CUSTOMISED LAYER DEPOSITION FOR CHEMICAL REACTOR APPLICATIONS

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Abstract

This paper discusses the development and application of an adaptive slicing algorithm for use with Digital Light Processing (DLP) for the manufacture of micro chemical reactors. Micro reactors have highly complex constructions and DLP has a proven ability to deliver features at the micro level with high accuracy. However, DLP fails to provide a truly smooth profiled surface finish which could influence fluid flow through entrance and exit apertures and along snaking micro channels. Ensuring smooth surfaces will minimise energy losses in the fluid flow path. Generally, layer based manufacturing techniques incur a trade off between build time and resolution. The algorithms used in this study attempt to mitigate this to some degree by calculating locations where high resolution is required through surface profiling techniques and adjusts the layer thickness accordingly. It is proposed that this adaptive layering technique may improve surface roughness and reduce friction related energy losses along micro channels within chemical reactor applications.

Introduction

Micro reactors are devices used for heat and mass transfer. This is achieved by bringing two fluids in direct or indirect contact in confined “micro channels” that essentially have lateral dimensions less than 1 mm. The designated term “micro” comes from the manufacturing techniques originally used for microelectronic fabrication. Due to their ability to manufacture micro features consistently, it is now possible to integrate sensors and actuators into these devices. Through the integration of multi functions and a reduction in size, micro reactors have become highly efficient and cost effective.

Micro reactors are frequently manufactured by various techniques such as “Deep Reactive Ion etching” (DRIE); the so-called “Lithographie, Galvanoformung, Abformtechnik” (LIGA) technology; micro-electro-discharge machining (μEDT); rapid prototyping; and micro-injection moulding, etc. Irrespective of the particular manufacturing technique, the basic construction of micro reactors consists of elements such as, inlet ports, outlet ports and micro channels, which define pathways for fluid flow. In case of conventional reactors, channels are usually circular in cross section as they can withstand high pressure, but for micro reactors the cross section of the channels can be either circular or square.

The flow of fluids in a duct is measured by the Reynolds number, “N_{Re}”, which is dimensionless and is the ratio of inertial forces ($V\rho$) to viscous forces (μ/L) in a fluid:

$$N_{Re} = (V\rho\, L)/\mu \qquad (1)$$

where V is the mean fluid velocity (m/s), ρ is the density of fluid (Kg/m^3), and μ is the dynamic viscosity (Pa.s).

Equation (1) can be modified for flow through a circular pipe:

$$N_{Re} = (V\rho\, L)/\mu = VL/\nu = QD/\nu A \quad (2)$$

Where ν is the kinematic viscosity, $\nu = \mu/\rho$ (m^2/s), Q is the volumetric flow rate (m^3/s), A is the cross section area of the pipe (m^2), and D is the Diameter (M).

For non-circular channels, which are described by major and minor dimensions, the "hydraulic diameter, D_h" becomes applicable. The hydraulic diameter is defined as four times cross sectional area of the flow path, divided by the wetted perimeter:

$$D_h = 4A/P \quad (3)$$

where A is the cross section area (m^2) and P is the wetted perimeter (m).

In theory, the Reynolds number for the laminar flow regime ($N_{Re} < 2300$) is where viscous forces dominate the flow behaviour and the fluid experiences shear losses. However for $N_{Re} > 2300$, a turbulent flow regime exists, which is dominated by inertial forces which tend to produce eddies and rapid fluctuation.

Fluid flow in micro reactors occurs mostly in the laminar regime because of the low accumulated volume of micro channels. For the design of reactors, N_{Re} is calculated based on flow rate of the fluids; the aspect ratio of the channels; and the physical properties of the fluids. Loss of energy while flowing through these channels is defined by a friction factor "*f*" which is a function of N_{Re}. Referring to the Darcy friction factor, the magnitude of friction is inversely proportional to N_{Re} for the laminar regime. However for turbulent flow, the friction factor depends upon the ratio of surface roughness (ε) to the flow diameter (D).

Consequently for laminar flow ($N_{Re} < 2300$) it is apparent that the friction factor does not depend upon the flow diameter. Similarly, for $N_{Re} > 2300$ (i.e. turbulent region) the friction losses in the fluid depend on the surface roughness. As micro reactors have low accumulated volumes, we can assume that micro reactors are operated in laminar regimes. However, various investigations of fluid flow in micro channels have reported deviations from the theoretical behaviour [1]. The transition of laminar to turbulent flow has been reported to occur as early as $N_{Re} < 800$ and completely changes into the turbulent regime starting at N_{Re} in the range of 1000 - 1500.

In this case, it is important to notice that the surface roughness in the micro channels is significant with respect to the flow diameter. Furthermore, occurrence of the turbulent flow regime at relatively low values of N_{Re} results in further losses due to formation of eddies [2]. Behaviour of fluids at lower N_{Re} therefore becomes highly complex and the experimental results show deviation from the theoretically expected values.

Because of these observations, we are stimulated to investigate possible solutions to this problem. Here we propose that modifying the manufacturing process can bridge the gap between the theoretical and experimental performance of the micro reactors. In this work, Digital Light Processing (DLP) based manufacturing has been exploited. DLP is based on the Texas Instruments digital micro mirror (DMD). DMD is a micro electromechanical (MEMS) device containing a matrix of micro mirrors. Each one can be individually used as a reflector to define an image pixel and to achieve high resolution projection for stereo lithographic fabrication.

The surface roughness of parts created using DLP techniques varies greatly depending on how the surface is orientated relative to the build direction. The top and bottom surfaces, which are parallel to the glass substrate, can be significantly smoother than faces perpendicular to the substrate; which often suffer from surface irregularities caused by the very nature of building components in layers. If surfaces are angled relative to the substrate than the problem can exacerbate by a phenomenon known as the 'stair step' effect. In order to achieve high fidelity three dimensional builds of micro reactor devices, a number of software tools have been developed which recognise features such as flat planes and changes in contour profile of the STL CAD file and adapts the position and thickness of layers accordingly. For this work to be successful it is important that the developed slicer has at the very least a comparable resolution to that of the DLP system.

Software Development

To create a part using the DMD masking technique an STL file which describes the 3D solid model is firstly mathematically sliced and each slice converted into a 2D greyscale image mask (Bitmap image). The Perfactory software suite generates then assembles all the layer masks, along with an XML document containing system and build parameter settings, into a convenient JOB file (a disguised ZIP file). The pre-processed JOB file can then be transferred into the Perfactory system for construction. The software facilitates the generation of slice data with minimal control of system parameters and with no control over layer mask generation. From a user perspective the Perfactory software is simple to use. However, the challenges set out in the work discussed in this paper requires a greater understanding of how system parameters and image masks influence hardware performance and part quality; with a view to improving accuracy, resolution and surface roughness of fine features. Previous work at Liverpool has shown that reducing the layer thickness has a significant effect on increasing DLP surface quality [3]. However, this work was limited in layer thickness range between 25 and 100 microns. This range was constrained by the Perfactory software. The software developed in this work will enable experiments to expand on previous work by constructing test pieces with a user defined value of layer thickness and with building parameters adjusted accordingly. This section describes the development of software tools to create a JOB file; including adaptive STL slicing (FS Slicer), image mask generation and the construction of the parameter settings file.

The slicing algorithm reported in this work has evolved from a machining algorithm based on CAM-style slicing with a defined tool shape. This code (written in C++ and Python) has been discussed before but some recent modifications give reason for further discussion here [4]. Conventional CAM requires the specification of a tool geometry; a cylindrical shape with a torus, spherical or flat tool tip. The FreeSteel slicer (FS-Slicer) divides this tool shape into several parts: a tip (sphere, torus, or flat disk), a shaft (cylindrical or conical) and optionally any number of cylindrical or conical holder shapes. The shaft diameter is not restricted to the tip diameter; a shaft smaller than the tip, for example, can be used to model lollipop cutters. To start, all triangle objects contained in the STL file that describe the surface geometry are read into a single array. Each triangle object has three pointers to edge objects and triangle normal. Edge objects point to two end points and up to two adjacent triangles (it is then possible when looping over all triangles to avoid using the same edge twice or when looping over edges to consider the same triangle multiple times (maximum of 3).

The slicing algorithm is then given a *z* height at which contour data points are required (See Figure 1a). The algorithm then builds a weave (grid) at the given z height which is constrained by an area slightly larger than the bounding box of the cross section of interest (see Figure 1b). Contours, or tool paths, are then calculated for the tip centre by passing the tool along the weave lines. When the tool shape touches the contour of the STL the contour point is recorded. Triangle objects are grouped into containers so that when calculating a cutter position only the triangles that belong to the containers covered by the cutter shape need considering (prevents the need for looping over all triangles).

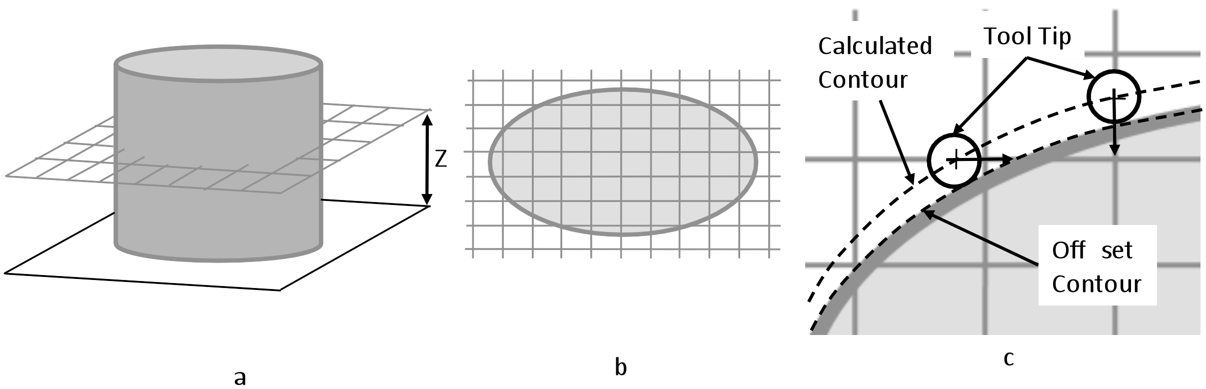


Figure 1: (a) location of slice height in z direction, (b) slice contour and weave and (c) calculation of contour data with tool tip.

When a shaft height is specified the algorithm will continually ‘look up’ for triangles above the slice height that would otherwise interfere with the tool shaft; preventing any contour data collection of undercuts (This is a desired feature of 3 axis tool paths). Hence, for the purpose of slicing for use with SFF technologies, shaft height needs to be set to a value equal to or lower than the value of the layer thickness.

The resulting contours have to then be processed through two steps: (1) Unwanted contours "inside" the volume modelled by the STL have to be filtered out (the algorithm would pass the tool along both sides of the contour because any triangles above the tool tip will not interfere with this tip) and (2) an inward offset equal to the radius of the tool tip has to be applied because contours are referenced to the centre of the tool (see Figure 1c). As the tool marches around the weave the algorithm also records contour type according to the right hand rule of vector orientation i.e. whether the contour describes a core or cavity. Once the tool has passed over the entire weave, a new *z* height is selected and the process repeats.

The resolution of the contour data is governed principally by the resolution of the weave and this is defined by the user. The size of the tool tip determines how efficiently the tool can navigate small features and internal corners. In this work weave resolution was set to 0.01mm and a flat disk for the tool was selected with a radius of 0.05mm. These parameters were chosen based on a trade-off between speed of slicing and resolution; maintaining a JOB file generation time equally comparable to the Perfactory software suite. It is also worthy of note here that a larger tool tip can be impervious to breaks in STL triangulations. It is quite common to use propriety software to fix holes in STL files before they are sliced. Using a tool tip with a defined area prevents the cutter from 'falling' through holes in the STL geometry and sustains contour point calculations.

There are also a number of additional software features that have been utilised in this work to improve build accuracy/resolution and/or reduce build time. The first is a flat planes algorithm that detects the *z* height position of all horizontal surfaces (see Figure 2a). This is done by looping over all triangle objects and comparing the normal's (which are cached) with a user defined slope parameter. This is done to ensure contour data is collected close to these regions. It should be noted that a slice cannot be taken exactly at the position of a flat plane because the tool will be approaching the triangles from side on. The second is an adaptive slicing algorithm. The algorithm works in one of two ways. Either, layer thickness value can be defined for a given z height range or the layer thickness values can be automatically modified with changes in the geometric profile of the STL file (see Figure 2b). The latter works by calculating the largest step value between the current layer *zhi* and the layer below *zlo* (see Figure 2c). If the step change is greater than a user defined threshold value then a second slice is taken half way between *zhi* and *zlo* and the calculations repeat. The iterations continue until the maximum step change equals or falls below the threshold value. All contour data for all layers is stored within a single dictionary where keys (z height and contour type) point to the data point arrays.

Once all contour data has been collected the contour point arrays are passed into an imaging function to create the image masks for each layer. The first step is to scale the contour data according to the resolution of the DMD processing chip and the size of the projected area on the Perfactory build platform (see Equipment and Experimental Methods Section). A PNG image equal in resolution to the DMD chip is then created (1400 x 1050 pixels) and all pixels are initialised to black (0,0,0). The scaled contour points for each layer are then used to construct a series of filled polygons, the colour of which is determined by contour type in each layer (core = black, cavity = white). Since each polygon is drawn on the image in layers it is important that the larger contours are drawn first. Each sliced layer is centralised on the PNG image. Hence, the software currently has no capability to nest multiple parts in one build operation.

Image masks created using the FS-Slicer software were comparable with image masks created using the Perfactory software suite. However, one noticeable difference with the FS-Slicer generated images was the absence of a border, one pixel in width, that surrounded each layer silhouette (see Figure 3). However, the existence of the border in Perfactory generated layer masks was not prevalent and its greyscale intensity (ranged between values of 20 and 200)

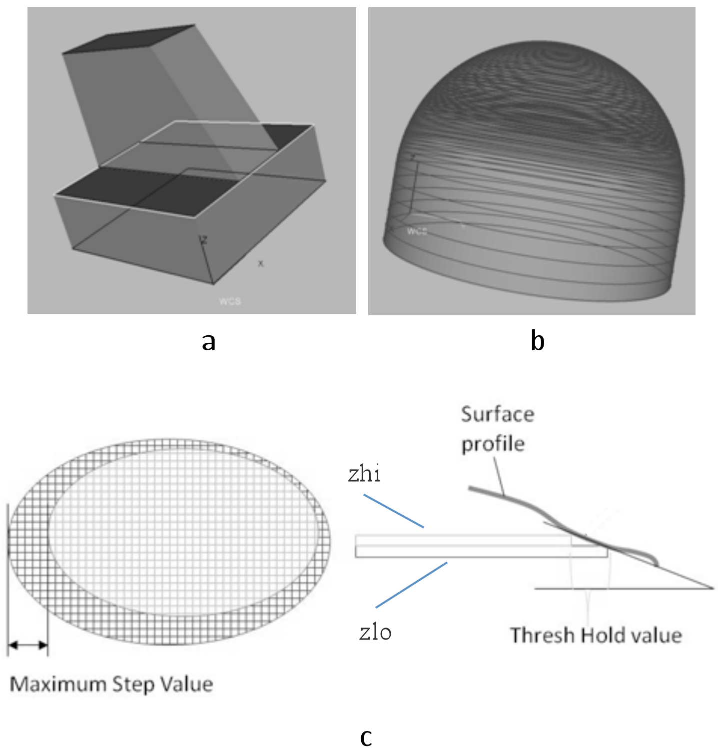


Figure 2: (a) detection of flat planes, (b) adaptive slicing and (c) calculation of slice height in adaptive slicer.

changed in a perceived random manner when viewing a number of different image masks. This suggested on first inspection that the border may not be intentional; perhaps a consequence of some image scaling technique that implemented anti-aliasing methods. For this reason no attempt was made to recreate a border in image masks produced using the FS-Slicer software. Further thought is given to this phenomena in the Discussion and Conclusions Sections of this paper.

To engineer a fair comparison test it was also important to be aware of the Enhanced Resolution Mode (ERM) implemented by the Perfactory system to improve part resolution and surface roughness; the mechanism by which improvements are sort was observed but not fully understood by the authors at the time of writing this paper. When ERM mode is active each sliced layer is processed twice. Each repeated layer is digitally offset from the first by shifting the image by one pixel in both x and y directions (observed by examining consecutive image masks); an analogy would be building a brick wall where bricks in adjacent layers are staggered. It is also worth noting here that the border phenomenon discussed previously often has a higher intensity in the second image mask. The ERM methodology was implemented in the FS-Slicer software in this work.

Once all image masks had been generated the FS-Slicer software saved each mask into a JOB file along with an XML file that contains all machine and layer parameter settings. The parameter settings chosen for the experiments discussed in this paper are discussed in Equipment and Experimental Methods Section.

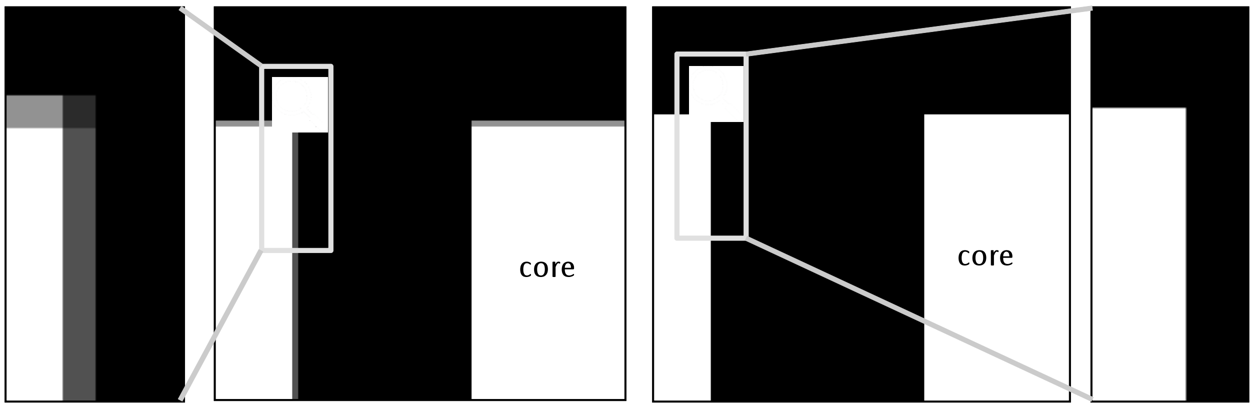
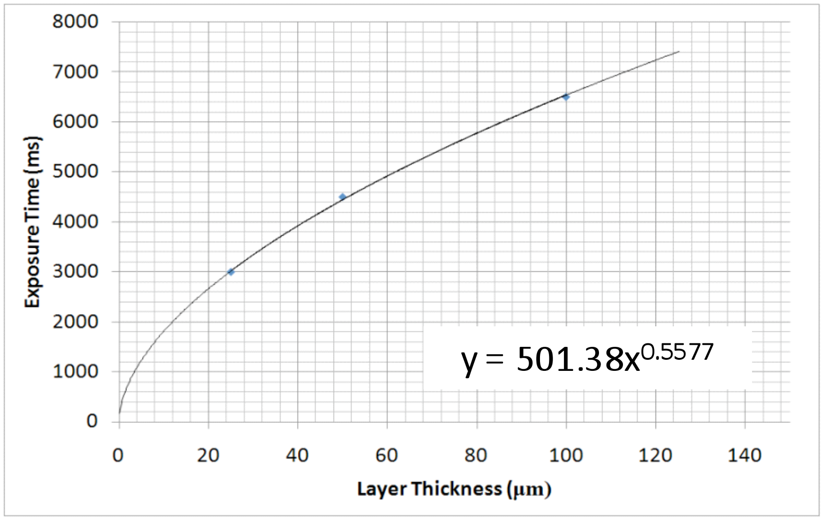


Figure 3: (a) Perfactory Image mask showing greyscale border and (b) FS Slicer Image masks showing no border.

Equipment and Experimental Methods

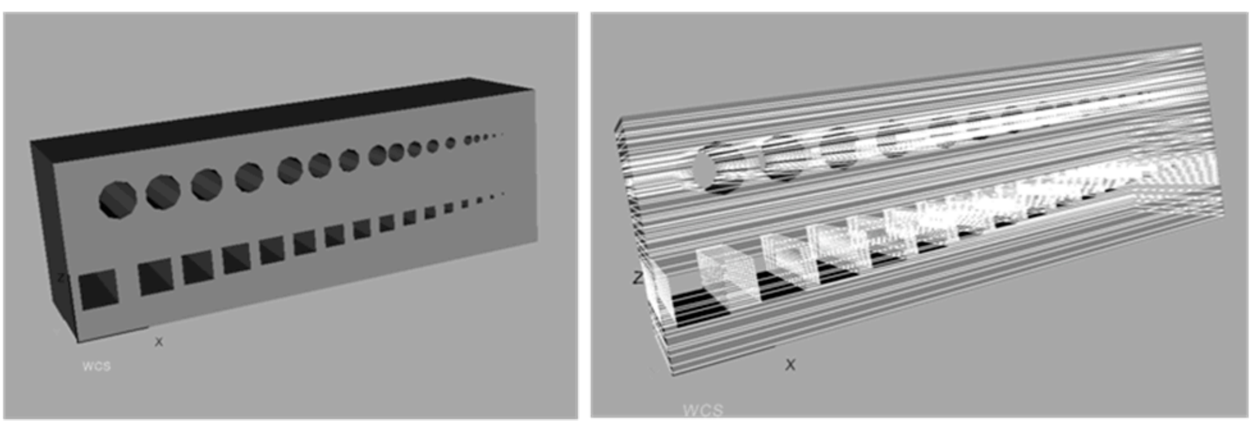
Fabrication of test specimens containing square and circular micro channels was done using a Perfactory Mini DLP apparatus supplied by Envisiontec, Germany. The apparatus utilised a DMD masking technique for creating a successive number of layer masks (bitmap images) to define repeated layers of a controlled geometry by selectively photo-polymerising a liquid resin layer by layer. Parts realised in this way can be thought of as containing a large number of elemental voxels (volumetric pixels). However, the resolution of each voxel is anisotropic. This is because resolution is principally controlled in the x-y plane by the density of mirrors on the DMD chip and the focussing optics. In the z direction, resolution is governed by the stepper motor controlling the build height of the platform. In this work, projected light from a DMD chip, with a mirror density of 1400 x 1050, was focused using the Perfactory SXGA+ 85mm optics onto a build platform of size 27.77mm x 20.83mm. This gave a voxel resolution of 20 microns in the x-y plane. The resolution in z is set by the layer thickness. The stated accuracy of the system is ±7.5 microns. These values do not take into account any material volumetric changes during polymerisation. An incandescent bulb is used as the light source. This emits a spectrum which is filtered to appear green (approximately 510nm) at the build surface. The manufacturer regards the specific wavelength to be proprietary knowledge and therefore further information is not disclosed. Greyscale can also be implemented in image masks which causes the mirrors on the DMD chip to rapidly fluctuate between their on and off states giving reduced exposure per unit time.

The photopolymer used in this study was an acrylic resin; Pentaerythritottri/tetra-acrylate (CAS 4986-89-4) + 1,1,1 Tri- hydroxymethylpropyl - triacrylate (CAS 15625-89-5, 3524-68-3). It is otherwise known as PMMA and is supplied by Envisiontec using the pseudonym R5 – a general purpose building material. The machine parameter settings as recommended for this material were used in this work. The only exception was the time of exposure (T-Exp) which was interpolated from Perfactory recommended settings using a power function for 25, 50 and 100 micron layers (see Figure 4). Gold coating of the PMMA components was done by evaporation using an Edwards S150 coating system. Surface roughness was evaluated using a Veeco Wyko NT3310.



Parameter	25µm Layers	50µm Layers	100µm Layers
Burn in range exposure time (ms)	7000	10000	12500
Standard range exposure time (ms)	3000	4500	6500
Projector Brightness (mW/dm^2)	530-580	530-580	530-580

Figure 4: Calculation of Exposure time interpolated from Perfactory recommended values.



a b

Figure5: (a) Square / Circle channel block and (b) Square / Circle channel block showing variable layer thickness.

A series of cubes of size $10mm^3$, each differing in layer thickness (15, 20, 25, 50 and 100 microns) and by the method in which image masks were generated (Perfactory with ERM on and Off and FS Slicer with ERM on and off), were chosen to investigate surface roughness. ERM is discussed in greater detail in the Software Development section of this paper. Average surface roughness values were measured on faces perpendicular to the build direction. A second series of blocks, of size 24x6x8mm, were also constructed which contained a number of square and circular channels which varied in square side length / diameter from 0.1mm to 1.6mm (see Figure 5). The channel size and shape were representative of typical micro reactor channels and were built to investigate shape integrity, accuracy and surface roughness. To speed up the build process the layer thickness remote from the channels was set at 100 microns and was reduced to values of 15 and 20 microns in regions local to the channels (see Figure 5b). Building the test specimens in this way reduced the time of build by several hours. Post manufacture cleaning of all parts was undertaken for 15 minutes in an isopropanol bath before secondary curing took place in air for 6 hours.

Results and Discussion

An Envisiontec Perfactory DLP system has been used to successfully construct test specimens to investigate surface roughness and build accuracy for square and circular cross section micro channels. Image masks which contain the silhouette of the cross section of each sliced layer was created using software tools shipped with the Perfactory system (PF-Slicer) and software tools written at the University of Liverpool (FS-Slicer). Dimensional analysis on the constructed test samples (FS samples and PF samples) verified the relative accuracy of the two methods for preparing JOB files.

The results from surface roughness experiments are summarised in Figure 6. Cubes of size $10mm^3$ were constructed using experimental conditions discussed in the previous section. As expected, for vertical walls parallel to the build direction, the surface roughness was generally improved with ERM mode active and for a smaller layer thickness. Previous work at Liverpool suggested that the combination of more pronounced voxels and deeper voids caused increased surface roughness when ERM is not active. These differences are likely caused by changes in the polymerisation process and the micro shift in light location as the second exposure occurs [3].
There were however some notable new observations that have fuelled a revised philosophy. Firstly, the surface roughness of vertical walls in parts produced using the FS-Slicer was less dependent on ERM mode. This is particularly noticeable at a 100 micron layer thickness where ERM off gave a significantly better surface finish. Furthermore, for every cube built, the surface roughness was smoother in FS samples than equivalent PF samples. The absence of a greyscale border around image masks created using the FS-Slicer is the likely cause. This suggests that the reduced exposure intensity per unit time at part edges, which gives rise to less pronounced voxels at these locations, actually increases surface roughness in vertical walls. This may also account for the greatest mismatch in surface roughness between ERM modes and slicing techniques at larger values of layer thickness. The micro shift in light intensity between consecutive layers when ERM mode is active may also exacerbate the problem. Although the latter remark is speculative at this time.

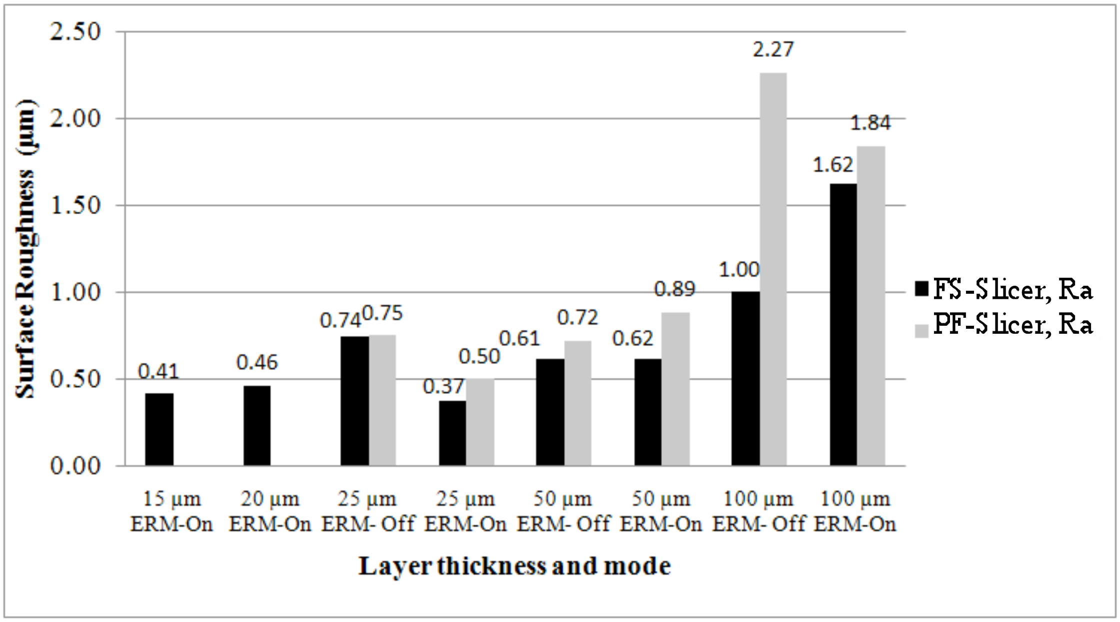


Figure 6: Root mean squared (Ra) surface roughness values for vertical walls built using DLP.

Figure 6 also suggests that no benefits can be gained when attempting to improve surface roughness by reducing the layer thickness below 25 microns. Moreover, with further reductions in layer thickness, from 20 to 15 microns, the surface roughness appeared to deteriorate further. Two possible reasons have been identified. Firstly, the mechanical positional accuracy of the build platform (±7.5 microns) becomes more significant as the layer thickness reduces. Secondly, the calculated values of exposure time were interpolated from only three data points which is likely to give inaccurate results. This may also explain surface discontinuities observed on part surfaces which are a likely cause of residual stresses from overexposure of thin layers.

Figure 7 and Figure 8 show microscope images of typical examples of square and circular channels built using the DLP apparatus respectively. In accordance with the discussion above, the square cross section channels exhibited improved shape integrity when built with image masks generated using the FS-Slicer. In general, the square channels displayed a uniform cross section throughout their vertical wall height which improved with reducing layer thickness. This included increased squareness in channel corners and fewer surface imperfections when visual comparisons were made with PF created samples.

In contrast, the circumference of circular channels were found to be highly irregular and the perimeter contour extremely pixelated. To put this observation into context, PF samples constructed with channels with the largest of diameters (>0.6mm) and with the smallest layer thickness (25 microns) could only be considered at best as "near to circular". In comparison, circular channels constructed using the FS-Slicer software were significantly worse, demonstrating a total breakdown in the integrity of the perimeter profile for all values of layer thickness investigated (see Figure 8). This somewhat disappointing result has again been attributed to the missing greyscale border in the FS-Slicer image masks. It was speculated that the reduced exposure intensity per unit time at part edges acted to smooth (or the more likely

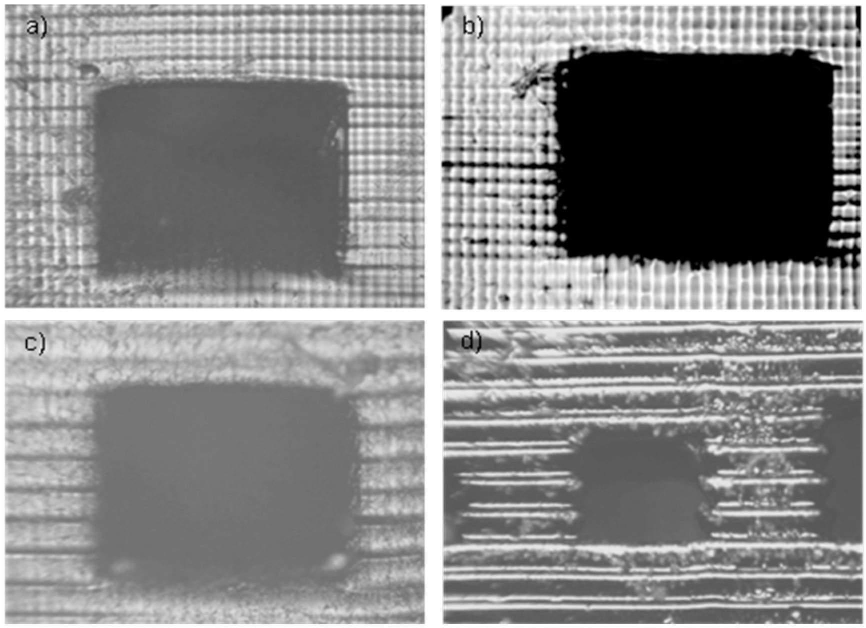


Figure 7: Micrographs showing square channels with a wall length of 300 microns constructing using (a) FS-Slicer @ 20 micron layer thickness, (b) PF-Slicer @ 25 micron layer thickness, (c) PF-Slicer @ 50 micron layer thickness and (d) PF-Slicer @ 100 micron layer thickness.

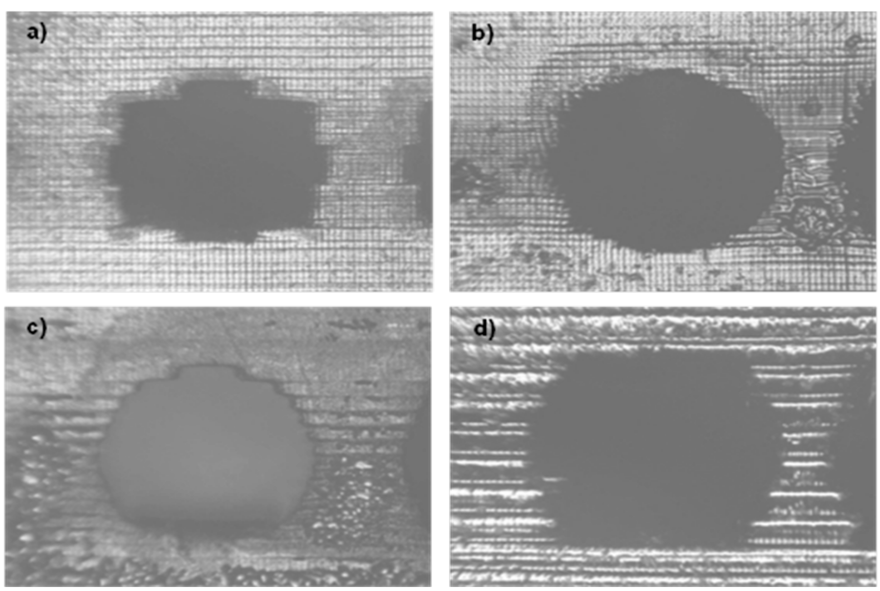


Figure 8: Micrographs showing circular channels with diameter 600 microns constructing using (a) FS-Slicer @ 20 micron layer thickness, (b) PF-Slicer @ 25 micron layer thickness, (c) PF-Slicer @ 50 micron layer thickness and (d) PF-Slicer @ 100 micron layer thickness.

to "blur") the stair step phenomenon associated with inclined or circular contours since voxels become less pronounced. It was also speculated that the image shift associated with an active ERM mode, causing a micro shift in light intensity, aided smoothing.

Conclusions and Future Work

The absence of a one pixel wide grey scale border around the perimeter of layer masks (for both ERM modes) has been shown to give improved surface roughness in vertical walls built using DLP. Further improvements could also be made by reducing layer thickness although the selection of an appropriate exposure time becomes dominant at very small values (>25 microns). Circular channels generally exhibited pixelated perimeter profiles which only started to show good shape conformity when the ratio of channel diameter to layer thickness exceeded a value of 24 (there is scope for refinement of this value with more experimental data). This value was reached using a combination of an active ERM and a grey scale border. At the time of writing this paper the individual role played by these two enhancement techniques was not clear or which, if any, were the more dominant. The apparent arbitrary nature to the level of grey in the border regions compounded this lack of understanding and therefore gives rise to an essential area of further research. What is clear from the research is the need to use ERM and a grey scale border to improve fidelity of inclined and profiled surfaces – features which are inherent in micro reactors. Because circular micro channels were not faithfully reproducible in this work using DLP, investigations of fluid flow behaviour through them was not justifiable. The authors however believe there is opportunity to improve shape reproduction in DLP through modifications to the image masks and processing method. This solicits the question for the need of a 'rich adaptive slicer' in which layer masks, layer thickness and ERM are actively changed with changes in contour profile of the STL model.

Acknowledgements

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