Micro-texturing channel surfaces on glass with spark assisted chemical engraving

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Abstract
Micro-texturing of glass micro-channel surfaces during machining by spark assisted chemical engraving (SACE) technology is reported. A range of surface textures is obtained from feathery-like to porous spongy-like textures. It is demonstrated that the texture formed on the channel surface is a mimic of the electrolyte flow patterns induced during machining. The electrolyte viscosity is found to be the most significant factor influencing the channel texture among other factors including tool-work piece gap, machining voltage, and tool travel speed. Pulsed voltage, used to control the local temperature at the machining spot, has also proved to influence the surface texture. As a result, different channel surface textures were obtained during SACE machining by controlling the mentioned parameters. Further this work demonstrates the capability of SACE to both micro machine and texture glass surfaces in one machining operation. The present results add to the importance of SACE technology, knowing the significance of channel micro-texturing in microfluidic and biomedical applications.

1. Introduction
Nowadays, with the evolution of nanotechnology, nano/micro texturing of surfaces has become a requirement for micro devices utilized for optical, electronics or biomedical purposes. It is from this perspective that extensive research on surface texturing has been carried out in the past years where a large variety of methods were proposed for modifying the surface texture of different materials. These methods include heat treatment, ion irradiation, laser ablation, and chemical etching. By irradiating silicon surface with ethanol cluster ions, micro patterns with different sizes (3 μm to 100 μm) were obtained [1]. Mücklich et al. proved that Laser Interference Metallurgy can be used to fabricate periodic micro-patterns on metallic samples by the interference of several laser beams [2]. Moreover, various patterns were created on silicon, TiO2 and steel surfaces by laser ablation [3,4]. Anisotropic etching conducted in aqueous alkaline solutions [5], reactive ion etching [6], wet acidic etching [7], and vapor texturing [8] were also used for nano-texturing of single and multi crystalline silicon. For texturing PTFE tape surface, hot embossing by Ni mold was applied [9]. Eliaz et al. modified the surface texture of ground titanium by heat treatment of the sample after immersing it in NaOH or H2O2 bath [10].

Micro-texturing has already found wide applications especially in optoelectronic and biotechnological fields. For example, micro textured surfaces can be used for photovoltaics and bio-mineralization [3], in addition to being employed as scaffolds for growing animal cells [11]. Surface texturing is also crucial for increasing the hydrophobicity of surfaces [4] and minimizing the reflection losses in solar cell devices [12].

By modifying the surface texture inside a channel, the fluid flow and thus the heat-transfer characteristics of the channel change [13]. In addition, the surface texture affects the cell adhesion onto the surface. For example, the texture of channels coated with hydroxyapatite (by electrochemical deposition), influences the adhesion of osteoblastic cell (bone forming cell) on the surface [10].

Spark assisted chemical engraving (SACE) is a promising micro-machining technology used to machine non-conductive materials, mainly glass, by electrochemical discharges [14,15]. So far, the focus of the research conducted in the field has been to obtain smooth surfaces. Within this perspective, it was reported that pulsed voltage machining, tool rotation, and tool vibration improve the quality of the glass machined surface [16–19].

This paper reports the micro-texturing of channel surfaces on glass during micro-machining by SACE technology. The surface texturing and micro-machining was done in one operation. Channels with different surface textures were manufactured using the electrolyte flow phenomenon while applying different voltages, varying the gap between the tool and the glass surface.
and changing the electrolyte viscosity and the tool travel speed. A mechanism responsible for the formation of these surface patterns is proposed and discussed.

2. Experimental set-up

In order to machine channels on the glass surface, the workpiece along with the tool and the counter-electrode are immersed in an aqueous sodium hydroxide solution. For high enough potential difference between the tool and the counter-electrode, a gas film (through which electrical discharges are generated) is created around the tool-electrode by bubble coalescence [20].

The setup is composed of a processing cell and a machining head (Fig. 1(a)) mounted on a XYZ Cartesian robot (Newport; positioning error is less than 1 μm). The work-piece, a standard microscope soda lime glass slide (Thermo Scientific Inc.), together with the counter electrode (stainless steel) are placed inside the processing cell while the tool is held by the machining head. The tool-electrode was a stainless steel needle, 500 μm in diameter, and had a tip of a few tens of microns (Fig. 1(b)). The machining head is mounted on the Z-axis of the Cartesian robot. It is composed of a flexible structure free to move parallel to the Z-direction giving an additional degree of freedom denoted by z, a voice-coil actuator able to act on this degree of freedom and an optical sensor that measures z (Fig. 1(a)). The machining head can operate in two modes. In the first mode, it is used as a profile meter (free to move in z-direction; voice coil actuator is switched off), while in the second mode, the z degree of freedom is eliminated by means of a PID controller driving the voice-coil actuator (i.e., the machine head assembly follows the motion of the Z axis of the Cartesian robot). The work-piece is mounted on the processing cell fixed on the XY axes of the Cartesian robot.

In order to study the effect of the electrolyte viscosity on the channel surface formed patterns, a set of experiments was conducted while using different electrolyte concentrations (10 wt%, 20 wt%, 30 wt%, and 40 wt% NaOH having viscosities of 1.7, 4.5, 13, and 39 mPa s, respectively, at room temperature). For every concentration three parameters were varied: the gap between the tool and the glass surface (5 μm, 10 μm, and 15 μm), the machining voltage (30 V and 32 V), and the tool travel speed (5 μm/s, 10 μm/s, and 20 μm/s). With respect to the supply voltage, different duty cycles (20%, 40%, 60%, 80%, and 100%) were used. The tool travel speed was chosen based on [21].

Path planning algorithms (Catmull-Rom splines [22]) were used to manufacture the straight line channels. In order to maintain a fixed gap between the tool and the glass surface while machining the channel, the z coordinates of 20 equally spaced points were measured prior to machining on the glass surface to correct for machine misalignments and variations in glass thickness. The Z coordinates of the trajectory were measured using the profile meter mode of the machine head. Before following the trajectory, the tool-electrode was moved to the start position (work-piece surface) using the XYZ linear stage (Z coordinate of the work-piece was determined with the profile meter integrated in the machining head). Then the tool was lifted upwards by the desired machining gap (5 μm, 10 μm, or 15 μm) above the work-piece surface using the second mode of the machining head. A specific machining voltage was applied between the two electrodes and the tool followed the calculated trajectory while moving with a constant velocity (5 μm/s, 10 μm/s, or 20 μm/s). Five channels, of 3 mm length each, were machined for each set of variables. For a given set of conditions, no significant variation between the five channels could be observed. Note that the machining gaps, as quoted here, are adjusted while the tool-electrode is at room temperature prior to machining. During machining, due to thermal expansion of typically 10 μm [23], this gap will change.

The micro-channels were observed with an optical microscope (Nikon Measuring Microscope MM-400/SL) equipped with a Quadra Chek200 measurement apparatus. Further, a stylus profilometer (XP-200) was used to measure the channel profile.

3. Machining mechanism

3.1. Formation of surface patterns

The following mechanism is proposed to take place. The temperature of the spot below the tool-electrode tip is known to be around 600 °C based on previous studies [23–26]. This leads to two main effects: accelerated glass etching and formation of a glass melt.

Because the temperature in the machining zone is above NaOH melting temperature (318 °C) and below its boiling temperature (1388 °C), a layer of molten sodium hydroxide forms locally. This promotes glass etching by OH radicals [Fig. 2] [24]. However, the local glass surface is not uniformly etched by the electrolytic solution at the same rate. Hence, the surface texture formed is believed to be a mimic of the local high temperature electrolyte flow pattern. Due to the confinement of the micro-channel, the induced electrolyte flow is opposite to the tool travel direction. The forced flow is probably the result of the repeated formation of the gas film, passing by the bubble coalescence and the electrical discharges generation prior to breaking down. Other effects such

![Fig. 1](image-url)
as convection, electro-migration or high local magnetic fields may as well be responsible for the local flow during machining. Further investigations are required to quantify the contribution of the mentioned individual effects.

The second effect is the locally reduced glass viscosity due to the high temperature in the machining zone. Hence, glass may be locally pushed away over a few micrometers either due to the pressure exerted by the gas film (because of its repeated formation and breakdown) on the glass surface, or by the mechanical contact between the tool and the work-piece. The formation of small bump-like structures is therefore possible.

In summary, it is proposed that the texture obtained depends on the electrolyte concentration that influences the wave propagation through the channel and the heat transfer coefficient. Hence, the patterns created on the glass surface are essentially a mimic of the local flow of the hot electrolyte induced by the machining process.

3.2. Stick and jump effect

Depending on the machining gap and the tool travel speed, tool “stick and jump” effect may occur. During machining, a glass bump shaped layer is deposited around the tool (Fig. 3(A)), trapping it at that position. This causes the tool to bend and then to jump to another position on the glass surface as depicted in Fig. 3(B)-(D). The tool then penetrates quickly in the glass (Fig. 3(D)-(E)). Indeed, from gravity feed drilling, it is known that penetration rate is typically about 100 μm/s [27].

Based on Fig. 3(D) the following relation is derived to estimate roughly the distance \( \Delta x \) between two consecutive holes

\[
\Delta x \approx \sqrt{2L\Delta y},
\]

with \( \Delta x \) the height of the bump deposited on the glass surface and \( L \) the length of the tool electrode.

4. Results and discussion

4.1. Influence of electrolyte concentration

Fig. 4 shows representative samples of the channels machined using 10 wt%, 20 wt%, 30 wt%, and 40 wt% NaOH at 28 V and 5 μm/s travel speed. Note how the surface texture changes significantly with different electrolyte concentrations. It evolves from a branched feathery-like texture, towards smooth spongy-like (porous) texture as the electrolyte concentration increases. However, when using 30 wt% and 40 wt% NaOH electrolytic solutions, cracks may form on the surface (Fig. 5). The appearance of cracks for electrolyte concentrations higher than 30 wt% may be due to the increased thermal conductivity of the electrolyte (heat conductivity reaches a maximal value for concentrations above 30 wt% [28]) resulting in fast cooling of the glass.

In the case of 10 wt% NaOH, the surface patterns observed on the channel surface have similarities with the Kelvin wake patterns. The classical methods for predicting the Kelvin wake are based on the assumption of irrotational inviscid flow [29]. These patterns consist of two wake V-shaped lines which diverge in direction opposite to that of the moving body. On the interior of these two lines, feathery wavelets exist which are circular arcs. For the machined channels, as the flow is restricted by the channel parallel walls, the waves propagate inside the channel. Thus, the patterns extend up to the channel edges. This effect is

Fig. 2. Material removal mechanism: during machining a glass melt is formed below the tool due to the high temperature in the machining zone (around 600 °C). The etched glass leaves the machining spot in the form of NaSiO₃.

Fig. 3. Machining mechanism; (A) machining at a single spot on the channel surface; (B) tool stuck at the same position due to the torus formed around it; (C) tool bending and jumping; (D) tool bended after jumping to another position on the surface (neglecting the tool movement in the upward Z-direction); (E) formation of another hole, surrounded with a torus, on the channel surface after the tool machined at the position reached in (D).

Fig. 4. Channels machined at a voltage equal to 28 V and a speed equal to 5 μm/s while using different NaOH concentrations (10 wt%, 20 wt%, 30 wt% and, 40 wt% NaOH). The surface texture evolves from branched feathery-like towards smooth spongy-like texture as the electrolyte concentration increases.
observed in Fig. 4(A). The angle between the wavelets and the tool travel direction is about $37^\circ \pm 2^\circ$, similar to the angle obtained with the Kelvin wake patterns.

For higher NaOH concentrations, the electrolyte is more viscous. Hence, the Kelvin wake pattern theory is no more applicable. As shown on Fig. 4, the wake patterns disappear for higher electrolyte concentration (30 wt% and 40 wt% NaOH) where a smooth uniform channel surface is formed. In summary, the higher the electrolyte viscosity is, the smoother will be the micro-channel surface.

The surface roughness was measured by recording the channel profile for the four different concentrations using a stylus profilometer (XP-200 profilometer). The roughness was then calculated using the ten-point mean roughness method where it is found to be equal to 2.3 μm, 1.5 μm, 0.8 μm, and 0.5 μm for 10 wt%, 20 wt%, 30 wt%, and 40 wt%, respectively.

4.2. Influence of tool travel speed, machining gap and voltage on channel depth

Fig. 6 shows a representative example of channels machined at 30 V, 5 μm gap in 10 wt% NaOH for different speeds ((A): 10 μm/s; (B): 20 μm/s). The wake pattern formed on the surface for both speeds is similar, which is in agreement with the Kelvin wake pattern theory stating that the angle of the wakes is independent of the speed of the moving body. However, channel (A) (around 23 μm deep) machined at lower speed is 11 μm deeper than channel (B).

As mentioned in Section 3.2, for tool speeds higher than the material removal rate, the stick and jump effect occurs. This explains the formation of shallow holes surrounded by tori on the glass surface for high tool speeds (as depicted in Fig. 7 for 20 μm/s tool travel speed). The deposition of the upper edge of a torus on the lower edge of the previous one, as can be very clearly seen on Fig. 7(F), confirms the glass re-deposition process during machining due to the fact that the heated glass is pushed by the tool. In summary, the tool speed mainly affects the channel surface texture since it influences the uniformity of the deposited glass on the surface. In addition, for high speeds holes appear on the surface due to “stick and jump” effect.

Fig. 7 illustrates as well the effect of the machining gap on the surface patterns. On one side, the gap influences the depth of the machined micro-channels and on the other side it significantly affects the distance between the tori centers. It is observed that the channels machined at 5 μm, using 10 wt%, 20 wt%, 30 wt%, and 40 wt% NaOH, had mean depths of about 13 μm, 18 μm, 30 μm, and 40 μm, respectively. For all the concentrations, the decrease in channel depth is about the same as the increase in the tool-work piece gap.

Another indication for the existence of the stick and jump effect is shown on Fig. 9 depicting a channel machined in 30 wt% NaOH with a voltage of 30 V, a gap of 10 μm and a speed of 20 μm/s. The holes appearing on the surface are generally not perfectly aligned due to tool bending and jumping to another position on the surface.
deeper than that machined at 30 V. Note that for 32 V the glass layer deposited around the holes is more pronounced compared to the 30 V case where semi-circular patterns appear on the surface. Furthermore, since the temperature in the machining zone is probably higher (and therefore the glass viscosity lower) than that for the case of 30 V, the formed tori overlap more. This results in an overall homogenization of the channel surface. In the case of 40 wt% NaOH, a smooth almost homogenous surface is obtained. This smooth surface is the result of the combined effect of the high electrolyte viscosity, as described in Section 4.1, and the homogenization effect of the higher machining voltage.

4.3. Channel extremities

Fig. 10 shows the beginning (Fig. 10(A)) and the end (Fig. 10(B)) of a channel machined with 10 wt% NaOH and a speed of 10 μm/s, and the end of a channel machined at 30 wt% NaOH (Fig. 10(C)). For 10 wt% NaOH, in the beginning of the channel (Fig. 10(A)) the upper part of the hole has the wake pattern, whereas the lower edge is covered with a layer (deposited on the surface as the tool is moving onwards). However, for the channel end (Fig. 10(B)), the pattern is spread all around the hole (the wake is formed in the radial direction: from the hole’s center to its circumference). For 30 wt% NaOH (Fig. 10(C)), the channel end has a smooth surface. These observations allow us to get a better insight into the surface patterning mechanism. It can be concluded that the resulting surface texture passes through two consecutive stages. During machining, the electrolyte flows in the radial direction away from the tool tip, due to the gas film dynamics, where glass is etched and re-deposited. As the tool moves forward, successive etching and re-deposition will occur, resulting in a uniform pattern behind the tool and burying the pattern created ahead of the tool. The mentioned process is repeated until the channel is completely machined. A unidirectional texture is eventually formed which is most apparent for the case of 10 wt% NaOH. The different textures appearing on the channels extremities for both 10 wt% and 30 wt% NaOH (the texture being smoother for 30 wt%) are due to the change in the local electrolyte flow when the bulk electrolyte viscosity changes (for 30 wt% the viscosity is about 10 times higher than for 10 wt% NaOH). This confirms our suggestion that the patterns formed on the channel surface are a result of variation in electrolyte viscosity.

4.4. Patterns with pulsed voltage

It was demonstrated in the previous sections that the concentration of the electrolyte, hence its viscosity, affects the surface texture. Not only does the electrolyte viscosity change depending on the concentration, but also its thermal conductivity which affects the glass local temperature. Recent studies showed that by changing the machining voltage duty cycle, the local temperature of the glass in the vicinity of the tool can be tuned.
from about 600 °C down to 200 °C [23]. Hence, pulsed voltage supply with different pulse-off times was used to investigate the effect of the glass temperature on the resulting texture. Fig. 11 shows a series of channels machined using 10 wt% NaOH, 5 µm/s speed and different pulse duty cycles (80%, 60%, 40%, and 20%) of the time during which the voltage was on, resulting in 400 °C, 300 °C, 200 °C, and 100 °C temperatures, respectively [23]. The pulse on-time was chosen to be 80 milliseconds (slightly lower than the mean gas film life time) and the off-time was varied. Note how the surface texture, initially V-shaped for high pulse duty cycle (Fig. 11(A)), passes through a smooth transition towards a thin branched texture as the pulse duty cycle decreases (Fig. 11(D)).

Fig. 12 shows channels machined with tool speeds of 5 µm/s, 10 µm/s, and 20 µm/s and two pulse duty cycles (80% and 20%).

As in the case of a DC voltage described in Section 4.2, increasing the tool travel speed makes the tool stick and jump effect more apparent. As the duty cycle is lower, the heated region beneath the tool becomes smaller and the local temperature decreases. Thus, the etched region by the hot flowing electrolyte is very close to the tool position. As a result, the texture formed extends on a very close area to the tool and it is composed of thin, almost horizontal, branches that extend from the middle of the channel to its walls (Fig. 12(B), (D), and (F)). For higher pulse duty cycles, less time is available to allow cooling down the locally heated region. Therefore, the heated zone occupies a larger surface area around the tool, over which the hot electrolyte flows. Note that, in this case, the wake lines that extend from the channel center to its walls are inclined (Fig. 12(A), (C), and (E)). This proves that the heated zone is wider for higher pulse duty cycles. With respect to the tool speed, the wake pattern is more uniform for lower speed, as discussed in Section 4.2. For low speeds, the material removal rate and deposition is more uniform along the channel length. Fig. 12(F) depicts a channel machined using 10 wt% NaOH, 30 V (20% duty cycle), and 20 µm/s speed. A Kelvin-like wake pattern is formed inside the holes created on the surface (zone 1) due to the stick and jump effect and beside the channel walls (zone 3). However, a smooth surface appears for the area surrounding the tool (zone 2). The wake pattern formed in zones 1 and 3 is due to the glass etching by the flowing molten NaOH. Note that inside the hole, the wake pattern is unidirectional due to its formation at the moment the tool bends and jumps to another spot. Zone 2 is smooth due to the mechanical contact between the surface and gas film upon its repeated formation and breakdown, taking into consideration that in this case low pulse duty cycle is used. This allows the gas film to smooth out the repeatedly formed patterns on the locally hot glass surface (low viscosity) in the tool vicinity. This is a strong indication that the electrolyte flow caused by the repeated gas film formation, not the tool horizontal motion, is the cause of the pattern formation on the surface.

5. Conclusion

In this paper, it was demonstrated that electrochemical discharge machining can be used as a tool to change the surface texture of glass micro-channels. The surface texture obtained is dependent on the concentration of the NaOH electrolyte used, i.e., its viscosity. It was demonstrated that for low electrolyte concentration, feathery-like patterns similar to Kelvin wake patterns are formed on the channel surface while for high electrolyte concentration spongy-like porous texture results. For high electrolyte concentrations, however, cracks may form on the channel surface. Moreover, it was shown that the channels machined at low speed (5 µm/s) had a uniform surface texture and flat walls as compared to channels machined at higher speed (10 µm/s and 20 µm/s). With higher tool speeds, the channels become shallower. Hence, by tuning the electrolyte viscosity and the tool-electrode speed channels with different patterns and sharp edges can be obtained. It was also shown that the tool-work piece gap influences the depth of the machined micro-channels. For all the concentrations, the decrease in channel depth is almost the same as the increase in the tool-work piece gap. The tool speed also affects the channel depth where the depth decreases as the speed increases. Pulsed voltage was used to control the glass and electrolyte local temperature. It was demonstrated that as the tool pulse off time and speed decrease, a more uniform and smoother wake pattern is formed. The results of this work will broaden the field of application for SACE micro-machining allowing it to enter in the development of high precision microfluidic as well as biomedical micro-devices.
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