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Enhanced imaging of Subsurface Damage in optical glass SF6 with Optical Coherence Tomography using KOH wet etching

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Abstract. Mechanical cracks induced during grinding of brittle materials known as subsurface damage (SSD) reduce mechanical and optical properties of optical components. A characterisation of SSD is needed to guarantee a good quality and to optimize individual processes and processing chains. Current research focuses on non-destructive methods such as optical coherence tomography (OCT) to evaluate SSD depth and distribution and to replace currently established, but time-consuming and labour-intensive destructive methods. Yet the imaging of SSD remains challenging, even with high-resolution OCT providing a high sensitivity. The presented work proposes a combined measurement approach of enhanced SSD imaging by using a potassium hydroxide (KOH) wet etching process prior to OCT measurement. An etching process using 30% KOH at 80°C is applied and resulting etching rates are analysed. It is shown by an iterative etching experiment on optical glass SF6 that the KOH etching process enhances OCT signals of SSD under the surface, revealing up to 2.4-times deeper maximum SSD depths using an identical measurement setup.

1 Motivation

Mechanical removal processes in optics manufacturing of brittle materials go hand in hand with the initiation of unwanted cracks called subsurface damage (SSD). As described by Preston, those cracks extend far below the surface [1]. SSD reduce the optical and mechanical properties of optical components and therefore need to be tracked and removed. There exists a variety of measurement methods for SSD as summarized in review papers [2],[3], and even tough several non-destructive methods have been proposed, the mainstream procedure remains a destructive taper polishing method.

Optical coherence tomography (OCT) has been identified as a generally suitable measurement method for non-destructive SSD detection [4] and since then profited from improvements in OCT hardware and commercial OCT systems. Nevertheless, challenges for a broad and industrial application remain. SSD detection with OCT may still struggle with measurement artifacts in low intensity signals, making weak signals and smallest SSD being hard to detect. With the proposed combined characterisation method using etching and OCT, an easier and more reliable measurement and evaluation using OCT is strived for, which still could be considered as non-destructive in intermediate processes, as revealed SSD must have been removed in following steps anyway. With the known SSD depth, those subsequent processes can be optimized to a minimum removal depth.

2 Material and Methods

Measurements have been conducted on a sample of optical glass SF6 (Schott AG, Germany). The sample was ground by an iterative loose-abrasive grinding process using grain sizes of F230 and F800. Additionally, the sample has been measured by a destructive reference method called spherical polishing which has been described by Seiler et al. [5] Reference SSD depths were determined to be in the order of 60 µm.

Raw data was acquired by a commercial spectral-domain optical coherence tomography (OCT) system GAN621 (Thorlabs GmbH, Germany) slightly adapted to suppress specular reflections on the sample surface using a cross-polarization method. Measurements were taken from an area marked using ultra-short pulse laser machined deep holes as persistent labelling throughout the iterative etching process. The laser marking was later used for co-registration of iterative measurements.

Iterative etching experiments were conducted with 30% potassium hydroxide (KOH) solution (Carl Roth GmbH+Co. KG, Karlsruhe, Germany) on a hot plate heated to 80°C. Etching parameters have been chosen based on literature values [6] to provide a relatively fast etching rate with KOH as etchant. Etching rates were determined on separate cuboid samples of the same material with all faces being either polished or ground to investigate the influence of the initial surface condition on the etching process. Average etching rates were calculated from 5 repeated runs with 8 h etching duration each.

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Data evaluation of OCT measurement was performed using a custom Matlab® program. It consists of co-registration of the individual measurements using the laser markings, thresholding, binarization and filtering of the grayscale values and subsequent visualization of relative depths between surface and deepest defects at each specific location in so-called color-coded depth maps as shown in Fig. 1, already described elsewhere [7].

3 Results and Conclusion

Etching rates were repeatedly determined on separates samples of the same material SF6 for ground and polished surfaces. Surface topographies were characterized using a white light interferometer TMS-1400 (Polytec GmbH, Germany). As expected, ground surfaces showed higher etching rates R_{SF6} than polished surfaces as listed in Table 1. The increase in etching rate by a factor of ~ 5.9 from polished to ground surfaces can only partially explained by the increase in effective surface by a factor of ~ 3.6 due to surface roughness. The effective surface is quantified based on ISO 25178 by the developed interfacial area ratio (Sdr) and the deduced relative area r (r = 1 corresponds to a perfectly flat surface), calculated as follows:

$$r = 1 + Sdr \qquad (1)$$

Table 1. Surface characteristics and etching rates of optical glass SF6 in KOH (30%, 80°C) on ground and polished surfaces.

	ground surfaces	polished surfaces
S_q [nm]	3,600 ± 128	25.3± 1.6
S_z [µm]	45.353 ± 1.127	0.294 ± 0.007
Sdr [%]	264.66 ± 12.74	0.42 ± 0.06
r	3.647	1.004
R_{SF6} [nm·h^{-1}]	822 ± 8	139 ± 8

SSD depths were calculated based on the color-coded depth maps as seen in Fig. 1. As characteristic values, we use an SSD depth $SSD_{A\%}$ related to an areal threshold A%, indicating that for a given depth, only A% of the en-face area still contains a higher SSD depth, and 1 - A% of the en-face area at this depth is defect-free. As threshold A% in this paper, we chose 0.1% as for the destructive reference method. The maximum SSD value (SSD_{max}) corresponds to the deepest SSD being captured by our method, equal to A% = 0.01% Additional to standard SSD depths, depths relative to the initial surface are calculated by addition of the etching depth t_e, see Table 2.

$$SSD_i = SSD + t_e \qquad (2)$$

Not only quantitatively but also in visualizations of SSD distribution, the influence of etching on OCT measurements can be identified, e.g. see Fig. 1. It is shown that etching prior to OCT measurements improves the capability of the measurement systems to reveal SSD depths as well as their areal distribution. Looking at the evolution of SSD depth as well as etching depth at the time stamps given in Table 2, it can be deduced that etching seems to significantly broaden the cracks to enhance OCT imaging but doesn't initiate a significant crack growth as the depth between 1 h and 16 h of etching doesn't continue to significantly increase.

Table 2. SSD depths and calculated etching depths for iterative etching steps.

time stamp	before etching	after 1 h of etching	after 16 h of etching
t_e [µm]	0	0.8	13.2
SSD_{max} [µm]	109.0	267.0	259.0
$SSD_{max,\,i}$ [µm]	109.0	266.2	245.8
$SSD_{0.1\%}$ [µm]	32.0	256.0	188.0
$SSD_{0.1\%,\,i}$ [µm]	32.0	255.2	174.8

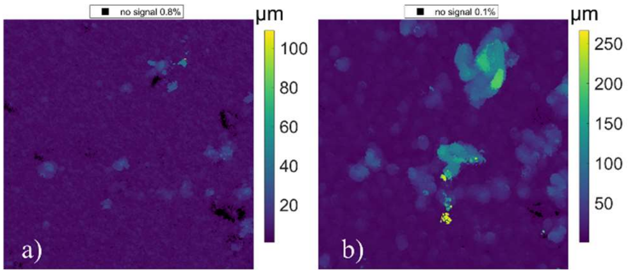

Fig. 1. Co-registered color-coded depth maps (FOV: 500 x 500 µm²) showing subsurface damage in SF6 during an iterative wet etching process in KOH (30%, 80°C) solution a) before etching. b) after 1 h of etching.

The proposed method of enhanced imaging of SSD using OCT and a prior etching process tackles difficulties in imaging low-intensity signals originating from deep and small defects. It is shown that with the proposed etching parameters, OCT signals from subsurface cracks in ground optical glass SF6 can be improved, even revealing additional SSD information. Future work will focus on a deeper understanding of the subsurface etching process as well as evaluating the method on other materials.

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