Simultaneous improvement of surface finish and bonding of centrifugal microfluidic devices in cyclo-olefin polymers

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Machining of fluidic structures

Fluidic structures were directly milled in injection-molded blank COP discs (1.2 mm thick Ø120 mm discs; COP grade: Zeonor 1060R from Zeon) using a 3-axis milling machine (DMG DMC 1035 V). The micromilling was optimized with respect to type of milling tool (single tooth end mill 0.5 and 1 mm diameter), spindle speed (37 000 rpm), feed rate and cut width, in order to achieve low surface roughness (with R_a in the range of 300-500 nm for optimized processes, but can be as high as 2 µm). The disc fixture during milling ensured minimal warpage of the disk and mechanical stability, specifically for machining of 0.9 mm deep structures.

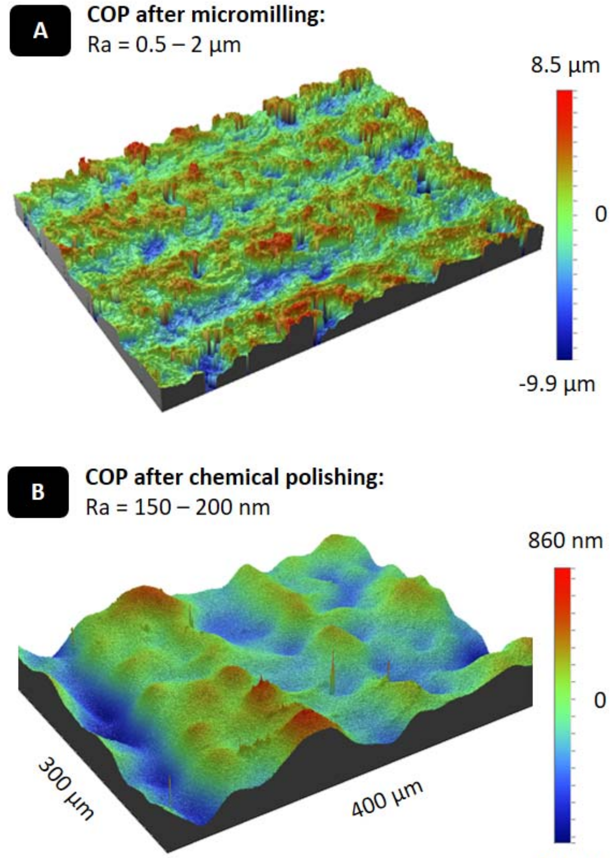

Figure 2: Roughness measurements after milling (A) and after milling followed by chemical polishing (B).

Surface polishing and bonding

A single exposure to cyclohexane vapor was used for both reducing the surface roughness (after micromilling) and as a surface treatment prior to thermal bonding (illustrated in Figure 1). The discs were sonicated in DI water for 10 min and dried with compressed air. Next, the discs were placed inside a petri dish in a Teflon holder with the bonding side facing a cyclohexane bath (disc surface ca. 4.5 mm above the level of cyclohexane) and kept for 4 to 6 min. Immediately after, the discs were aligned using a jig, pre-bonded manually and placed inside an automated hot press between two supporting glass plates.

This was followed by thermal bonding at 65˚C for 30 min, followed by 15 min at 25˚C before removing from the press. The bonding force applied was varied between 1 and 15 kN in the bonding trials. Upon completion of the bonding process the disc was removed from the press and allowed to cool down. At least four discs were tested for each of the bonding conditions.

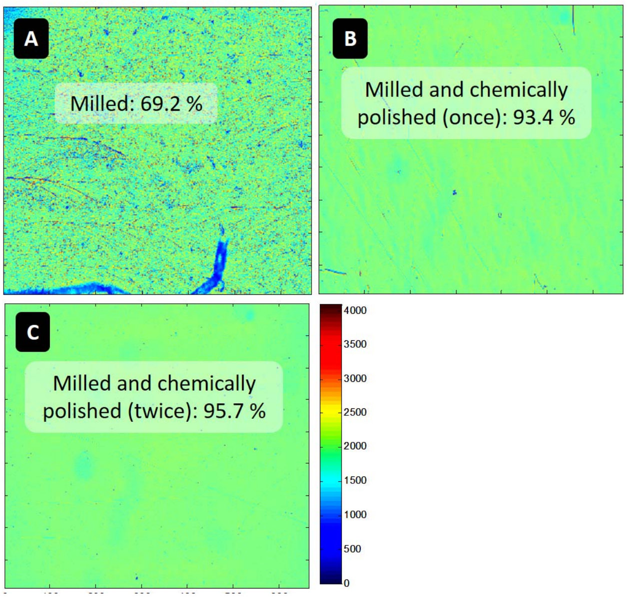

Figure 3: Comparison of microscope images of COP surfaces after milling (A) and after chemical exposure (B, C) with average transmittance values collected in the range 400 to 650 nm indicated. The transmittance value after chemical polishing is close to that of a part injection molded with a polished mold.

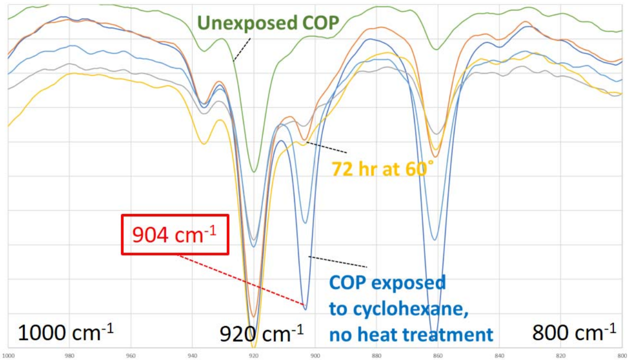

Figure 4: FTIR analysis of COP surface after milling, polishing and heat treatment to eliminate cyclohexane (corresponds to 904 cm^{-1}) from the COP surfaces: peak reduction can be observed for cyclohexane treated surface after heat treatment; no cyclohexane peak is found on unexposed COP surface.

Characterization

Surface roughness and dimensional integrity of fluidic structures were assessed before and after the chemical polishing step using WLI (Veeco® Wyko NT9800) and optical microscopy (Olympus® DSX100). FTIR-ATR measurements (PerkinElmer® Spectrum One with diamond crystal) were performed with 4 scans and 3 areas per sample. Water contact angles (Kruess® DSA 100 drop shape analyser) of the COP surfaces were measured with drop volumes of 3 μL and 10 μL.

Optical transmittance was characterized using a custom-made setup. A plane monochromatic wave (bandwidth of ~ 4 nm) was focused onto the COP surface at normal incident. The transmitted light was collected by a 2x microscope lens and focused onto a CCD camera.

Figure 5: Contact angle measurements (3μL drop volume) of COP surfaces before milling ("intact"), after milling, and after milling and chemical polishing.

The shear stress required to debond COP substrates was measured using a Dage 400Plus multipurpose bond tester. Test samples (5 x 5 mm^2) were milled out of the COP-COP stack bonded at optimal conditions from three positions on the disc (R18, R30, R55). The force was applied to the centre of the top COP substrate in the bonded sample.

The fluidic tests were carried out using an automated custom-made centrifugal test stand equipped with a stroboscopic light source, a camera for visualization of liquid flow and a motor. Rotational speed (< 5000 rpm) and time (< 30 min) were controlled during leak tests.

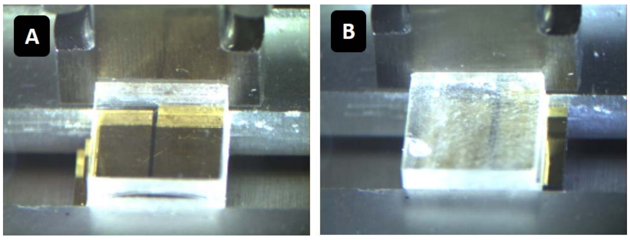

Figure 6: Shear test: COP-COP bonded sample before (A) and after (B) delamination (shear stress of 15.7 MPa).

RESULTS AND DISCUSSION

Reduction of surface roughness

It was demonstrated that a 6 min chemical polishing step is sufficient to reduce the surface roughness from 500 nm (R_a after micromilling is in the range of 300-500 nm for optimized milling processes, but can be as high as 2 μm) down to 100–150 nm, see Figure 2. Multiple exposures to cyclohexane yield further reduction in surface roughness by a few percent. However, maximum two subsequent exposures are recommended in order to avoid distortion of channel and chamber dimensions.

Surface transparency

It was shown that chemical polishing gave transmittance values in the range 92 – 95 % as measured with our setup. These values were comparable to the reference parts (prepared by injection molding with a polished mold). Transmittance data with corresponding images are summarized in Figure 3. Comparison of optical transmission of COC surfaces treated with various solvents, including cyclohexane, can be found elsewhere[6].

Physical and chemical surface properties

Residues of cyclohexane were found on the COP surfaces even after 72 hours of drying at 60˚C (Figure 4). However, this residue is expected to be negligible for typical surface-based chemistries. The wettability of the COP surface is slightly increased by milling and polishing (Figure 5), based on the static water contact angle measurements. This might eliminate the need for hydrophilic coatings[12] and surface treatment for some fluidic applications.

Bonding

The same polishing step was used to prepare COP discs for thermal bonding. Bonding conditions were optimized for both large and shallow detection chambers, and shallow micro channels, resulting in a transparent interface. It was found that 65 ˚C and 10 kN are optimum conditions for achieving a tight and transparent bond without the largest chambers collapsing.

Bond strength measurements on test structures (Figure 6) indicated that delamination occurred after applying a shear stress of about 6.8 MPa (mean value). The highest shear stress value measured was 15.7 MPa. The bond strength was found to vary depending on the area of the disc, with lowest value of 0.6 MPa measured for the samples taken from the outer edge of the disc. However, even the lowest value is still sufficient for leak-tight device operation and comparable to some previously reported data for COP-COP bonded microfluidic chips[4,13].

Successful testing of a centrifugal device with surface quality suitable for optical detection (e.g. in transmission mode) was demonstrated, see Figure 7.

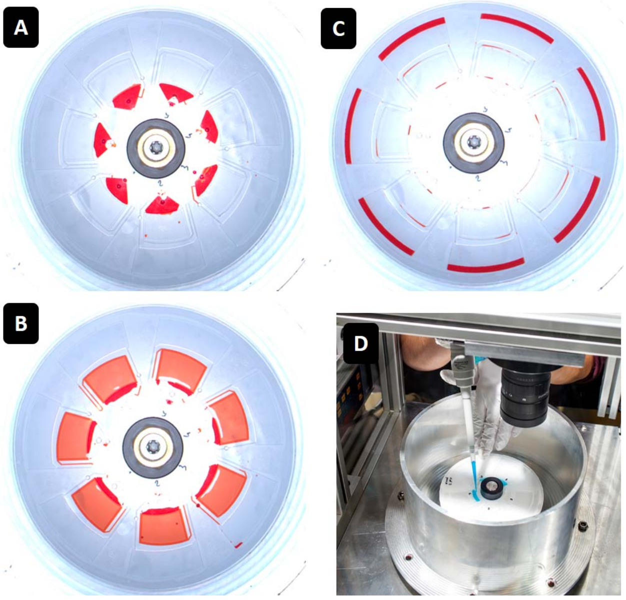

Figure 7: Example of a centrifugal device manufactured using the discussed prototyping method: loading of reagents – chamber A (A), transfer of reagents for incubation – chamber B (B), transfer of reagents to waste via a siphon valve – chamber C (C), disc loading (D).

CONCLUSIONS

We have demonstrated manufacturing, characterization and testing of COP-based centrifugal microfluidic devices. For COP-based microfluidic prototypes, micromilling in combination with chemical polishing is an attractive alternative to injection molding and hot embossing (expensive inserts and masters are required), and to 3D printing (3D printed COP parts are not available), when microfluidic structures with low surface roughness and high transparency are required.

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