruptions of slot waveguides. For a smoothly aligned LIPSS array the laser radiation, which hits the ablation surface perpendicularly, is well confined inside the slots (simulation results in upper insert Fig. 4). In the area between them where ridges from the already formed LIPSS are located, the laser radiation is penetrating deep into the substrate material. If such a waveguide arrangement is interrupted by a scratch like in Fig. 4 the confinement of the laser radiation is broken as the calculated intensity distribution depicts in the lower insert of Fig. 4. Neighbouring nanometer sized bores are now in conditions to join and grow towards pinholes with several micrometers in diameter (Fig. 3 and Fig. 4). Such pinholes funnel the laser light directly into the membrane and create extremely high intensities at the edges and tips causing excessive thermal load and structural stress (visualized at the tip of the pinhole by polarizing microscopy in Fig. 3 lower left insert).

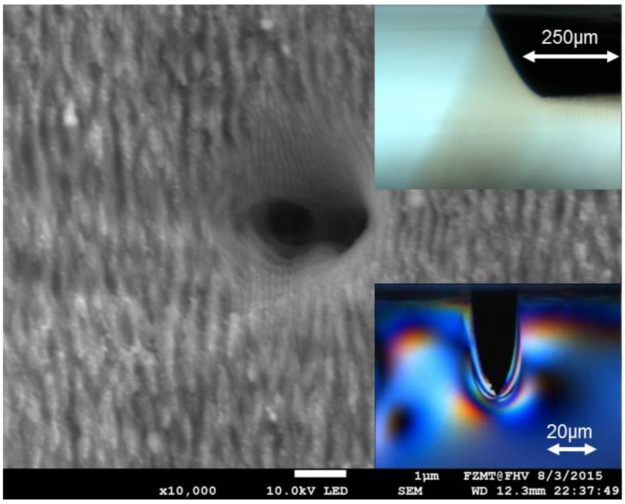

Fig. 3: LIPSS in the horizontal plane of a SiC membrane alter beginning at the edge of the generated pinhole from 500 nm spatial increments to 250 nm over the entire sloped sidewall.

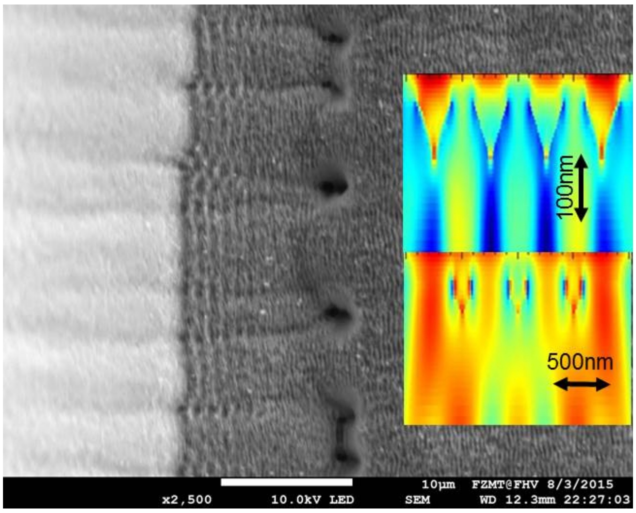

Fig. 4: LIPSS and pinholes are generated in the size of several hundreds of nanometer. Predominately at positions of scratches radiation is no longer well confined inside the waveguide (insert) and adjacent bores can combine to pinholes with several micrometers in diameter.

3.2. Pinhole and Stress Related Surface Structures at the Membrane

Induced stress at the diaphragm is accumulating at the corner area of the bore. The side wall quality is dependent mainly on the scanning method, laser parameter, light polarization and laser delay time. Structural distortions at the side wall due to delay issues in laser on/off timing can result in membrane stress influencing the surface texture of the membrane.

In Fig. 5, a standard xy-scan procedure was used (scan pattern and orientation of the laser polarization spatially fixed for all consecutive scans). The first scan moved in the x-direction from left to right and back from right to left shifting the parallel scan line 5 µm upward in the y-direction until the total surface area was covered by the scans. Next, the same procedure was performed in the y-direction from up and down. At the start and return positions the scanner has to accelerate and slow down. If the delay time is not properly adjusted with respect to the scan speed the first pulse at the start position and the last pulse at the return position will not hit the correct x-coordinates (in the case of an x direction scan). In Fig. 5, this caused a positioning error at the side wall of 30 µm and scratch-like distortions at the side wall prolonging down to the corner between wall and membrane. According to our simulations and experimental observations, LIPSS interrupted by scratches provide good conditions for pinhole growth (Fig. 4). The funnelling of laser power via the pinholes into the substrate causes thermal and mechanical stress locally influencing the ablation ratio. This can generate a stress induced surface structure on the whole membrane surface typical for the specific scan strategy. Figure 5 shows such a wave-like pattern on a SiC membrane produced by an xy-scanning strategy at constant polarization in the x-direction.

Rotating the scan pattern between the consecutive xy-scans by e.g. 15° as depicted in the insert of Fig. 6, changes the side wall damage pattern causing a different membrane stress profile and surface texture. The distortions are now distributed more evenly, fewer scratches and pinholes are generated at the corner of the membrane. Mechanical stress is consequently reduced resulting in a much smoother surface texture with respect to the standard xy-scanning method. However, also in the pattern rotation procedure the polarization direction was constant in the x-direction and we observed pinhole formation all over the membrane when the laser power was increased beyond 470 mW for SiC [10]. Spots of higher stress levels are generated and the membrane quality degenerates predominately at the corner zone where an increased ablation ratio can be observed. In one experiment we rotated the sample with about 30 revolutions per minute while performing a standard xy scanning procedure with constant polarization orientated in the machine's x-direction. By that means as well scanning direction and polarization are permanently changed relative to the ablation area. The laser power was set to such a high level that pinholes were created all over the membrane area when the ablation was made without sample rotation. The results with rotating sample can be seen in Fig. 7. The geometrical surface quality at the side wall, at the critical corner zone and the membrane itself was very smooth. All the pinholes were successfully supressed. Unfortunately, this method is not useful when an array of

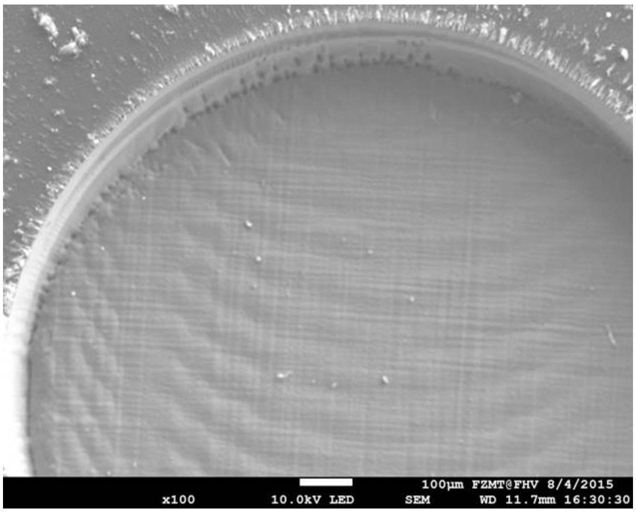

Fig. 5: Wrong delay time setting of the xy-scanner causes poor side wall quality. Scratches at the corner area promote pinhole formation, generates stress and wave patterns at the membrane.

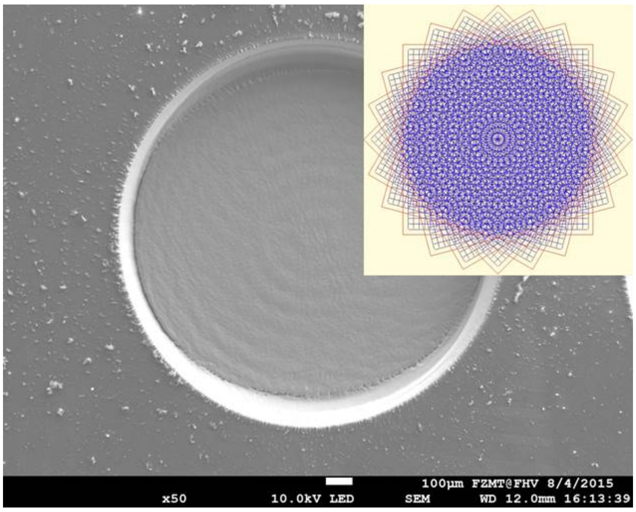

Fig. 6: Structural distortions at the sidewall and in the corner area of the SiC membrane are reduced by 15° rotation of the scan direction between consecutive scans (insert). This causes also a smoother surface pattern at the membrane with respect to a standard xy-scan as shown in Fig. 5.

membranes or contour pattern other than circular shall be produced. We obtained very similar high quality membranes without pinholes and a sufficiently reduced stress related surface texture when we combined the pattern rotation approach with a 90° polarization flip between consecutive scans.

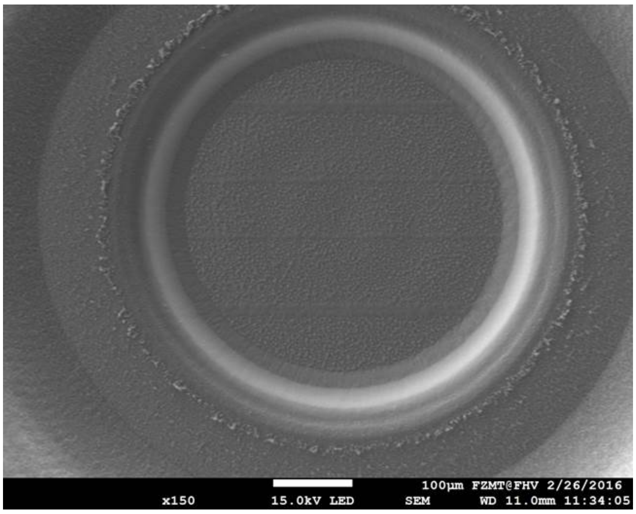

Fig. 7: A rotating sample (30 rpm) generates less scratches, smoother side walls and membranes without pinholes and surface wave structures. Pinhole formation is most effectively supressed by simultaneous rotation of the polarization direction and the smoothening effect by the scan pattern rotation.

At the membrane the formation of pinholes was already suppressed by the 90° flipping of the polarization direction, while keeping the xy-scan pattern orientation unchanged. The most effective single measure to avoid pinhole formation is the flipping of the laser polarization direction between consecutive scans by 90° or the permanent rotation of the sample. This disables the slot waveguide function of the LIPSS and as a consequence interrupts the growth process of pinholes. It is not possible to achieve the same effect by changing the scan direction only as such an action does not suppress the waveguide performance of the LIPSS. But it is possible to smooth the irregularities at the side wall of a structure caused by delay issues of the laser on/off timing.

3.3. Back Reflection Related Damage

In Fig. 7, one can see four shallow lines on the bottom of the bore. This surface modification at the ablation side of the membrane was caused by an existing structure on the back side of the sample. On the SiC sample with an approximately 250 nm thick step structure on the back side of the diaphragm (the laser ablation is made at the front side), we realized that reflection and focusing effects contribute significantly to the quality of ablated membranes. The insert in Fig. 8 depicts this structure on the backside of the sample. The corners and edges of a step are comparable to a lens of very short focal length. Light from back reflections is collected and accumulates in a small area inside the material close to the surface of the thin membrane causing the surface damage along the step edge at the front side (ablation side). This damage is shown enlarged in Fig. 8 and at the complete membrane area in Fig. 7. A significant circular distortion on the sample backside is also visible in the insert of Fig. 8. Its causes are the corresponding focusing and light funneling effect of small pinholes in the corner area of the membrane depicted in Fig. 5 and Fig. 6. We visualized these focusing and laser light funneling effect of pinholes in the lower right insert of Fig. 3 using a transparent polymer sample. During the membrane fabrication, the focused funneling of laser radiation causes a permanent stress and temperature related change of the refractive index in the zone with higher light intensity and we made this visible in a polarizing microscope. This type of damage is a focusing effect and not influenced by the light polarization, but using a fixed polarization ablation procedure, the distortions from focused back reflections become likely an activator for pinhole formation. Figure 8 shows the damage at the front side of the membrane where the regular ablation is performed. During ablation the sample was rotating, changing scanning track and polarization continuously. However, the pattern of the structure on the back side of the 68 µm thick membrane is clearly reproduced at the ablation plane. The direct laser radiation and the back reflected focused portion add up to a locally higher light intensity. Besides damage on the back side, this causes an increased ablation ratio at the front side along the reflection pattern depicted in Fig. 8. Pinhole formation starts at a certain threshold what we demonstrated in [10]. Consequently, pinholes at the membrane will first occur along this back reflection induced distortion pattern. Without flipping or rotation of the laser polarization this problem appears already at thicker membranes or lower laser power. The most vulnerable area for front and backside damage remains the corner zone of the membrane. The highest impact to improve the membrane quality and reduce the damage there was provided by the permanent change of the polarization direction by 90° flipping or constant rotating to suppress the waveguide function of the LIPSS. A better delay time adjustment additionally reduces sharp grooves and obstacles at the side wall of the membrane cavity. Also, other measures like the scan pattern rotation at 15° increments (Fig. 6) or permanent sample rotation are smoothing the side wall and corner of a membrane. All measures in combination gave the best result depicted in Fig. 7 but the main contribution was clearly by the elimination of the waveguide function of the LIPSS. In SiC we are

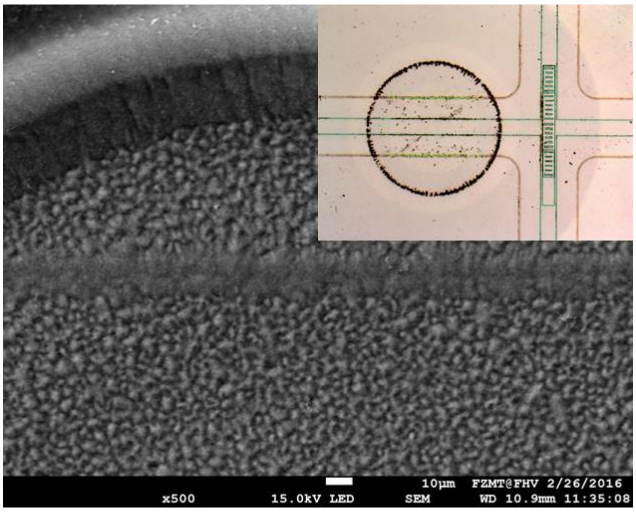

Fig. 8: A 250 nm step structure is the cause of damage at the back side and the front side of the membrane. Focused laser back reflections are too intense to be neglected.

currently able to produce diaphragms in the range of 70 µm to 80 µm using 90° polarization flipping after consecutive scans. At constant polarization direction, the limits are between 100 µm and 150 µm. Substrate purity and structural quality additionally influence the threshold for pinhole formation and consequently the minimum thickness achievable for a diaphragm. As soon as micrometer sized pinholes are formed the focused laser radiation funneling process into the substrate starts and within a few consecutive scans, the membrane is destroyed. In lower refractive index material (glass, sapphire, diamond) we observed LIPSS too, but there was no pinhole formation like in SiC or Si. We could produce much thinner diaphragms than in SiC, the thinnest one of 14 µm thickness was obtained in glass without polarization flipping. This is a further indication that the waveguide function is the main cause for damage, in low refractive index material the performance of slot waveguides is reduced.

In the attempt to produce a thin membrane in a 100 µm borosilicate glass sheet, we could maintain a constant ablation ratio of precisely 1.5 µm per scan without readjusting the focus position. The focus was set at the beginning of the structuring process onto the front surface (entrance side of the laser beam) of the sample. However, as soon as we obtained a diaphragm thickness of 12 µm the laser caused damage on the glass surface at the backside of the diaphragm (Fig. 9). In the very next scan the membranes cracked and disintegrated. One scan before glass particles started chipping off the surface the approximately 14 µm thick membrane was totally intact (insert of Fig. 9). Sharp edges of the chipped spots in combination with back reflections generate focusing effects of the same type as described for SiC and contribute to a higher ablation speed. In addition, the absorption of the laser light is increased at the now rough backside and in fact, front and back side of the diaphragm is located at the same distance with respect to the focal plane (88 µm and 100 µm respectively) leading to a double side ablation. Mechanical stress generated by the laser ablation, focused radiation back reflection and double side ablation destroyed the membrane within one scan. The slot waveguide issue is not relevant in glass and polarization flipping has a lower impact on the membrane quality (only radiation losses via the side wall of the bore depicted in the upper insert of Fig. 3 are influenced by the orientation of the laser polarization).

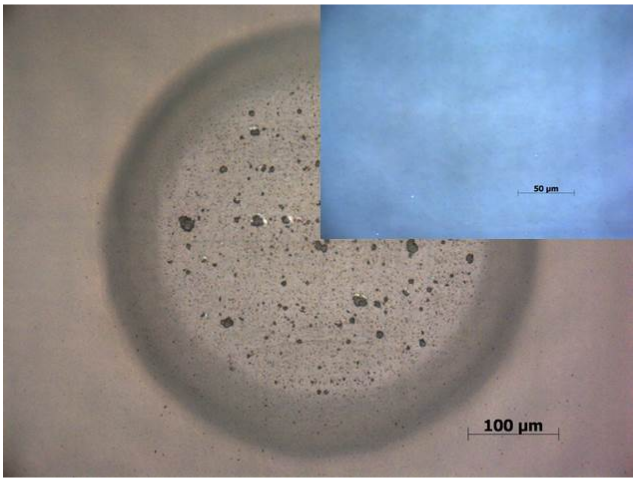

Fig. 9: Laser induced backside damage on a 12 µm glass membrane. The next consecutive scan destroyed the membrane. At a 14 µm thick membrane the glass surface is still intact (insert).

4. Conclusion

Surface homogeneity at the overall ablation area including side walls of the bore and backside surface of the membrane is essential to produce thin membranes especially in high refractive index materials (for example SiC). In such materials, LIPSS in combination with surface irregularities (grooves or scratches) promote the formation of pinholes. Pinholes funnel laser radiation into the bulk material causing structural damage and stress in the membrane. The edges of grooves, scratches and pinholes provide focusing structures with very short focal length. Hot spots of intense focused laser radiation can destroy a thin membrane. Especially the membrane corner area is vulnerable for pinhole formation. We developed an effective counter measure against pinhole formation which is the permanent change of the laser polarization direction after consecutive scans. In lower refractive index materials such as glass, sapphire or diamond we observed no threatening pinhole generation by laser ablation.

In these materials focussing effects of sharp structural edges or scratches are likely the main cause for structural damage in the membrane and measures to generate a smooth ablation area contribute most to the membrane quality. In high refractive materials LIPSS function very well as slot waveguides and consequently the ablation result is dominated by the laser polarization direction, focussing effects contribute secondary for the final optimization of the surface quality. This observation is in accordance with our simulation results for SiC. The calculated laser radiation distribution confirmed the experimental findings qualitatively that LIPSS in high refractive index material act as slot waveguides and promote pinhole formation at spatial disruptions. At locations of scratches and grooves interrupting the slot waveguides, the growth of pinholes is promoted by dislocated laser radiation prior confined in the slots. Changing the direction of polarization interrupts the growth cycle of the pinholes effectively and increases the quality of the fabricated membrane. We observed one effect, the transition of LIPSS into HSFL which requires both, a strong focusing effect at sharp edges and the effective slot waveguide function in high refractive index material. We demonstrated that femtosecond laser ablation is a powerful tool to generate a variety of structures for possible applications e.g. in MEMS sensor devices, microfluidic and biotechnology. Controlling the laser polarization is an effective method to influence the interaction of the laser radiation with the material used. For the laser radiation we demonstrated three different cases of main interacting mechanism responding very specifically to the substrate materials or geometric conditions. For good quality ablation results one has to consider micro focussing effects in glass, the slot waveguide function in SiC and the combination of focussing and waveguide function for the LIPSS to HSFL transition in SiC pinholes.

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Johann ZEHETNER was born in Amstetten, Austria in 1958. He received his Ph.D. from Technical University Vienna in 1992 for his research on diode-pumped and mode locked laser systems. He has acquired experience in research and industry at the Osaka National Research Institute, New Cosmos Electric Co. Ltd. Osaka, Emitec Japan in Tokyo and Trotec Laser Japan Ltd. At present he is senior researcher and lecturer at the Research Centre for Microtechnology, University of Applied Sciences Vorarlberg. His research interests include femtosecond laser ablation and materials processing for medical, fluidic and sensor technology.

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