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Rapid Prototyping for Micro-Engineering and Microfluidic Applications: Recycled PMMA, a Sustainable Substrate Material

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Abstract. Poly(methylmethacrylate), PMMA, is one of the most commonly used thermoplastics for the manufacture of micromechanical and microfluidic devices, due to its optical transparency, rigid mechanical properties, low cost and good workability in conjunction with its rapid prototyping and mass manufacturing. Recent advances in the rapid-prototyping fields have allowed the production of precise features compatible with microfluidic structures and accelerated the conversion process from bench-side to mass market. For example, to address the need for fast design cycles using material compatible with mass manufacturing, we have developed an ultrafast prototyping technique for the manufacture of multi-layer PMMA micro devices (doi:10.1007/s10404-016-1823-1) and described a method to choose the right PMMA for this prototyping technique (doi:10.3233/978-1-61499-792-4-181). PMMA is a petrochemical-derived material and the rising demand for single-use disposable devices will inevitably result into increased medical plastic waste. To address this problem at the design/prototyping stage, we explored the possibility of utilizing recycled PMMA (Re-PMMA) as the substrate material in our technique. The aim of this work is to compare commercially available recycled PMMA (Re-PMMA) with pristine PMMA (pPMMA) in conjunction with our prototyping technique. The information reported here will provide a practical guide to researchers when selecting Re-PMMA material for a more sustainable approach to micro-engineering and microfluidic rapid-prototyping.

Keywords. Rapid prototyping, PMMA, Recycled, microfluidic,design for sustainability

Introduction

Poly(methyl methacrylate), (PMMA), is a thermoplastic material widely used for producing optical fibres, and a wide range of other consumables [1, 2]. Since the late 90s, thanks to its low cost, good workability both at the prototyping scale and at the

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mass manufacturing scale, PMMA has been used as substrate material for microfluidic applications. Some microfluidic applications on PMMA substrate include the analysis of amino and nucleic acids, peptides and proteins [3]. Given that an increasing number of these biomedical microfluidic devices are produced in single-use, disposable, format, a design for sustainability (DFS) approach when designing new microfluidic devices is desirable. Briefly, a DFS approach considers the full life cycle of the final device. Starting from the raw material extraction, the DFS includes an analysis of transportation, transformation of the raw material into intermediate material, the manufacturing process both at the prototyping scale and at the mass manufacturing one, the use of the device and its possible end of life scenarios. We have recently developed a safe and cost effective rapid prototyping method for the assembly of complex 3D microfluidic devices in PMMA [4]. This method involves the use of a CO_2 laser cutter to shape the micro-channels in PMMA layers that are then bonded together using a 2 min ethanol assisted bonding method, enabling fast production cycles (15 min from design to test) (Figure 1A).

Taking a DFS approach, recycled PMMA (Re-PMMA) is proposed as a novel substrate material for microfluidic medical components. Thermoplastic materials can be recycled with different technologies including mechanical recycling, or chemical recycling. Mechanically recycled PMMA has been used for the production of optical fibre sensors [6] and microfluidic devices [7]. Despite optimum results in terms of final optical qualities, in the case of single use medical devices, a sterilization step should be included prior to mechanical recycle the device. On the other hand, Re-PMMA from chemical recycling is commercially available from different brands. In chemical recycling the used thermoplastic is depolymerized through a heating process to break the macromolecular chains. After a distillation process all the impurities are separated to obtain recycled methyl methacrylate monomer with a purity up to the 99.8%, that are polymerized to obtain Re-PMMA with the same optical, mechanical and thermal properties of the pristine PMMA, (pPMMA). In this article, chemical and physical characterisation tests are carried out to validate the suitability of Re-PMMA for microfluidic rapid–prototyping in comparison to pPMMA.

1. Materials and Method

1.1. PMMA Material

Re-PMMA sheets of 3 mm thickness were purchased from Cut Plastic Sheeting UK (100% recycled Clear Greencast Acrylic Sheet). pPMMA were purchased from Weatherall Ltd (UK) (Clarex® cast) from Easter Road Plastic LTD (Oroglass® cast) and from Engineering and Design Plastic LTD (UK) (Perspex®)

1.2. DSC Analysis

A DSC 2010 (TA Instruments) was used to measure thermal properties of Re-PMMA and p-PMMA (Clarex®, Oroglas® and Perspex®) in order to reveal the presence of impurities. 14 mg of each sample were placed in a 30 μl aluminum pan and heated at a rate of 10°C/min from 30 to 250°C. All tests were carried out in triplicates.

1.3. Laser Set-up

A commercial CO_2 laser cutter Epilog Mini 18 (30 W) from Epilog was used. The laser speed can be adjusted from 0 to 100%, where 100% corresponds to actual speed of 85 mm/s. The laser power can be adjusted from 0 to 100% corresponding to actual power of 30 W. The software CorelDraw (Corel Corporation, Canada) was used to transfer drawings to the laser cutter.

1.4. Re-PMMA Bonding

To bond together the different PMMA layers, absolute Ethanol (%w > 99.95%w, Sigma-Aldrich, UK) was used as a solvent. A custom-made heating plate maintained the layers at the correct temperature during bonding. As a source of pressure, we used a Bonny Doon Classic 20-Ton Manual Press (Rio Grande, USA). After laser cutting the desired shapes using a CO_2 laser cutter (Epilog Mini 18, Epilog, USA) PMMA layers were cleaned with clean-room tissue and ethanol to remove dust. 10 $\mu l/cm^2$ of ethanol were then spread between each layer before bonding at 70°C, 5 tons at the ram (corresponding to 15 bar) and for 2 minutes.

1.5. Shear Stress Analysis

The shear stress testing has been described in details in [4]. Briefly, standard tensile test equipment (Instron 3367, Instron) was used with an elongation speed of 0.02 mm/s and recorded the highest load to date before sample break. The two sides of the dumbbell specimens were laser cut from 3.0 mm thick sheets and then bonded together according to section 1.4. Six specimens were used for each set of parameters to ensure reproducibility of results. Statistical analysis was carried out with a Student's t-test.

1.6. Burst Test and Maximum Flow rate Analysis

In order to assess the quality of the bonding method on the Re-PMMA and the ability to effectively seal the channel, burst test analysis were carried out. Chips with a 300 μm wide channel leading to a dead-end were manufactured sandwiching two 3 mm layers, one of which served also for the fluidic connection. A syringe pump (Alladin, WPI), was used to actuate flow of a (5mg/mL) fluorescein solution within the specimens at 2 mL/min. As there is no way out, the air enclosed within the channel is gradually forced in a smaller volume and its pressure increases. Pressure changes were monitored with a pressure sensor (uPS 1800-T116, Labsmith). The same set-up was used in open-end chips, changing gradually the flow rate (from 5 to 300 mL/hr) to characterise the maximum flow rate allowed within the channel.

1.7. Kerf Width and Kerf Depth Characterization

Kerf width and kerf depth measurements were carried out cutting 1 cm slits in 2 mm thickness PMMA sheets from each supplier. Laser power, speed and frequency were chosen as input parameters to find the minimal features achievable in terms of kerf

width, and the optimum parameters to completely cut through the sheet. All the measurements were conducted via image analysis on ImageJ (version 1.51r) from photographs acquired with a Dino-Lite Edge digital microscope (AM4115T-GFBW, DinoLite, Taiwan). For each measurement, at least 3 readings were taken.

2. Results

2.1. DSC Analysis

Differential Scanning Calorimetry is a powerful tool enabling the understanding ofmaterial thermal behavior in terms of crystallization, melting and glass transitions. PMMA, as an amorphous polymer, does not have any crystallization phase. The resulting DSC thermograms should theoretically show only one glass transition phase and a melting region. If a thermoplastic polymer shows more than one glass transition phase, this phenomenon could be due or because of the composite nature of the polymer itself or because it is under nanoconfinements [8]. In a previous study [9], we have shown using DSC that the presence of impurities inside the material can affect the bonding strength. In Figure 1A the thermogram of Re-PMMA does not shown any additional phase transition, Similarly Perspex cast do not show any secondary peaks. On the contrary, both pPMMA Clarex® cast and Oroglas® show a secondary Tg. The associated energy to this additional phase transition in Oroglas® is 7.8 J/g, 3 times bigger than its first transition phase and four times larger than Clarex® second peak and was previously associated with lower bonding strength as shown in Figure 1B [9]. These results show that Re-PMMA can be adopted in conjunction with our bonding protocol.

2.2. Bonding Strength

Accordingly to the DSC results, Re-PMMA bonding strength is as comparable to the bonding strength previously reported for Clarex® and Perspex® materials (Figure 1C) [9], During the shear stress tests not all the specimens were achieving delamination and some of them were arriving at break before the beginning of the delamination process.

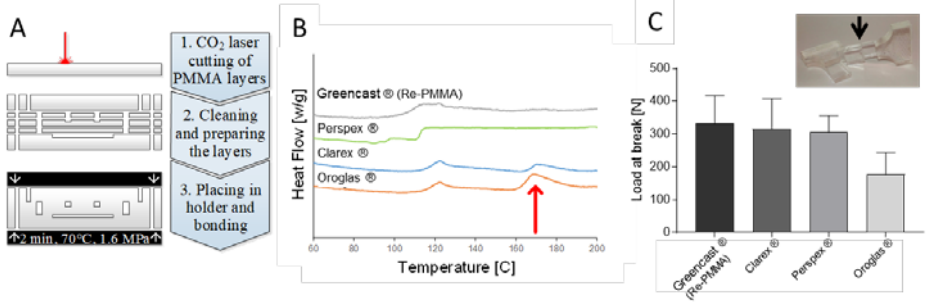

Figure 1. A) Overview of PMMA rapid-prototyping technique described in [4]. We propose a sustainable alternative by replacing pristine PMMA by Re-PMMA B) Thermograms of the Re-PMMA, Perspex®, Clarex® and Oroglas® ; C) Maximum bonding strength of Re-PMMA, Clarex®, Perspex® and Oroglas® ; In Inset, Photograph of the broken specimen (Re-PMMA). The black arrow points where the material break, showing the other part of the specimen still bonded.

2.3. Burst Test and Maximum Flow Rate

To determine the failure modes of our Re-PMMA prototyping technique, we determined the critical pressure and the maximum flow rate at which the microfluidic devices started leaking or delaminating. No delamination phenomena were noticed in all the devices tested. The maximum operative pressure was recorded to be about 528 kPa, no leaking from the connectors or delamination were observed, but a back pressure was noticed at the syringe pump and recorded from the set-up used as a pressure drop, as reported in Figure 2B. This underlining the robustness of the bonding method. The maximum operative flow rate recorded was 300 mL/hr (Figure 2B); above this, leaking of the fluorescein solution was observed from the microfluidic connectors.

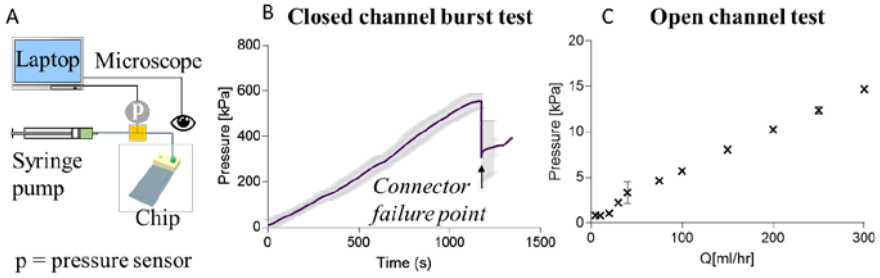

Figure 2. A) Experimental set-up; B) Burst test in close channels (width 250 µm). Maximum pressure difference in function of flow rate. Deviation is shown in grey; C) Open channel tests. Input flow rate in function of pressure. 300mL/h was the pump maximum flow rate. For some points the error bar is shorter than the symbol.

2.4. Laser Cut Characterization

We have investigated the influence of the CO_2 laser power and speed to the kerf width and depth. In general, a lower speed allows results in a larger kerf width. On the other hand, at higher speed, deposited energy is reduced, resulting in a smaller kerf width. On the contrary, higher power result in larger and deeper cut with respect to a lower power. The smallest feature achievable was recorded to be 100 µm of kerf width and depth, using a laser power of 3 W and a speed of 85 mm/s (the maximum operative speed) using a 1.5" lens and focused onto the material surface.

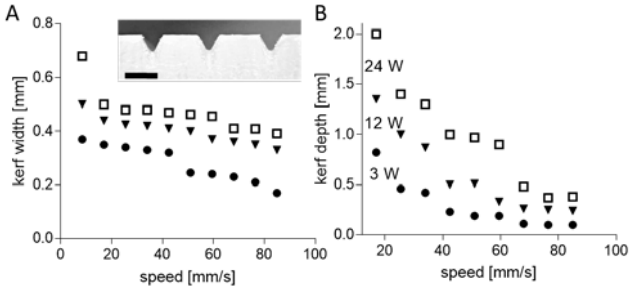

Figure 3. (A) Influence of laser power and speed on the kerf width (B) kerf depth. In insert a cross section picture of a channel engraved with a power of 12 W and a speed of 15 mm/s. scale bar is 500 µm.

3. Conclusions

The purpose of the current study was to validate the use of a commercially available chemically recycled PMMA in conjunction with a rapid prototyping technique to manufacture microfluidic devices sustainably. A DSC analysis did not show the presence of impurities inside the material, suggesting the suitability of Greencast™ PMMA material with our bonding protocol, without optimisation of the bonding protocol. Several other manufacturers of chemically recycled PMMA were contacted for samples but none supplied material. Although it can be anticipated that other chemically recycled material may also be suitable for prototyping microfluidic devices, a DSC analysis is recommended before embarking in manufacturing. At the time of writing, Greencast™ is more expensive than pPMMA, however, it is a more environmentally sustainable approach when embarking in the production of microfluidic devices for research, teaching and public engagement activities

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