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Capillary Sharp Inner Edge Manufacturing

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ABSTRACT

This paper presents production conditions of classical optical grinding and polishing method, when producing sharp inner edges on fused quartz glass capillary or other brittle materials. A sharp inner edge of capillary is one of the necessary conditions for successful surface tension measurement of very small liquid samples. Tested production process has two stages. First, the smooth capillary tip surface is achieved with ductile mode fine grinding. Then, grinding continues with gradual decreasing of grinding load. Finally, rests edge chipping defects are minimized with a polishing process. A task to perform quality check of manufactured capillary inner edge was found out to be very complicated. We present a review of our experiences with four experimental techniques for sharp 90° edge sample quality check.

INTRODUCTION

This paper presents a labor technique and experimental conditions that have been used to achieve a sharp inner edge with minimizing of glass capillary tip chipping common in slurry polishing. Sharp inner edge of capillary is one of the necessary conditions for successful liquid surface tension measurement using Ferguson method [1]. Although this experimental method is not a standard method for liquid surface tension measurement, it is the only method, which can be used for surface tension measurement of small amount of liquid at supercritical state [2].

The Ferguson surface tension measurement method is based on measurement of pressure difference across a small (less than 1 mm³) liquid sample placed in the small diameter capillary, in condition of flat liquid sample meniscus at the capillary tip. Details of prepared experiment can be found in [3]. Sample meniscus flatness is very strongly affected with any defects present on the inner capillary edge like an edge chipping and dust or impurities contamination, what can be seen in the figure 1. To overcome mentioned problems an optimum technology of capillary sharp inner edge preparation and cleaning was searched.

A standard technique of capillary splitting is it's cleaving with some standard capillary cleaving tools. This technique is based on introduction of a small, defined mechanical stress onto the capillary surface layer and then its propagation across the capillary profile. When a cleaving occurs, mechanical stress propagates as a shock wave through the brittle capillary matter, where the wave front forms small lines or circles of variable height in the cut. These

defects concentrate close to mechanical impediments of the cut profile as holes of the capillary or hollow fibers, what is shown in cleaved examples in the figure 2.



Figure 1: Capillary tip meniscus shape affected by capillary inner edge defects and impurities

Cleaving technique gives sufficient shape and surface quality for fibers connection technique, but the quality of cleaved capillary inner edge is not sharp and smooth sufficiently for our meniscus shape measurement used in surface tension experiments. We have to finish the cleaved capillary tip to fulfill our quality needs.

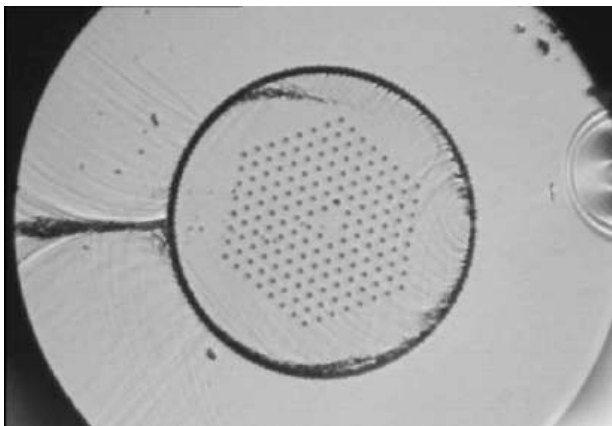


Figure 2: Typical geometrical defects of cleaved hollow fiber or capillary – left [www.crystal-fibre.com], AFM scan of cleaved hollow fiber visualizing surface geometrical defects– right [16]

Problems of capillary finishing using grinding or polishing are very weakly referred. Recently Kurobe et al. [4] referred on inner wall of a stainless steel pipe finishing using high-speed slurry flow only. More references can be find on fiber faces production [5-7], where the machining area has similar dimensions as our capillary tip. The problem of fiber tip optimal machining parameters set was studied with respect to optical surface quality achieving at minimal working time [8-11] especially. But the main technical problem of small glass area machining consists of material edge chipping [11]. This problem was well known for old gem

cutters, who says, any brittle materials should not be grind at angles 90° or less. Recent paper confirms this old experimental experience and it introduces a simple and universal relation of edge chipping under single point loading close to the workpiece edge [12]. This fact is traditionally solved with beveled edges of optics, what is not acceptable in our case.

Next in the text we describe a grinding process used for sharp inner edge fused silica capillary finishing. The grinding process limitation conditions are presented and we compare our grinded capillary inner edge chipping with theoretical end experimental results of references [11] and [12]. Finally the instrumentation problem of the capillary inner edge quality check is also presented.

Experimental set-up

As a good machine for our capillary edge manufacturing we decided to use a small home made grinding machine, originally designed for grinding of diamond crystals intended for single point diamond machining. The reasons were similar dimensions of a capillary tip to grinded diamonds and sharp edge enough achieved during diamond grinding. Used machine have had to be slightly modified. The original work piece holder head allowing two perpendicular working angles set of diamond machining was replaced with a career plate mount slightly above a grinding plate.

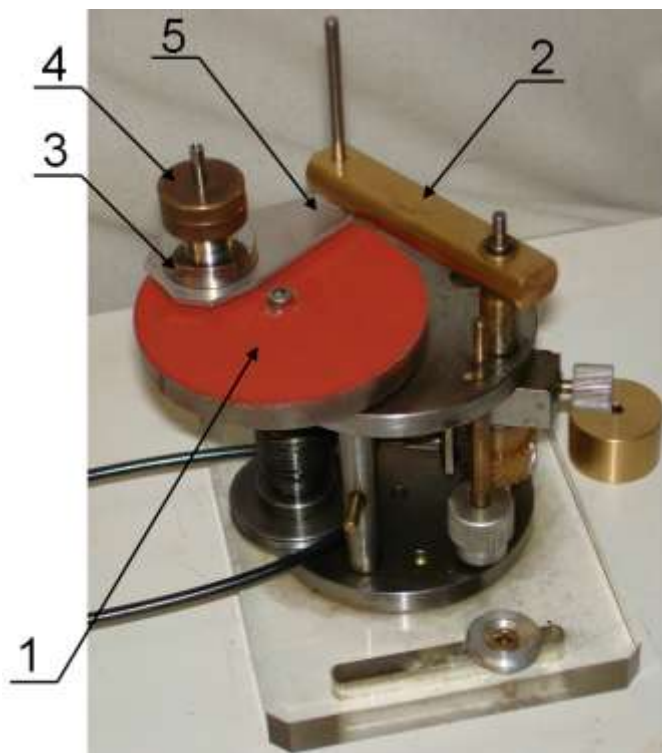


Figure 3: Real construction of the grinding machine.

Adapted grinding machine, described in detail in [13], is shown in the figure 3. Machine consists of cast iron grinding plate 60 mm in diameter – 1 driven by external electric motor with continuously controlled speed, movable lever – 2 and stainless steel work piece holder head – 3, loaded with adjustable weight – 4, placed inside the career plate – 5. Quartz glass

cleaved capillary of diameters 0.98/0.32 mm and length 35 mm is fixed with a spring collet and placed to the holder head in the career plate. Capillary load was set with weight assembly. Capillary perpendicular position was given with geometrical tolerance of stainless steel holder head of diameter 19.6 mm and it was better than $5'$. Grinding plate speed can vary in range 60-800 rpm. Movable lever carried out pendulum movement of the workpiece on the grinding plate in the range of 50 – 84 % of its diameter. Velocity of this movement is proportional to the grinding plate speed with ratio 1:54. This movement causes the capillary grinding speed variation in the range of 0.75-1.25 of grinding speed at 40 mm diameter. This additional movement was used to uniform unevenness of the grinded capillary surface with no negative effect to the surface quality caused by induced speed variation. The more intensive influence to the capillary inner edge quality had the in-feed rate set by the capillary load in the range of 10-150 mN/mm².

Experimental conditions and results

We performed series of experimental observation of capillary tip within a machining process with the goal of achieving the sharp enough capillary inner edge. Machining process consists of classical optical grinding condition involved grinding using F 1200 diamond grit (grit dimensions 2.6 – 3.6 μm) and finishing using Fe₂O₃ dispersed on polishing pad.

Defects observed on the capillary surface during the grinding originate in two grounds. The first one is caused by brittle grinding mode; the second one originates in inner stress in the capillary induced within its manufacturing or dividing process. Both effects are enhanced with the increase of in-feed rate or by mechanical vibrations of the capillary during its grinding. It means the essential condition necessary for the sharp capillary edge achieving is to ensure a ductile regime [14,15] of surface removal. This condition, however, is not sufficient condition for satisfaction sharp edge manufacturing, cause of small defects appear on the machining edge in ductile regime too as it is seen in the figure 4.

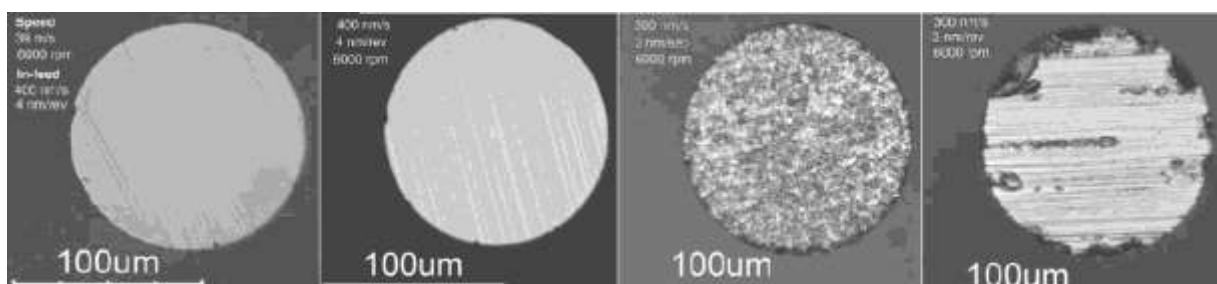


Figure 4: Example of machined optical fiber tip chipping using diamond grit from left to right 0,5 μm , 1 μm , 3 μm and 5 μm under similar machining conditions [11].

It is a consequence of individual grit cutting process during the statistical machining, where each acting grit is assumed to be a cutting edge with temporal variable removal rate close to the average value. While the ductile regime of surface grinding is limited with critical depth of grit penetration to the surface proportional to the loading force, the critical load condition of edge chipping exponentially changes close to the edge [12]. This exponential dependence of critical load causes the little variation of the grit load, what leads to the chip formation in the edge otherwise the machined surface quality maintains well.

In order to achieve minimum chipping on capillary edges we firstly refine capillary tip

with diamond tool. Then we ground the capillary tip to the smooth surface using a ductile regime of grinding with minimal edge damage. We tested different grinding speeds and loads. We found out the optimal grinding condition for our grinding machine and fused quartz glass capillary tip diameters 0.98/0.32 mm to be the load approximately 100 mN/mm², what induces the in-feed rate of 28 nm/s and 9 nm/s for 3 μm and 1 μm grit respectively. The sufficiently flat surface was achieved in 30-50 minutes of grinding, but few small defects up to 5 μm radial dimension from the edge were still observed. The process is frequently checked with laboratory microscope Meopta with magnification 225 in transmitting and reflecting beams. Then we decrease capillary grinding load to approximately 22 mN/mm², with in-feed rate of 1,7 nm/s to minimize residual chips dimensions. Finally capillary tip was finished with Fe₂O₃ longtime polishing using polishing pad. Polishing time depends on pad rigidity, in order to prevent sharp edge rounding.

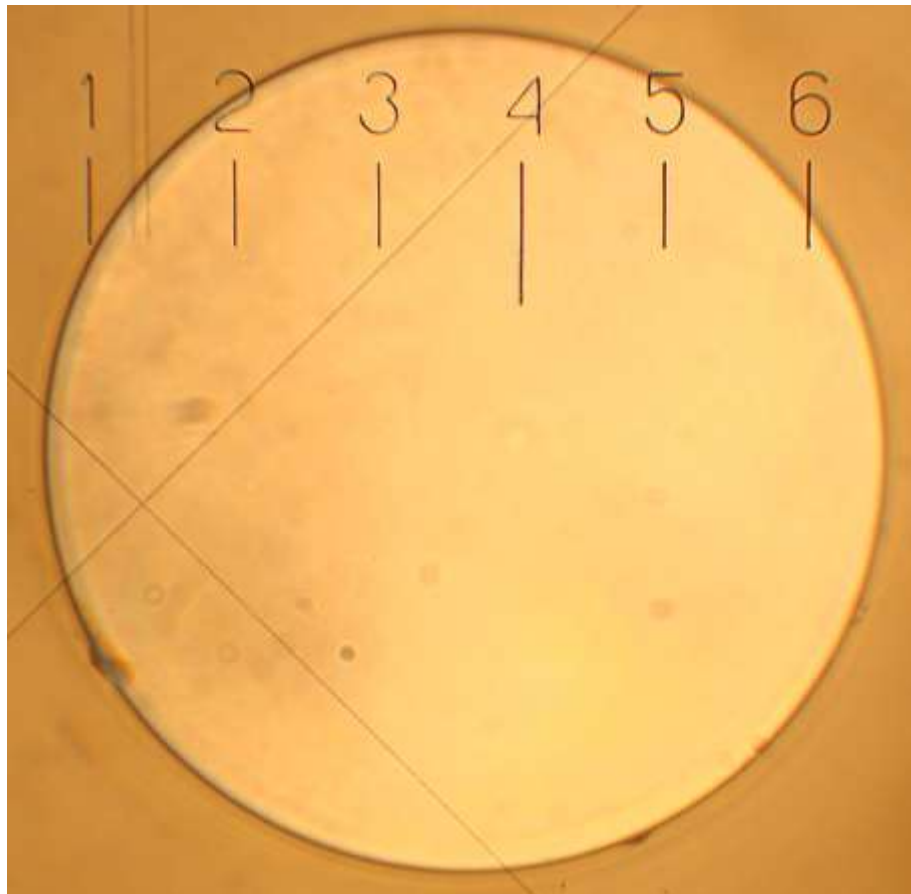


Figure 5: Grinded and polished capillary inner edge visualized in transmitting white-light beam. Grid line spacing is 58 μm. Interference fringes close to the edge can be observed.

This way we are able to manufacture sharp enough inner capillary edge with edge rounding less than 1 μm. The edge, unfortunately, is still affected with a number of residual cracks as it is shown in the figure 5. These cracks are small enough that its effect on liquid meniscus shape quality used in surface tension measurement is negligible.

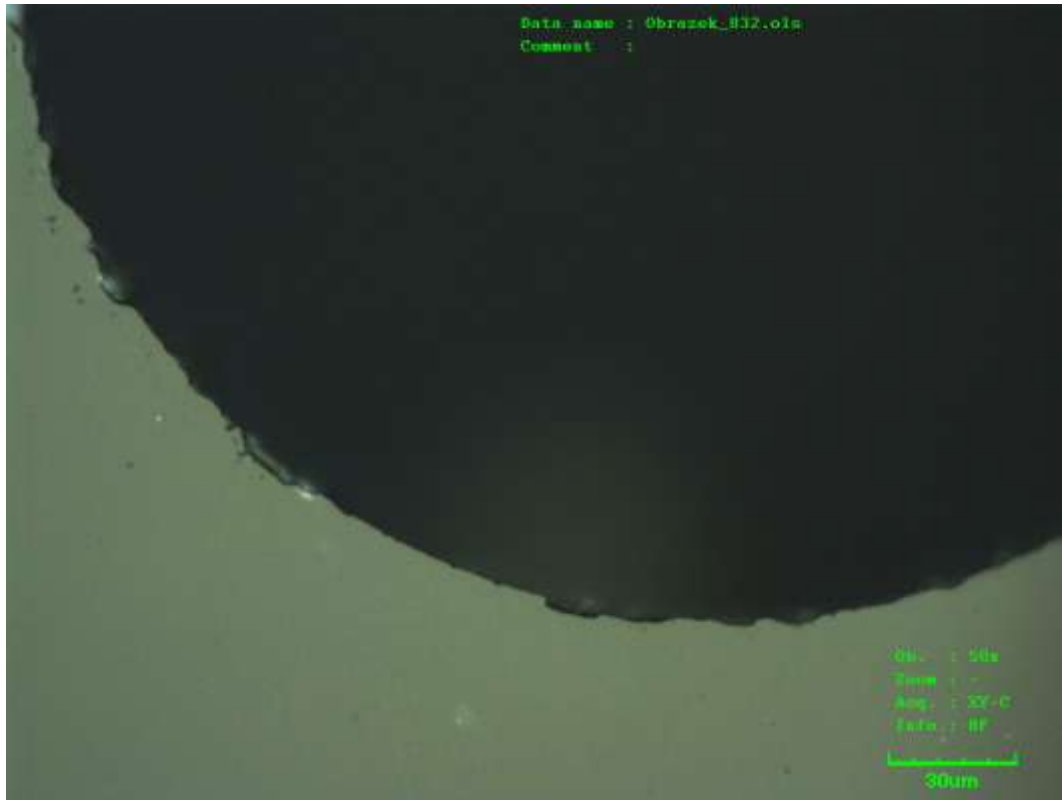


Figure 6: Detail of grinded capillary inner edge with F 1200 diamond grit taken with Confocal Microscope LEXT. Residual edge defects of 0.5 - 3 μm radial dimensions are seen.

Edge quality check and discussion

We performed machined capillary tip surface quality check systematically through the whole manufacturing process. We use a laboratory microscope Meopta with magnification 225 in transmitting and reflecting beams to optimize machining load and process termination. We are able to visualize surface and edge defects down to dimension 3 μm . We observed interference fringes close to the edge using conventional microscope with polychromatic quasi-collimating beam and back illumination in the image plane of the microscope too. During the experiments, we found out the fringes are probably caused by interference of beams reflected on capillary inner surface instead of edge diffraction effects. So we found out it is not possible to use it for edge quality detection, but it can be probably suitable to use it for inner surface quality check.

We had to solve another possibility to check the edge quality – its radius and dimensions of residual cracks. This process was complicated because of two purposes. For the first one a sharp edge on a brittle material is very sensitive to any mechanical load in general, so the contact less measurement methods or very delicate contact methods of the edge radius measurement can be applied only. Another difficulty arises from the problem to perform surface profile measurement on a step change of the profile gradient of value 90°. This condition limits the possibilities of majority of tested measurement methods.

For contact less capillary edge profile measurement we firstly try to use an optical profilometer MicroProf FRT (Fries Research & Technology GmbH). This system performs a topography measurement using chromatic white light sensor on 300 μm z-range sample with X,Y resolution 1 μm and Z resolution 3 nm. This system gives good results of capillary tip quality $R_a = 12$ nm, but the limiting factor for sharp edge measurement of this instrument happens its maximal 30° slope measurement angle. So this system was not possible to use for edge radius determination.

As a next measurement system we used a confocal laser microscope Olympus LEXT. This technique enables to measure 3D profiles on samples of wide dimensions 70 mm with resolution of 0.25 μm in horizontal axis and 100 μm in vertical axis with resolution of 0.12 μm . The inner edge capillary measurements were performed in two opposite 45° capillary inclined positions to maximize the extension of observed profile from the measured edge. Examples of two measured profiles of machined sharp inner edge and chipped edge of quartz capillary are shown in the figure 7.

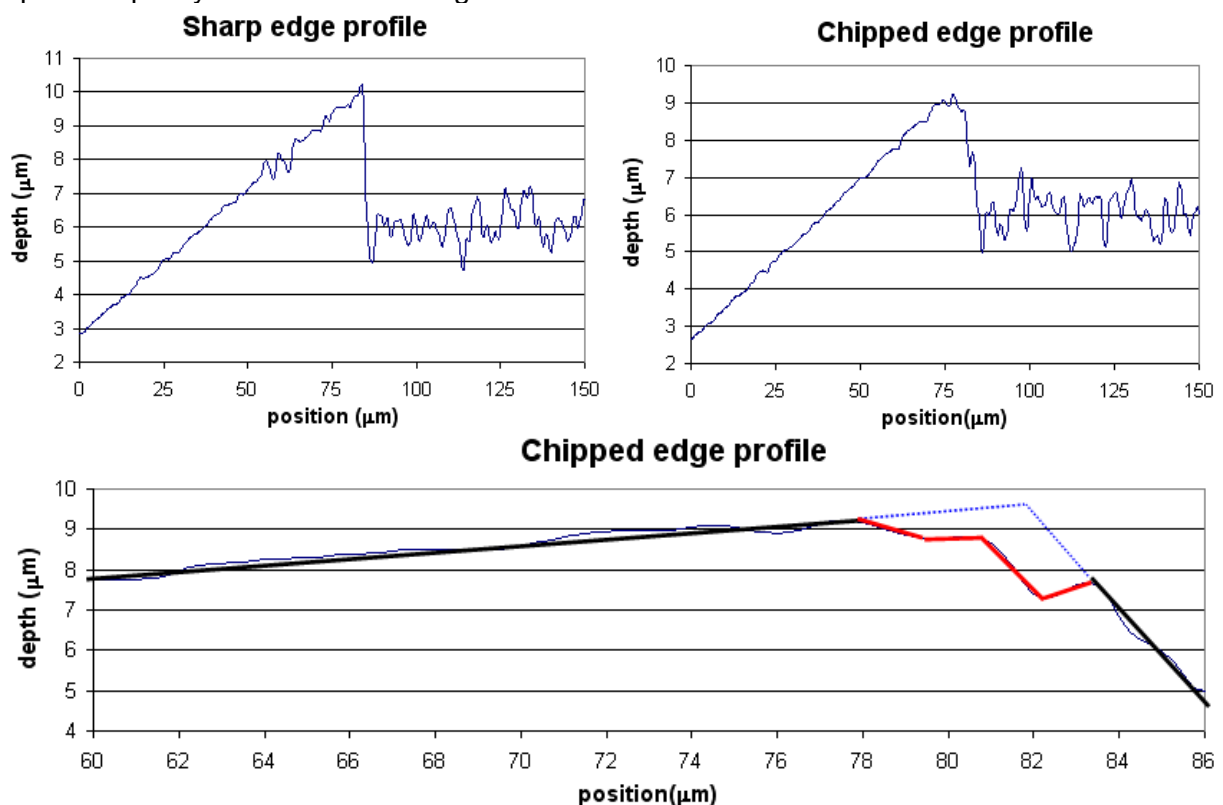


Figure 7: Measurement of capillary inner edge profile with confocal microscope LEXT: sharp edge – left and chipped edge – right, detail of the edge chipping - bottom.

Measured edge geometrical profiles show the presented grinding technique is possible to achieve sharp inner edge of glass capillary of radius in range of 0.1 - 2 μm . We found out the radial dimensions of these defects vary in range up to 2D after grinding, where D is grit average dimension. This result is in good agreement with fiber face chipping in grinding experiments [11] shown in the figure 4. Residual chipped edge profile data evaluation was more difficult because a conchoidal edge fracturing. If a multiple chipping occurs, as shown in the figure 7 in the bottom, such edge can be approximated with radius in range of 2 - 6 μm .

We check the profile of small chip cracks with LEXT microscope and larger ones with defocusing of an optical microscope and we found out its profile ratio $c/h \approx 2$, where c is a crack depth and h is a radial crack distance. This chip shape well corresponding to a glass instability point occurred under single point critical load [12]. It seems the observed edge chipping could be caused by a single grit close to the edge temporally loaded because of grinding tool instability or the single grit dimension greater than average grit dimensions.

The last method of the polished capillary inner edge quality checks we used an atomic force microscope (AFM) PSIA XE-100 equipped with high aspect ratio probe for final. This technique is limited with work range of 100 μm in X, Y-axis, Z-axis range 25 μm and 16-bit resolution on all axes. Next limitation comes from 12 mm Z-axis sample size. A contactless-mode Si cantilevers with resonant frequency around 330 kHz and typical tip radius of less than 10 nm was used. We used two capillary positions during the measurement. For the first position we wish to measure the whole 35 mm long capillary within maximal Z range usage. This would be theoretically possible when the capillary was inclined to 45°. Unfortunately the Z-axis range of the microscope was too narrow to find the inner capillary edge using embedded optical microscope and motorized movable stage. The second possibility a horizontal capillary tip measurement position had to be performed with cut down capillary.

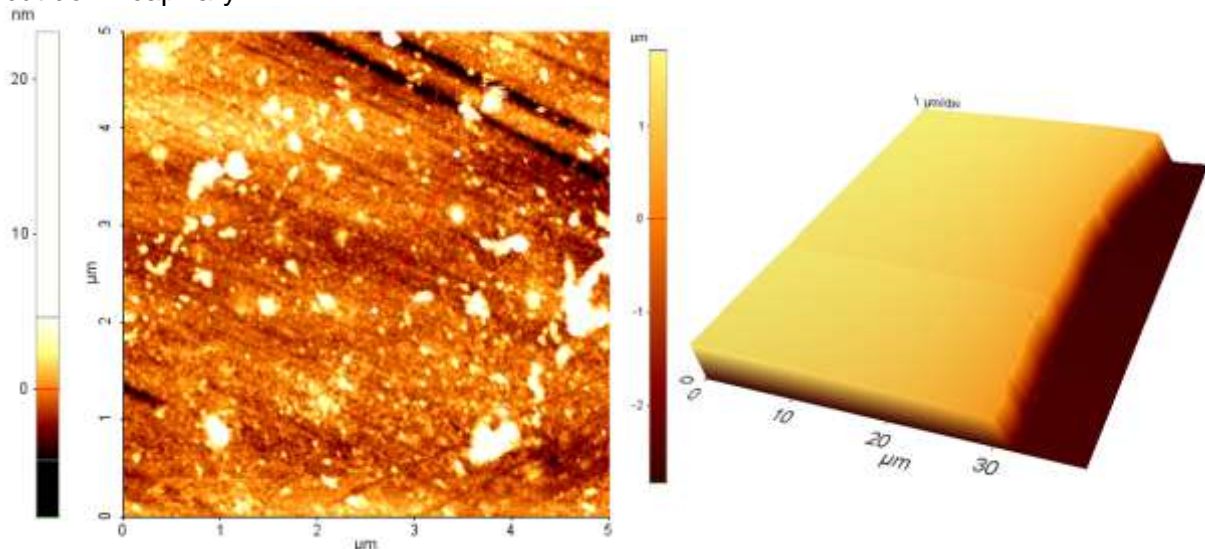


Figure 8: AMF scan of capillary polished surface – left and detail of chips on inner edge – right.

This measurement position was successful and shows high capillary tip quality up to $R_a = 0.6$ nm, $R_z = 2.7$ nm on clear tip surface, as it is shown in the figure 8 left. Machined capillary edge is still affected with residual chipping caused by small edge defects during grinding, because it would take too much time to polish it out completely. These residual chipping slightly deviate the real capillary edge from its ideal circularity up to 3 μm , as it is shown in the figure 8 – right, but these defects are present in limited edge length, usually down to 10% of the whole edge perimeter. We measured the edge radius between capillary tip and the inner wall using AFM microscope and it gives smaller radius values than 100 nm as for chipped as for non defected parts of the inner edge. We experimentally verified that such machined edge radius is sufficient enough for optical meniscometry necessary for liquid surface tension measurement using Ferguson method. Disadvantage of AFM measurement



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is, there is also not possible to measure individual cracks profiles because of its high depth. A high aspect ratio probe is not able to measure inner capillary wall profile down to 1 μm under tip surface and standard probes create artifacts, because of its 30° conical geometry.

CONCLUSIONS

Our results demonstrate the possibility to achieve very sharp edges down to 100 nm radius on the brittle material as a fused silica capillary using classical optical grinding and polishing technology in the applications where sharp edges are needed. Machine conditions, which allow achieving of sharp edges on brittle like glass materials, involve ductile grinding mode, gradually decreasing of load during the grinding and finishing polishing. This method, unfortunately, not eliminates on brittle material edge chipping completely. We observed a capillary edge chipping up to 5 μm radial dimensions, but our experiments of liquid sample surface tension measurement using Ferguson method shows that such capillary chipping is acceptable for this measurement. This edge chipping with radial dimension fewer than two grit diameters during ductile mode grinding is comparable to fiber edge chipping of fiber face grinding experiments [11]. Our machined capillary chipping geometry compared to a single point indentation experiments [12] shows the chipping can be caused by single grit highly loaded close to the capillary edge. It means the edge chipping can be minimized by better mechanical stability of grinding machine and better homogenization of grit dimensions. Finally we performed capillary inner edge radius quality check using few different experimental techniques too. The best results give us measurement with use of confocal laser microscope, where large enough samples as 35 mm long capillary with 90° surface morphology can be measured.

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