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SURFACE MODIFICATION OF CYCLIC OLEFIN POLYMERS: INTEGRATION OF AIR ATMOSPHERIC PRESSURE IN MICROFLUIDIC DEVICES

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ABSTRACT

The exact control of fluid flow and reactions at the microscale that is made possible by microfluidic devices has transformed a variety of scientific disciplines, including biology and chemistry. Because of their high level of optical transparency and biocompatibility, cyclic olefin polymers, often known as COPs, are among the most prominent materials used in the production of microfluidic devices. This research concentrates on surface modification strategies for COPs, more especially addressing the incorporation of air atmospheric pressure in order to improve device performance. Surface modification is very necessary in order to customize the wettability and functioning of the surfaces of microfluidic devices. In this research, we investigate a unique method that modifies the surface characteristics of COPs by utilizing the air's atmospheric pressure as a driving force. We produce regulated surface roughness and chemical modifications by carefully managing air pressure and temperature. This leads to enhanced fluid management and analyte interaction within the microchannels.

Keywords: *Cyclic Olefin Polymers, Air Atmospheric Pressure, Microfluidic Devices*

INTRODUCTION

It is a type of microfluidic technology known as droplet-based microfluidics. This sort of microfluidics makes use of the interplay between flow shear force and surface tension in microchannels to separate two distinct immiscible fluids into discrete droplets with nanoscale or smaller volumes. Extremely high rates of droplet generation can typically result in the production of thousands of microdroplets every single second. Surfactants are often introduced into one of the liquid phases in order to assist an improvement in the dispersion stability of the created droplets. This helps to prevent the merging of numerous droplets, which ensures that the microdroplet system is able to maintain its stability even when subjected to high temperatures. It is possible for each droplet to function as its own microreactor, which would considerably increase the specific surface area of the overall reaction system. This would make it possible to speed up the different reaction rates as well as the heat transfer rates, while simultaneously lowering the risk of cross-contamination.

As a direct consequence of this, droplet-based microfluidics has steadily become one of the new instruments in the disciplines of molecular biology, drug production, single-cell research, and nanomaterial synthesis. Notably, droplet-based microfluidics has already been successfully applied to digital polymerase chain reaction (PCR) analysis in the field of molecular biology. This includes the QX series of digital PCR instruments produced by the Bio-rad Company, which are widely used as promising molecular diagnostic devices. In addition, this analysis was performed using a droplet-based microfluidics system. Digital PCR

devices, as opposed to real-time fluorescence quantitative PCR instruments, are able to gather data from absolute quantitative analysis by means of the observational counting of individual microdroplets following amplification. This is in contrast to real-time fluorescence quantitative PCR instruments. In addition, the reagent consumption may be decreased by using the microdroplet approach, which ultimately results in a higher level of heat transfer effectiveness throughout the PCR process.

Disposable microdroplet generation chips are frequently added into digital PCR machines. This is done in an effort to prevent the cross-contamination of biological material, which might potentially have an impact on the correctness of the subsequent experimental results. It has been determined that polymer injection-molding chips are the best option for digital PCR equipment since they have a large manufacturing capacity and are very inexpensive. The copolymers of cycloolefin (COC) are frequently used as raw materials for the polymer injection molding of microdroplet generation chips because they display the qualities of outstanding optical transparency, chemical resistance, low water absorption, and good biocompatibility. As a result of these merits, they are frequently used.

The COC have several benefits, but due to their low wettability, they cannot be employed with certain hydrophobic liquid reagents that are routinely used. As a result, the relevant surface modifications are still necessary. For instance, Roy and colleagues used a plasma treatment on the surfaces of COC to assess the hydrophilicity of those surfaces. This was accomplished by varying the quantities of various gases, the power levels, and the duration of time. In addition, the surface morphologies and wettability of COC were analyzed and compared under a variety of situations. Additionally, the affects of surface modification on the bonding strength of COC chips were investigated. The accompanying experimental results indicated that the processing of COC surfaces by plasma treatment can not only render them hydrophilic but also increase the associated bonding strength. This was proved by the fact that the plasma treatment In addition, Balamurugan et al. investigated the surface fluorination modification of several polymer materials, including PC, PMMA, and COC. They discovered that enhancing the surface hydrophobicity of the materials led to an improvement in the dimensional homogeneity of the microdroplets that were formed. An technique that consisted of simultaneously bonding with a solvent and fluorinating the surface was utilized by Su et al. in order to achieve both bonding and the endowment of hydrophobicity in a single step.

However, the current research mostly focus on the intuitive enhancement of the uniformity and stability of water-in-oil droplets generated by a chip hydrophobic treatment in a laboratory setting. However, in the industrial production process, far larger considerations need also be addressed. For instance, variations in the microchannels during the bonding of different batches, pressure fluctuations caused by the constant operation of the pressure pump in droplet generation devices, and a variety of other factors can all contribute to the changes in microdroplet sizes. These factors include the small differences in the microchannel sizes of the chips that occur during mass production; pressure fluctuations caused by the constant operation of the pressure pump; and variations in the microchannels.

In contrast, for PCR systems, an invalid microdroplet will be discovered during the subsequent PCR data processing if the microdroplet size limit is exceeded. This will occur when the associated microdroplet size tolerance is exceeded. A poor homogeneity of microdroplet sizes does not correspond with the statistical concept of the Poisson distribution that is followed in the construction of PCR equipment, and this is something that should be brought to your attention as well. It is hypothesized that the changes in the microdroplets caused by such varying factors can be mitigated through the hydrophobic treatment of microchannels, which will allow for more uniform microdroplets to be stably generated and an increase in

the number of effective microdroplets required for PCR detection. This will decrease the sensitivity of the microdroplet generation process to pressure fluctuations and the precision of microfabrication.

In this study, a comprehensive investigation was carried out into the surface morphologies, functional groups, and contact angle (CA) modifications of COC chips both before and after the application of a fluorination treatment. The droplet creation process of COC microchannels and the uniformity of the microdroplet sizes were compared and studied between the chips with and without fluorination treatment between one week of fluorination treatment and four months of fluorination treatment. These comparisons were made between the chips with fluorination treatment and those without fluorination treatment. The impacts of the storage duration of the fluorination-treated COC chips were also clarified, in addition to the implications of microchannel fluorination on the regularity and stability of the creation of microdroplets. In addition, the effects of water-phase pressure on variations in droplet sizes as well as the process of droplet formation were investigated while all other circumstances were held constant. The fluorination-treated COC-based chips that were used in this work were, as a final step, included into a PCR test. This body of work has the potential to act as an essential reference for the mass manufacture of droplet microfluidic chips for commercial use in the future.

OBJEACTIVES

1. The Study Surface Modification of Cyclic Olefin Polymers.

2. The Study Integration of Air Atmospheric Pressure in Microfluidic Devices.

RESEARCH METHODOLOGY

In recent years, chemistry, biology, and medicine have devoted a substantial amount of their research efforts to the development of microfluidic systems for many regular activities. This is owing to the fact that the tiny format results in a decrease in the amount of time required for analysis as well as the volume of reagents required. Microfluidics has been successfully applied to the fields of genomics (the genotyping or sequencing of DNA), proteomics (the identification of proteins), and clinical diagnostics (the detection of viruses or other pathogens). However, substrate materials need to be selected very carefully in terms of cost, biocompatibility, mechanical properties, chemical properties, optical properties, and ease of fabrication, amongst other properties.

Standard lithographic manufacturing technique, in conjunction with the use of glass or silicon substrates, has been the focus of a significant portion of the early research and development on tiny chemical analysis systems. In contrast to glass and silicon, polymers are an appealing material that is perfectly suited for single-use disposable devices due to the fact that they are easy to fabricate, have high biocompatibility, and are inexpensive. The use of polymeric materials in chip-based systems has been the subject of an increasing number of research endeavors. In a number of in-depth reviews, various manufacturing methods of polymer microfluidic devices, including as casting, laser ablation, imprinting, hot embossing, and injection molding, as well as their applications for genetic research, have been examined. In the process of fabricating microfluidic devices, several polymeric materials, including as poly(dimethylsiloxane) (PDMS), poly(methyl methacrylate) (PMMA), and polycarbonate (PC), have been utilized.

The vast majority of hydrophobic polymers are already available on the market for use in microfluidic applications. Polycarbonate (PC), poly (PMMA), polydimethysiloxane (PDMS), and copolymer of 2-

norbornene ethylene (also known as 'cyclic olefin copolymer,' or COC are some of the materials that fall into this category. Because of its hydrophobic character, the substance could be difficult to handle when it comes to liquids in microfluidic devices. Wetting hydrophobic channels requires the use of an external pump, as opposed to the use of capillary force, which is used to load samples into hydrophilic channels. Because of the hydrophobic interactions, the surfaces may be able to seize certain chemicals from the solution as it travels through the channels. This results in a change in the concentration of certain compounds in the solution, which in turn affects the reliability of quantitative tests. A proper functionalization of the surface of the polymer microchannels would make it possible to manage the flow and adsorption processes, and it would substantially increase the microchips' dependability in general.

The current body of research provides a multitude of illustrations of surface alterations carried out on a wide variety of polymers. Because they seldom provide selective patterning on the device, most of these methods are not appropriate for use with microfluidic chips and should be avoided. This need gives an advantage to processes that are initiated by ultraviolet light. The cyclic olefin copolymer is one of the most widely used polymer materials, and it has a good optical clarity in the ultraviolet light spectrum that is comparable to that of glass and PDMS (>80% transmission at 320 nm wavelength), in addition to a low background fluorescence. This material has been utilized to create microfluidic devices for clinical diagnostics because to the fact that it possesses an excellent mix of optical clarity, mechanical strength, and a low cost. Our research focused on the behavior of fluids in surface-modified polyolefin microchannel networks. The surface of these devices was changed with a procedure that involved UV-mediated grafting after they were produced with a method that involved hot embossing. The imbibing flow that was driven by capillary force, pulsed drop motion, contact angle hysteresis, and loading of separation media were all explored by us. The use of these devices for the separation of nucleic acids was demonstrated using the polymer devices after they had been built.

Applications such as surface activation, wound treatment, and sterilization are finding more and more usage for atmospheric-pressure low-temperature plasma jets (APPJ). The capacity to modify the active species created in the plasma jets so that they are compatible with the treatment needs is essential to the development of effective applications that make use of these systems. The purpose of this research is to give an optimization of the influence that APPJ has on cyclic olefin polymers (COPs). The wettability of the surface of COP was shown to have a correlation with the geometry of the water contact angle. For the purpose of process optimization, historical data satisfying the D-optimal criterion were utilized. On the surface of the COP, the influence that plasma parameters have such as the input voltage, the frequency of the plasma power supply, the air flow rate, and the distance between the nozzles was examined. The findings of analysis of variance (ANOVA) indicate that the suggested model may be utilized to traverse surface wettability in a highly effective manner. It was determined that the optimal working condition for achieving the smallest water contact angle could be anticipated. According to the findings, the COP surface modification is impacted by all of the parameters that were investigated; however, it appears to be more sensitive to plasma frequency and distance, as well as air flow rate.

Electrochemical Detection on Microfluidic Device

For the purpose of detection in capillary electrophoresis on microfluidic devices, the electrochemical detection modes of amperometry, conductimetry, and potentiometry are the three most prevalent methods utilized. due of its high sensitivity and the simplicity with which it may be included as a detection element in a microfluidic device system, amperometric detection is an attractive option. This is due of its high

sensitivity. It does, however, suffer from interferences caused by the high voltage (HV) employed in electrophoresis, which is often utilized within microfluidic devices for the sample separation and transport. Because of this, there is frequently a requirement for decoupling in order to ground the separation voltage prior to the detection cell, and a significant amount of research and development has been carried out in this area over the past several years. The application of conductimetry as a method of detection is garnering an increasing amount of attention. It is a technique that may be used to detect compounds that do not have any electrochemical activity, and as a result, it is a technique that is complimentary to the amperometric detection method. However, conductometry has a lesser sensitivity than other measurement methods.

Amperometric Detection

The method can be utilized in either a three-electrode (working, auxiliary, and reference), or a two-electrode (working, and reference), system to accomplish the desired results. Only those compounds that are oxidized or reduced at the specified potential are identified when they emerge from the micro channel when amperometry mode is engaged because the potential of the working electrode is held at a constant value. In spite of the fact that amperometry is the electrochemical detection approach that is most frequently utilized in microfluidic devices, its commercial usefulness is constrained by two drawbacks. Before the species reach the detector, which maintains a constant potential lower than 2.0 V, decoupling is necessary because it reduces the magnitude of the high-voltage electric field (on the order of several hundred V/cm) at the end of the channel. This occurs before the species reach the detector. In the second place, the repeatability of the detecting current is something that has to be worked on. The consistency of the WE and the reproducibility of the location of the WE in relation to the outlet of the separation channel are two factors that are extremely important to the repeatability of the detection current.

Device fabrication

Construction of molds. A depiction of the fabrication method for the polymeric microfluidic chip can be seen in, which is a modified version of a previously published approach. On the silicon wafer, the Shipley AZ9620 photoresist from Microchem in Newton, Massachusetts was spin-coated (2000 rpm for 30 seconds) before being soft-baked at 90 degrees Celsius for 2.5 minutes. After exposing the photoresist to UV light for fifty seconds with the help of a contact aligner, the resultant wafer was developed for thirty seconds with the help of an AZ 400K developer. After a hard bake at 110 degrees Celsius for thirty minutes, the wafer was etched in a buffered solution of hydrogen fluoride for four minutes to remove the top layer of silicon dioxide (at a rate of around 100 nanometers per minute), and it was then placed in a silicon deep reactive ion etcher to remove silicon to the necessary depth of between fifty and one hundred micrometers. Acetone was then used to eliminate any leftover photoresist that was present.

Embossing done using heat. The COC (Zeon Chemicals, located in Louisville, Kentucky) was cut into the necessary size after being cleaned with acetone for two minutes in an ultrasonic bath. The silicon master was brought into contact with the chip by applying heat (154 degrees Celsius) and pressure (250 pounds per square inch) from the top and bottom plates of the hot-press machine. After then, the chip and the silicon master were allowed to naturally cool down in order to alleviate the tension. To prevent cracking the silicon mold, extreme caution was required throughout the process of totally detaching the chip from the master. The Alpha-step 500 surface profiler made by KLA&Tencor was then utilized in order to estimate the depth of the channel. A drilling machine was used to create access holes at the desired place measuring between 300 micrometers and 2 millimeters in diameter. The same hot press equipment was used to apply pressure

(695 psi) and heat (123 °C) to the two pieces of plastic for a period of 10–15 minutes in order to glue them together.

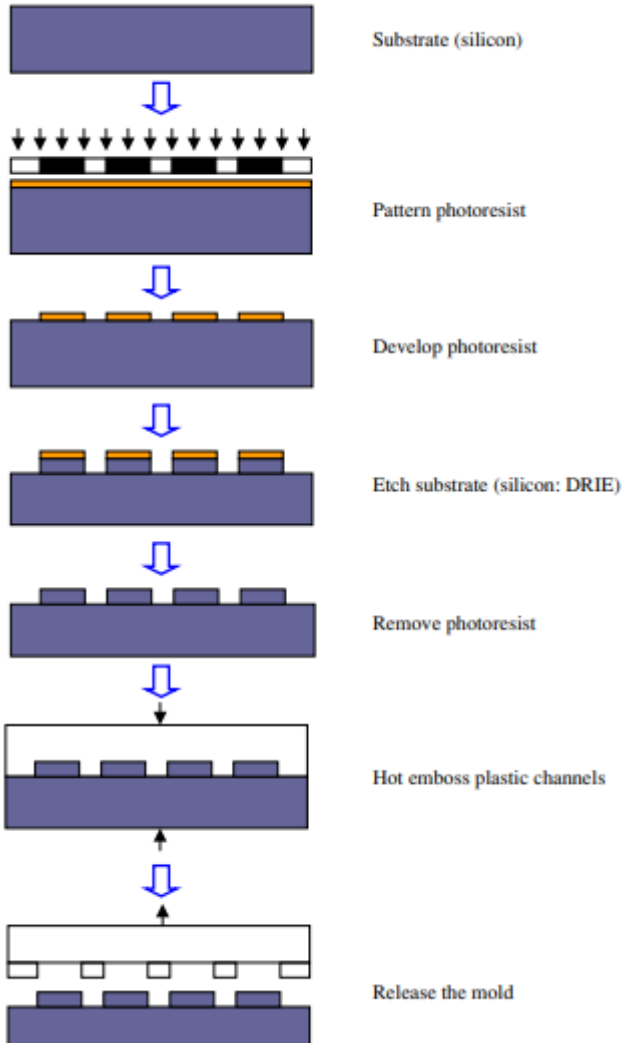

Figure 1. Process flow to fabricate a silicon mold and hot-embossed plastic devices.

In addition to the wafer-level thermal bonding, an optical adhesive was used to connect the individual bits of plastic. The photografting process, which is going to be explained in the next part, was commonly used to treat the plastic surfaces so that they became hydrophilic. A very thin layer of adhesive was spread over the rim of the device, and then it was allowed to seep into the crack. After thirty minutes of exposure to a UV light source with a wavelength of 365 nm, the bond was healed. A scanning electron microscope was used in the manufacturing of the PDMS device, which resulted in the preparation of cross-section SEM photographs. In addition, PDMS devices were manufactured by employing the same silicon mold. The PDMS base and the curing agent were combined at a ratio of 1:10, and the mixture was then degassed in a vacuum chamber until there were no visible bubbles. After that, the liquid was put into the silicon mold, and it was dried at a temperature of 70 degrees Celsius for three hours. The substrates were first washed with IPA and then oxidized in a plasma etcher (Reactive Ion Etcher 2000, South Bay Technologies) for one minute. This was done in order to bind two separate pieces of PDMS. After that, the two pieces were immediately pushed gently into touch with one another and then placed in an oven preheated to 90 degrees Celsius for ten minutes in order to ensure that they would bind.

DATA ANALYSIS

Surface modification

This diagram illustrates the fabrication method that is used to manufacture silicon master and polyolefin plastic chips. The development of silicon surface and bulk micromachining techniques led to the selection of silicon as a candidate for the role of master material. Because three-dimensional silicon molds can be easily fabricated, not only can planar structures be easily produced, but also three-dimensional topology can be easily realized as well. demonstrates the silicon mold as well as the gadgets that were manufactured utilizing the silicon mold. The anisotropic nature of dry etching makes for a great crosssection profile of the microchannels that are produced as a consequence. The surface of the polymer may be altered by exposing it to UV light, which will cause the surface to change from being hydrophobic to hydrophilic.

The static contact angle of water on a native COC, in contrast to the reduced contact angle surrounding a treated COC surface. After grafting, a hydrophilic surface that is reasonably consistent may be achieved using this approach. Over the course of one week, we kept an eye on the contact angle, and during that time we didn't notice any clear signs of aging. Other methods, such as the oxygen plasma treatment, have the benefit of allowing the hydrophilicity of the surface to be modified by altering the reaction circumstances (for example, the gas for the plasma, the power, and the time). This is one of the advantages of these methods. Even though the range of our experiments was somewhat restricted (10–20 minutes of exposure, continuous source), we did not find any variation in the hydrophilicity of surfaces as a function of UV irradiation. Recent research indicates that the contact angle may, in fact, be adjusted by adjusting the amount of time that UV radiation is present. If the UV conditions can be further optimized, it may be possible to use the UV grafting procedures to a wider range of applications.

Fluidic handling

The capillary force may be loaded onto the surface more easily as a result of the surface treatment. displays the location of the meniscus as a function of time after a drop of DI water has been put on the microchannel's intake. When compared, the fluidic behaviors of capillary-driven flow in untreated and treated COC devices, glass chips that are naturally hydrophilic, and oxygen plasma treated PDMS microchannels are all quite comparable.

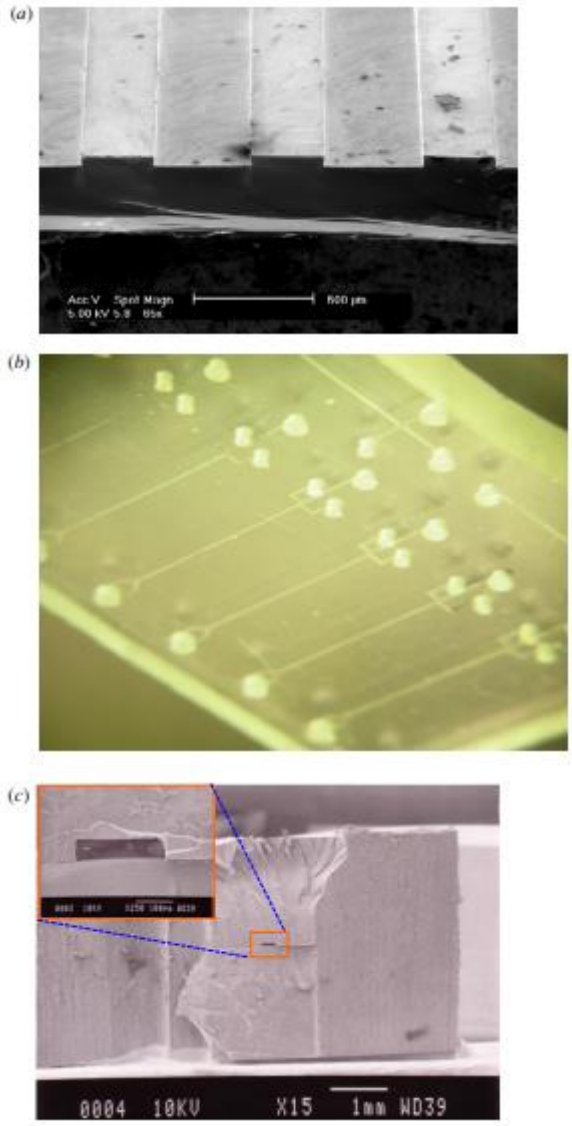

Figure 2. (a) Scanning electron micrograph of the microfabricated silicon mold. (b) Pictures of hot-embossed plastic devices. (c) Scanning electron micrograph of the rectangular cross-section of the plastic microchannel.

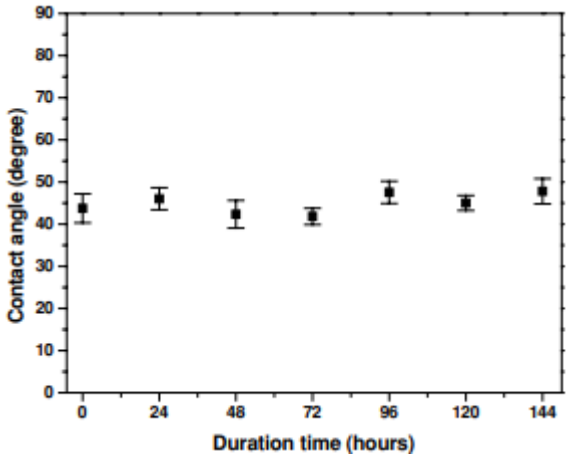

Figure 3. Contact angle of the photo-grafted COC surface as a function of duration time in the air at room temperature.

CONCLUSION

We describe in great detail the experimental setup and approach that was utilized in order to achieve surface modification. This methodology included the characterisation of changed surfaces by means of contact angle measurements, atomic force microscopy, and X-ray photoelectron spectroscopy. These investigations shed important light on the changes that are taking place at the COP surface and the effect those changes have on the behavior of the fluid. In this work, we illustrate the practical uses of air atmospheric pressure-modified COPs in microfluidic devices, demonstrating improved performance in areas such as droplet generation, mixing, and analyte capture. These enhancements have the potential to expand a variety of microfluidic applications, including chemical synthesis, drug discovery, and diagnostics. This study proposes a unique and efficient method for surface modification of cyclic olefin polymers in microfluidic devices by leveraging air atmospheric pressure as the driving force behind the process. The incorporation of this method carries the potential to improve the performance of COP-based microfluidic devices in a broad variety of scientific and biomedical applications.

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