



Local hardness and density variation in glass substrates machined with Spark Assisted Chemical Engraving (SACE)

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ABSTRACT

Spark Assisted Chemical Engraving (SACE) is an unconventional technique for surface micro-machining of non-conductive materials specially glass. SACE offers many advantages for fabrication of microfluidic and Lab on Chip devices. However the exact mechanism of material removal in this technique is not fully understood. Besides, the changes in the properties of the machined sample have not been studied so far. In this letter, the material removed from glass surface is evaluated and the results of nano-indentation test for measurement of the hardness of machined micro-channels surface is reported. Based on the amount of removed mass during machining and results of nano-indentation test on machined samples it is concluded that hardness and density of the machined zones decrease during the process.

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1. Introduction

Fabrication is one of the main issues in microfluidic devices. Several technologies have been developed in order to fabricate suitable devices for microfluidic and Lab-on-a-Chip applications [1,2]. Spark Assisted Chemical Engraving (SACE) is a non-traditional micro-machining technology for low-cost machining of micro-holes and micro-channels in non-conducting materials, such as glass and some ceramics [3,4]. Ability of SACE to combine local heating and local chemical etching has enabled it to take advantage of both chemical and physical processes for fabrication of microfluidic devices.

Machining takes place in an electro-chemical cell where in general the cathode is used as machining tool. When a voltage higher than a critical value is applied, bubbles grow so dense on the tool electrode that they coalesce into a gas film [5]. At this stage electrical discharges take place between the tool electrode and the electrolyte (Fig. 1). Machining happens if the tool electrode is brought to close vicinity of the substrate (less than 25 μm for glass [3]). Detailed descriptions of the fundamentals of the gas film formation have been reported in the literature [3,6]. Micro-hole drilling was the very first application presented for SACE [7–9], however recently fabrication of 2D and 3D micro-structures with SACE has gained interest [10,11].

The exact mechanism of material removal in SACE has not been fully elucidated yet. The process has been generally attributed to “melting due to local heating by the electrochemical discharges and

possibly other chemical effects” [3,8,12,13]. In addition to the lack of knowledge about material removal mechanism, the change of glass properties after machining has also never been studied. Analysing glass properties after machining can be helpful in understanding the material removal mechanism and would also lead to more insight into the final properties of the microfluidic device which could determine its range and degree of applicability. In the present study changes in hardness and density of the glass after machining are investigated by nano-indentation test and by analyzing the amount of removed mass during machining.

2. Experimental procedure

Standard glass sample holders for optical microscope with a thickness of 1 mm, (Menzel-Gläsler, soda lime glass) were used as working pieces to be machined with SACE technique. The experimental procedure, consisting of five steps is as follows:

- Cleaning of glass samples in an ultrasonic bath for 5 min with acetone followed by mass determination with an analytical balance (0.1 mg precision)
- 2D-micro-machining with a micro-channel pattern according to the procedure described in [10].
- Cleaning of machined samples in an ultrasonic bath with subsequent mass measurement and characterisation of micro-channel geometry with an optical microscope
- Preparation of sample for the nano-indentation tests by diamond saw cutting of the work-pieces
- Nano-indentation test on the micro-channel surface and the un-machined surface of the cut work-pieces

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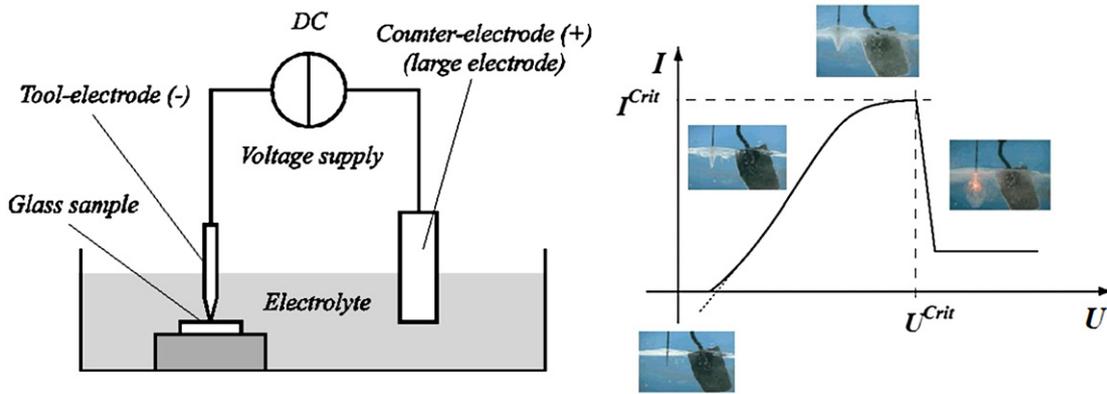


Fig. 1. Principle of the SACE process. (a) Schematic diagram of SACE setup, (b) Current–Voltage diagram for the electrochemical cell as machining takes places.

The electrolyte was aqueous sodium hydroxide solution (10, 20, 30 and 40 wt.%) prepared from NaOH purchased at Sigma Aldrich. In order to achieve micro-channels with good quality, the machining speed was chosen to be 5 $\mu\text{m/s}$ and the applied voltage to be 28 V [10]. For all the experiments a cylindrical, 0.5 mm in diameter, stainless steel tool was used and the length of the machined micro-channels was 40 mm.

The nano-indentation test was performed by the load-controlled indentation using a Hysitron Nanoindenter with a Berkovich pyramid indenter (edge radius: >40 nm). The load was increased up to 2000 μN , kept constant at this value and decreased to zero, each stage taking 5 s. It was found to be hard to accurately align the tip of the nano-indenter inside the micro-channel surface. To overcome this issue, the samples were placed in the nano-indenter such that the cross section of the micro-channel could be monitored by an optical microscope to assure that the tip touches the machined surface of the micro-channel.

3. Results and discussions

The amount of removed material from samples surface was measured by two different techniques: 1) Using an analytical balance and 2) Based on the removed volume as calculated from the geometry of the machined micro-channels. In the second technique, the removed volume was calculated by using an optical microscope to identify the geometry of the micro-channels. Glass density was assumed constant

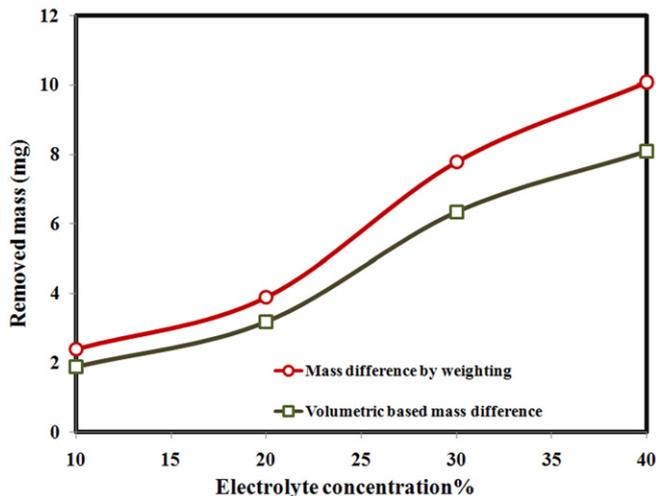


Fig. 2. Material removed based on measuring samples weight before and after machining and based on calculated volumetric removed mass.

before and after machining. Fig. 2 shows the removed material from glass surface calculated by the two methods. The results are contradictory to what is expected: the two techniques yield to different figures for the removed mass.

The analytical balance gives the mass of the samples with 0.1 mg precision and the geometry of the micro-channels is determined with 1 μm precision. The density of the samples (soda lime glass) was calculated by gravity metric methods to be 2.45 g/cm^3 . The density of soda lime glass in literature has been reported to be between 2.2 to 2.45 g/cm^3 [14]. According to these figures, it is concluded that the measured difference between the two techniques are significant and not an artifact of the experiment.

The percent deviation of the removed mass calculated by the two methods is shown in Table 1. This deviation is about 20% in all cases. This difference is attributed to the change in local density of glass in the machined areas. In order to confirm this change in glass property, the nano-indentation test was performed on the surface of machined micro-channels.

The nano-indentation test was performed on the free surface (un-machined area) of the glass sample five times in different points; the results show a hardness of 6.8 (± 0.1) MPa. The nano-indentation test was also performed on the machined micro-channels surface. The test for the micro-channels surface showed a hardness of 5.2 (± 0.5) MPa. Fig. 3 shows the force-displacement graph of nano-indentation test inside and outside the micro-channel which was machined with 30 wt.% electrolyte concentration. This difference in hardness justifies the previous hypothesis about the change in the physical properties of machined substrate and shows that the material becomes softer after machining. This softness is a clear indication of changes in glass density.

The percent deviation for the measured hardness of micro-channels surface and the hardness measured for the un-machined glass surface is about 23%. This is in the same range as the percent deviations shown in Table 1 for the removed mass measured with the above mentioned techniques. Therefore this change in local glass density explains the deviation of the two aforementioned methods to measure the removed mass.

A possible explanation for the change in density of the machined substrate could be the fact that the substrate is quenched quickly during machining. Decreasing of soda lime glass density by fast cooling has been reported in literature [15]. The local machining temperature is reported to be more than 600 $^{\circ}\text{C}$ [3,10]. The glass surface is locally exposed to the electrochemical discharges for a short period of time and cools quickly when the tool moves away. The temperature on the surface also drops dramatically with distance from the machining tool. As mentioned above, the machining voltage and speed are the same in all experiments. Therefore the exposed energy and the local machining temperature remain constant for all experiments. Since all samples are cooled with the same rate, the change in the density should be equal for all the experiments regardless of the electrolyte concentration. The equal percent deviations obtained in Table 1 confirm this.

4. Conclusion

2D-micro-machining with a micro-channel pattern was performed on the soda lime glass samples by Spark Assisted Chemical Engraving

Table 1
Percent deviation of the removed mass calculated from two different methods

Electrolyte concentration %	Percent deviation %
20	20.3
30	20.5
40	20.8

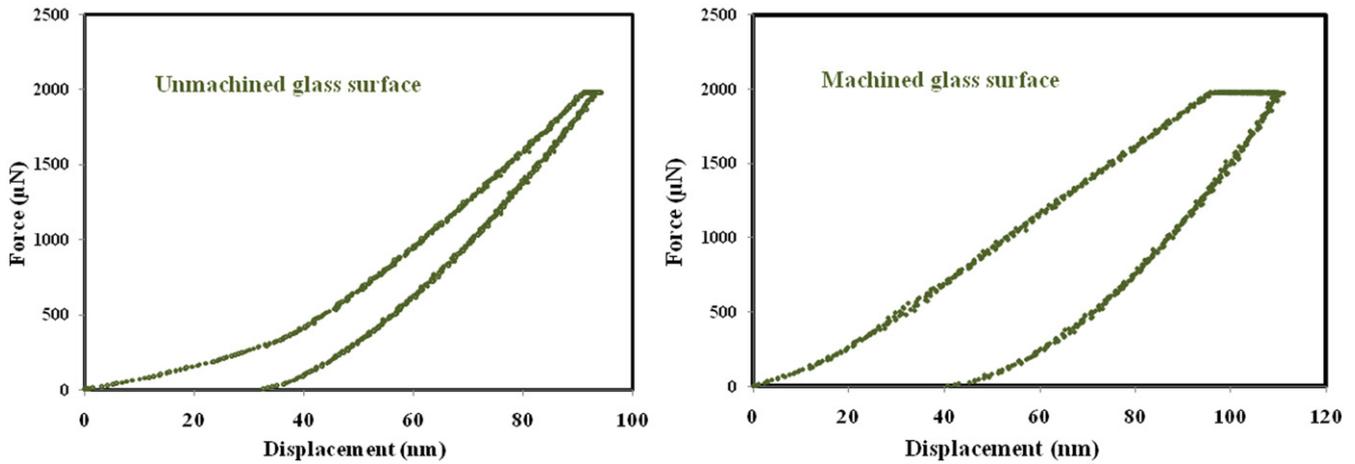


Fig. 3. Force–Displacement graphs of nano-indentation test before and after machining using 30 wt.% electrolyte concentration.

technology. The difference in hardness of the machined areas shows that the material becomes softer after machining. This softening is an indication of changes in glass density. The difference between calculations of removed mass by analytical balance and geometrical methods, followed by the results from the nano-indentation test indicate that the density of the machined surface decreases during the machining process. This change of density is attributed to the fast cooling of the work-piece during machining process.

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