A novel microfluidic system for the mass production of Inertial Fusion Energy shells

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Abstract. A system which can mass produce millimetre sized spherical polymer shells economically and with high precision will be a great step towards the Inertial Fusion Energy goal. Microfluidics has shown itself to be a disruptive technology, where a rapid and continuous production of compound emulsions can be processed into such shells. Planar emulsion generators co-flow-focus in one step (COFON) and cascaded co-flow-focus (COFUS) enable millimetre compound emulsions to be produced using a one or two step formation process respectively. The co-flow-focus geometry uses symmetric and curved carrier fluid entrance walls to create a focusing orifice-minima and a carrier flow which aids movement and shaping of the dispersed fluid(s) towards the outlet, whilst maintaining operation in the dripping regime. Precision concentric alignment of these compound emulsions remains one of the greatest challenges. However steps to solve this passively using curved channel modulation to perturbate the emulsion have shown that rapid alignment can be achieved. Issues with satellite droplet formation, repeatability of the emulsion generation and cost are also addressed.

1 Introduction

Inertial Fusion Energy IFE has shown promise since KMS Fusion’s endeavours in the early 1970’s[1]. In order to get a commercially viable system, challenging technical hurdles need to be overcome such as the development of high-power high-repetition driving schemes e.g. LASERs, target injection with tracking and beam-steering systems[2] among many others. Another key issue is the economical mass production of high quality spherical polymer shells. The study discussed herein describes improvements in the latter area where a continuous production of emulsions are processed into such shells using microfluidics. Current methods use a batch process for formation and polymerisation of shells which then undergo extensive sorting and lengthy characterisation to find a shell which meets the exacting specification[3]. Because of the stringent metrology, the associated costs are extensive so an extremely reliable production method, where individual characterisation is unnecessary, is the ultimate goal. One can exploit the characteristics of microfluidic flow, for example the low Reynolds numbers that are commonly observed occurring far below the turbulent flow threshold of (Re ≈ 2000) enables laminar flow (Figure 1). Furthermore a high surface to volume ratio allows rapid chemical reactions to be performed.

Figure 1: Laminar flow within a microfluidic channel (Re ≈ 0.05). Alternating streams of mineral oil dyed blue, intersecting within a machined channel of polytetrafluoroethylene PTFE and with a film lid of fluorinated ethylene propylene FEP. Channel depth of 1mm and scale bar is 2mm.
When two or more immiscible fluids at certain flow rates are delivered into a specific geometrical configuration, segmented flow conditions with serial trains of the fluids result. These are called emulsions where one fluid is dispersed within another, known as a carrier fluid. Three common geometries or ‘junctions’ can be employed to create such emulsions (Figure 2).

These junctions can be operated in one of two flow regimes, called dripping and jetting (Figure 3). It has been shown however that the jetting regime is more sensitive to external factors such as vibration and changes in temperature[4][5]. A tighter distribution of emulsion volumes, expressed as the monodispersity of diameters, is found when junctions are operating in the dripping regime[6][7].

2 Compound emulsion generation

Compound emulsions with their multiple encapsulated regions can be formed if two or more junctions are cascaded. For current IFE research the spherical shells are produced from a double compound emulsion which consists of a central inner fluid surrounded by an outer fluid which, in turn, is suspended in a carrier fluid (Figure 4). Where a liquid monomer is used as the outer fluid, this can be polymerised to form a hard and liquid-filled shell.

When two junctions are cascaded, the device can operate using either a one or two-step droplet formation process. The attention is at the first junction, which can either be driven to form segmented flow (Figure 5 (A)) or to form co-flow (Figure 5 (B)). The dispersed fluids can then be broken off to form compound emulsions at the second junction.

Great care must be taken to ensure the junction’s channel walls have the correct fluid to wall adhesion for the intended emulsion. For example, if a water dispersed in oil (W/O) emulsion is desired, the oil must
preferentially adhere to the junction’s channel walls rather than the water. Figure 6 shows the differing contact angles that result when either de-ionised water or mineral oil is placed on a polished surface of PTFE. The material is hydrophobic due to its low adhesion to water and therefore a high contact angle results but is also oleophillic with its adhesive affinity to mineral oil and hence has a low contact angle. It is the large difference in contact angle between the fluids towards the channel material that makes it ideal for the stable production of O/W emulsions. For compound emulsions where an oily monomer is used as the outer fluid, the configuration can be either water in oil in oil (W/O/O) or water in oil in water (W/O/W).

It is important to understand that millimetre size emulsions are much larger than is commonly associated with microfluidics. Because of the larger mass, inertial forces start to have a greater influence over that of interfacial tension, which dominates in the micrometre domain, thereby increasing the difficulty in their production. It would therefore be appropriate to say we are using millifluidics.

IFE shell manufacture using emulsions was investigated around 1979 by U. Kubo and others. However the flask-based agitation method used to make the emulsions was unsuitable for the required repeatability[8]. The process was greatly improved by M. Takagi with his invention of the triple orifice generator[9] which uses a co-flow type geometry and can produce repeatable high quality shells as shown by K. Nagai[10]. The geometric variability built into the design allows for a large range of emulsion diameters of around
0.5 - 6mm although the geometry can be simplified to a static planar design with the consequence of reducing the range. A recent study which used an enlarged cascaded T-junction[11] was shown to produce emulsions of 1.4mm to 2.5mm with a wall thickness between 0.5-0.75mm respectively albeit not the thin-walled shells (0.1-0.25mm) required for IFE. The flow into a T-junction is one-sided resulting in droplets with asymmetric internal flow eddies which could ultimately affect the shells quality. When choosing a symmetric flow junction however, extreme care must be taken to ensure that the flow and fluid entering each side is similar in all characteristics.

### 2.1 Setup of the microfluidic system

Microfluidic geometries are machined into plastic discs called ‘chips’. A transparent lid and quartz glass window is then placed on top and the whole assembly is compressed together using a bolted stainless steel captive housing with pipes transferring fluid from beneath (Figure 7). If the disk is chosen to be polytetrafluoroethylene PTFE with a fluorinated ethylene propylene FEP film lid, the hydrophobic material properties allow for W/O/O type emulsions to be formed without the need for surface modification or surfactant. The fluids used comprise of an inner water solution, density-matched to that of the outer fluid - trimethylolpropane triacrylate (TMPTA) monomer with an added photo-initiator as suggested by W. Nazarov[12]. Mineral oil acts as the carrier fluid. Once the emulsions are formed at the junction(s) they can be polymerised using ultraviolet (UV) light to form spherical, liquid-filled shells. Photo-polymerisation occurs in a photo-reactor which can be situated either on the chip or separately.

![Figure 7: An exploded-view of the microfluidic chip within a stainless steel housing. A transparent lid and quartz glass viewing window are placed on top of the chip with fluid transfer pipes connecting from beneath.](image)

### 2.2 The cascaded Co-Flow-Focus (COFUS) junction

The cascaded COFUS junction uses a two-step droplet breakoff process created when two separate junctions are cascaded together (Figure 8). Both are co-flow-focus COFUS geometries which are a hybrid between the co-flow and flow-focus type junctions (Figure 2). The curves redirect the carrier fluid flow from being perpendicular to the dispersed fluid into a shallow angle, like that of the co-flow. The lower curve allows a smooth translation of carrier fluid through an arc, to which the two sides form a flow-focusing orifice minima. The geometric shape of the COFUS junction therefore allows the dispersed fluid(s) to extend far enough into the junction to form millimetre sized emulsions while maintaining operation in the dripping regime (Figure 9). ([COFUS Video](video)) shows the junctions producing compound emulsions at a rate of 2.5Hz.
Figure 8: The cascaded co-flow-focus (COFUS) planar compound emulsion generator. Depth is to be
1mm. Scale bar is 2mm.

It should be noted that millimetre compound emulsions can also be formed using an enlarged and
cascaded flow-focus geometry (Figure 10); but under some flow conditions it can start to perform unde-
sirably by breaking up the compound emulsion and forming satellites. Compared with the average flow
velocity of fluid in the carrier channel, a high velocity flow is observed around the sharp, lower corners of
the carrier channel which flows into the outlet orifice. The carrier fluid contributes little to the movement
of the dispersed fluid towards the outlet, until the dispersed fluid bulges out with sufficient volume to
reach the high velocity flow (t=0.768). From here on it gets rapidly squeezed and accelerated through
the outlet orifice, but under certain carrier flow rates, 30ml/h in this example, the trailing edge which
contains two fluids, is pinched off. The emulsion-pinching effect observed in the enlarged flow-focus results
in undesirable compound satellite emulsion(s) along with the main compound emulsion (t=0.256-0.384).
For the cascaded COFUS junction the re-orientated carrier fluid flow aids the movement and shaping of
the dispersed fluid towards the outlet at lower flow rates than its flow-focus counterpart. Therefore lower
carrier flow rates can be used, which reduces the risk of unwanted emulsion-pinching.

Figure 9: Footage at the second COFUS junction shows the production of compound emulsions which
consists of a water inner fluid, TMPTA monomer outer fluid and a carrier of mineral oil. A small
TMPTA satellite droplet is created behind the main droplet t=0.12. Blue streak lines are to aid carrier
flow visualisation. Orientation of the carrier fluid aids the movement of dispersed fluid(s) towards the
outlet. The curves create a flow-focusing orifice minima which aids breakoff mechanism when blocked
by the dispersed fluid(s). Flow rates (ml/h); Inner(yellow) = 2 / Outer(red) = 2 / Carrier(blue/clear)
= 10. Time in seconds. Scale bar is 2mm.
Figure 10: An enlarged flow-focus junction producing compound emulsions consisting of a water inner fluid, TMPTA monomer outer fluid and a carrier of mineral oil. The small particles are for velocimetry measurement. Compound satellite droplets can be observed to be formed at $t=0.384$ due to the rapid acceleration of the main emulsion once it reaches the high velocity flow around the sharp, lower corners. The high velocity flow pinches the rear of the breakoff to form the compound satellites ($t=0.256-0.384$).

Flow rates (ml/h); Inner = 6 / Outer = 3 / Carrier = 30. Time in seconds. Scale bar is 2mm.

2.3 The Co-Flow-Focus in One Step (COFON) junction

An alternative design uses a one-step emulsion breakoff process with a junction called a co-flow-focus in one step or COFON (Figure 11). Here the two dispersed fluids are arranged coaxially using a slight offset between the fluid entrance nozzles to introduce one fluid inside another. The dispersed fluids are then broken off into compound emulsions by the carrier fluid flow within the co-flow-focus structure (Figure 12). Because the breakoff occurs over both the inner and outer fluids, compound satellites form from the thin strata of the breakoff process (Figure 13). The rupture of a coaxial strata can also result in water droplets within the outer layer which create voids within the polymerised shell and indicates why compound satellites should not be allowed to coalesce with the main breakoff. (COFON Video) captures the strata rupture event within the COFON junction.

Figure 11: One-step breakoff design of the co-flow-focus in one step (COFON). 1mm channel depth. Scale bar 2mm.
3 Satellite droplet issues and amelioration strategies

If droplets of viscoelastic fluids are formed using an orifice diameter within a particular range, satellite droplets are likely to be formed[13]. Being a continuous system, satellite droplets flowing chaotically around opens up a can of worms. Discussed are two passive methods that can remove the presence of the satellites. In the two-step cascaded COFUS design the first junction produces a water in TMPTA emulsion. At the second junction the flow of mineral oil leads the constituent dispersed fluids to be separated into droplets at the TMPTA segment, which leads to a satellite containing only TMPTA. By allowing the satellite to coalesce once more with the main breakoff, the impact of chaotically flowing satellite droplets can be reduced. For coalescence to occur, the thin film of mineral oil separating the two bodies needs time to drain away. This is called the drainage time[14]. Coalescence was accomplished using a stepped diffuser (Figure 14) which reduces the velocity of the satellites to a greater extent than that of the main breakoff. The multi-step arrangement brings the bodies together for enough time and under enough pressure to coalesce (Figure 15). The one-step COFON design has extensive issues with satellite formation because they are compound satellites that are formed when the dispersed fluid, which contains two fluids, is dispersed as compound emulsions. If the compound satellites are allowed to coalesce with the main breakoff, a void will be created in the outer fluid of the main emulsion which cannot then be easily removed. One way around the satellite droplet issue is to remove them after formation, before they have come into contact with the main breakoff. It has been identified that the Dean-flow passive sorter (Figure 16) could be used to separate the satellites from the main emulsion, although it has not yet been tested for sorting larger emulsions; it has been shown to work effectively in other microfluidic systems[15].
Figure 14: The stepped diffuser is joined directly after the second COFUS junction. Because the smaller satellite droplets have a lower velocity than the main breakoff, they eventually meet and press against each other until they coalesce. Each step is an increment of 0.2mm depth starting from 1mm at the junction. Distance between steps (semi-circle centre) is calculated by maintaining a consistent volume at each step, chosen to be equal to that of a 2.2mm diameter sphere. The steps ensure the confinement at the end of the diffuser does not limit the form of a droplet from being spherical. Scale is 2mm.

Figure 15: Footage of the stepped diffuser allowing satellites to coalesce with the main breakoff. Compound emulsions consist of a water inner fluid, TMPTA outer fluid and a carrier of mineral oil. Because a different clamping scheme was used in order to obtain unobstructed footage, flow leaked between the top of the side wall and the FEP film lid causing the emulsions to move to one side toward the end of the diffuser. For illustration of the coalescence, the carrier flow into the COFUS junction has been deliberately driven to exacerbate satellite formation. Satellites are comprised of TMPTA only.

Flow rates (ml/h); Inner=9 / Outer=6 / Carrier=48. Time in seconds.
4 Passive microfluidic methods for the concentric centring of compound emulsions

By far the most challenging task for the microfluidic method of shell manufacture is achieving micrometre-precision concentricity between compound emulsions in a predictable and cost effective way. Currently shells are manufactured in batches using flask-based chemistry, where agitation of the emulsions by propeller or by flask rotation during thermal polymerisation has been shown to significantly improve the concentricity\[3\][10]. However for IFE a batch process is unsuitable and a continuous method needs to be found. Di-electrophoresis centring techniques have indicated a possible strategy, however it is a relatively slow process, taking minutes\[16\]. NASA first observed that the fluids of a compound emulsion will become concentric if forced to oscillate at their resonant frequency\[17\]. Further on from the NASA work, a good model for the centring forces has been theorised by T. Norimatsu\[18\]. The model has focused on how the concentricity of the emulsion is affected by a step-response from a forced ‘perturbation’. The model leads to the idea of using modulations of the channel itself i.e. by using ‘passive compound emulsion centring by channel modulation’. Investigation of the influence that channel modulation has on the concentricity of a perturbated emulsion is an exciting area of research because the method could achieve high concentricity in a matter of seconds or even less.

One idea would be to use a single curve to create the step-response; however what was proposed was a design with multiple curved channel modulations. For an experiment sinusoidal modulations were added to a diffused photo-reactor’s outer channel wall (Figure 17). The shells produced from the modulated design were compared to those produced by the control experiment where the diffused photo-reactor did not have modulations (Figure 18).

Once the compound emulsions are generated by the first two COFUS junctions and have any satellites recombined in the stepped diffuser, they reach what is known as the accelerator. The accelerator serves both to further increase distance between emulsions without affecting the breakoff mechanism of the junction(s) and to allow for fine tuning of the velocity of the emulsions over the channel modulations. The diffusing of the outlet channel creates an amplitude modulation envelope and allows a range of emulsion sizes to be perturbated. Photo-polymerisation of the TMPTA emulsions starts during modulation, using a row of eight 375nm low power (≈0.2W) UV LEDs placed directly above the channel (Figure 22) and is fully polymerised when it passes two external high power 375nm (≈7W) UV LEDs placed opposite each other and orthogonal to the outlet tube. Due to the size constraints of the chip the outlet channel could not be made flat, augment the modulation or apply sophisticated amplitude modulation. However the comparison of results displayed a clear improvement in the sphericity and concentricity of the polymerised shells when using channel modulation (Figure 19).
5 Considerations for emulsion repeatability

The volume of each emulsion must be predictably repeatable to extremely tight tolerances in order to get a monodisperse population of shells. The fluid flow into the microfluidic junctions dictates the time period between the emulsions breakoff which relies heavily on cyclic pressure changes, caused when an emulsion blocks the output[19]. The fluid delivery system for the majority of microfluidic devices relies on the use of syringe pumps which are designed for dispensing volumetrically controlled fluid. Most syringe pumps use a motor connected to a lead screw to transform the rotary motion to a linear motion, thereby advancing the syringe’s plunger. The motor used is most often a stepper type due to its high torque, low cost, wide speed range and quantised rotational steps, which allows for precise volumes to be dispensed over time. However the inherent ‘cogging’ due to the steps causes a non-continuous rotation which translates into a non-continuous linear motion advancing the syringe plunger. The effect of this non-continuous syringe driving is known to cause pressure fluctuation of the output fluid flow[20]. It is therefore reasonable to assume that a system that uses multiple syringe pumps, delivering at different flow rates, causes unsynchronised pressure fluctuations. These can disturb the breakoff mechanism, which is manifested in varying the time period between emulsion breakoff. The sensitivity of the breakoff process to changes in pressure and the use of multiple stepper-motor syringe pumps could explain the variation in emulsion volumes observed by Moynihan[11].
5.1 Experimental method for testing emulsion repeatability

The time period between emulsion breakoff is used as the indicator for volume repeatability. From video footage the front of each successive emulsion passing an arbitrary line is timed to calculate the period between the occurrences for \((n)\) instances. To compare the use of stepper and continuous syringe pump drives, two KdS 200 dual syringe pumps were employed. One of these had its stepper motor drive replaced with a precision ironless DC motor with a high reduction, low noise gearbox and rotary encoder. This type of motor does not suffer from any form of ‘cogging’ due to the ironless rotor. The syringes used were 1ml SGE gas-tight syringes which have a 4.61mm glass bore and PTFE plunger. One syringe was filled with mineral oil and the other with de-ionised water and together were fitted to the drive pump. The outlet from the syringes was attached to the first junction of the COFUS (Figure 8) in order to make W/O emulsions and was filmed at fifty frames per second. The DC motor has less speed range than the stepper motor and therefore dictates the flow rates that could be compared. To calculate the flow rate of the DC drive, the output frequency from the rotary encoder was recorded and averaged over the experiment. The average frequency could then be converted and used to set the equivalent flow rate on the stepper motor syringe pump drive.

5.2 Results from the emulsion repeatability experiment

Comparing the standard deviation of the breakoff period between the two drives shows that the DC drive has a lower variation than the stepper drive at all flow rates tested between 1.6 and 16.43 \((\text{ml/h})\). The equivalent rotational speed of the leadscrew is calculated to be 1.51 and 15.51 revolutions per minute for a 1.05735mm pitch lead driving a 1ml, 4.61mm diameter syringe. Table 1 gives the standard deviation and coefficient of variation of the breakoff period with the raw data presented in (Figure 20). For the lowest flow rate tested \((1.6 \text{ ml/h})\) there is a notable peak in the breakoff period for the stepper motor drive at between instance 27 and 29 which is not observed from the DC drive. The sustained peak is possibly caused by the pump’s internal feedback strategy which is likely to correct for missed steps and drive the syringe at an uneven velocity.

| (Voltage (V)) | Equivalent flow rate (ml/h) | DC drive | Stepper drive |
|---------------|-----------------------------|----------|---------------|
| 2             | 1.6                         | (0.160)  | (0.616)       |
| 4             | 3.71                        | (0.049)  | (0.080)       |
| 8             | 7.91                        | (0.015)  | (0.050)       |
| 16            | 16.43                       | (0.009)  | (0.018)       |

Table 1: Variation in emulsion breakoff period for syringe pumps with a DC or stepper motor drive expressed as the standard deviation \((s)\) and coefficient of variation \((\%\)
5.3 Conclusions from the emulsion repeatability experiments

Delivery of consistent pressure and flow rate to the emulsion generators is essential for forming consistent droplet breakoff timing which affects the monodispersity of shells. Although improvements can be made by the DC drive replacement, there is still undesirable variation of the breakoff period. The variation could be due to a number of factors such as the stick-slip characteristics of the syringes, the alignment of the dual syringe pumps pusher bar and lead screw accuracy among others. It is therefore reasonable to conclude that further improvements might be achieved by using other fluid delivery systems.

6 Cost of shell manufacture

Cost is of great concern, especially with the requirement of over one million shells per day. The carrier fluid used so far has been mineral oil. However it is relatively expensive and density matching is complex in contrast to using water as the carrier fluid to create a W/O/W emulsion. Using water as the carrier fluid increases the surface tension between it and the outer fluid (TMPTA). According to the most out of roundness (MOOR) law[3] a higher static sphericity can be achieved with increasing surface tension. In addition, diffusion is greatly reduced due to the reduced mutual miscibility between the fluids.

Formation of a W/O/W emulsion using the two-step cascaded COFUS requires hydrophillic walls at the second junction. The surface modification was achieved by coating a machined PMMA chip and lid in a hydrophillic material. This can be stacked on top of the first junction, machined into PTFE with a FEP film lid. The high contrast in static contact angle, with a water droplet (PTFE > 100°) and (Coated PMMA < 5°) enables W/O/W emulsions to be generated (Figure 21). The amount of usable shells that the microfluidic device can make in its lifetime versus the cost of the device must be understood as it will affect the final shell cost. Reliability might be improved if the base material or Lotus leaf patterning[21] was used to create hydrophillic and hydrophobic areas respectively.
Figure 21: Millimetre sized W/O/W compound emulsions being formed at the second junction of the cascaded COFUS using a stacked chip of PTFE and a hydrophillic surface treated PMMA chip. Compound emulsions consist of a density matched water solution for the inner and carrier fluid and a TMPTA outer fluid. Flow rates (ml/h): Inner=1.5 / Outer=2 / Carrier=40. Time in seconds and scale bar is 1mm.

7 Conclusion

Microfluidics has shown a possible route to achieve the economical and rapid production of spherical polymer shells, although it is clear that one must control every part of the system to exceptionally tight tolerances in a holistic manner.

The novel planar geometries of the Co-Flow-Focus in One step (COFON) and cascaded Co-Flow-Focus (COFUS) generate compound emulsions in a one or two step breakoff method respectively. They have both shown an ability to produce 2mm+ size W/O/O double compound emulsions which satisfy the required dimensions for IFE. The COFUS has been further modified to generate W/O/W emulsions using PTFE for the first junction and coated PMMA for the second.

The formation of satellite droplets is observed using one and two step breakoff methods, the satellites themselves being comprised of either a single or a double fluid. If the satellite is of a single fluid, it can be recombined with the main breakoff by using a stepped diffuser; otherwise they could be removed using passive techniques such as the Dean flow sorter.

Use of channel modulations to perturbate and thereby aid concentric alignment of the emulsions is certainly an area of research to investigate further. A direct comparison between diffused photo-reactors with or without sinusoidal modulation showed an improvement in concentricity, sphericity and intactness when modulations are used.

A suitable fluid delivery system is one which is continuous and pulsation free, so as not to affect the emulsion breakoff process. Using an off the shelf syringe pump (KDS 200), variation in the time period between emulsions being generated was measured. Improvements were observed when the stepper motor drive was replaced with a geared down continuous brushed ironless DC motor. However the results of such modifications still fall short of requirements and other methods ought to be investigated.

By way of conclusion, controlled and cost-effective fusion energy must be achieved for the progression of mankind and the problem of the economic mass production of IFE shells might well be solved by using microfluidics, even though “nature is going to put up a fight”[22].

Figure 22: Science-selfie in front of the microfluidic system that uses channel modulation to improve the concentric alignment of compound emulsions. Insert to the left shows photo-polymerisation of emulsions in a modulated and diffused photo-reactor. 27/02/2014.
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