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Research article

Investigation of factors affecting the channel sealing processing mode of microfluidic chips using spin coating technology

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ABSTRACT
Nowadays, the research and production of microfluidic chips for various applications in bio/chemical fields are attracting significant attention from scientists around the world. The fabrication of microfluidic chips typically involves two steps: creating open microchannels and subsequently sealing them. The sealing of these open microchannels is a critical step that determines the ultimate quality of the product. Adhesive bonding and solvent bonding are two commonly used approaches for sealing open channels. Both approaches require the creation of an adhesive layer or a solvent layer sandwiched between the two substrate surfaces to facilitate bonding under the influence of chemical and physical reactions. Therefore, controlling the process of creating this layer plays a vital role. To achieve this, spin coating is a technique often used to quickly and simply create thin coating layers owing to the centrifugal force. However, this method also has many limitations such as limited thickness control, uniformity issues, limited material compatibility, and limited control over film structure. In this study, the authors conducted an investigation of the factors affecting the formation of coating layers using the spin coating technique. The investigation results showed difficulties in choosing the substrate placement position and selecting the appropriate spinning speed for the coating material. Additionally, the authors propose the incorporation of a new force, in conjunction with centrifugal force, during the coating process, which demonstrates potential in mitigating the formation of distinctive thickness profiles by reducing the thickness in the central region of the coating
Key words: Coating, Spin coating, Sealing of Microfluidic Chip

INTRODUCTION

Microfluidics is a science and technology that deals with the behavior, control, and manipulation of fluids that are constrained to a sub-millimeter scale. This interdisciplinary field merges principles from physics, chemistry, engineering, and biotechnology to design and develop systems that manipulate small volumes of fluids. These systems, often referred to as "lab-on-a-chip" devices, allow for precise handling and analysis of fluids at the microscale level. Microfluidic devices typically consist of channels, chambers, and valves fabricated on a small chip using microfabrication techniques. These devices enable various applications such as chemical synthesis, drug delivery, point-of-care diagnostics, and biological assays. The advantages of microfluidic systems include rapid analysis, reduced sample volumes, automation potential, and integration of multiple functions onto a single platform. Most microfluidic chip manufacturing processes involve two steps: channel fabrication and channel sealing. The channel sealing step plays an extremely important role as it determines the final quality of the microfluidic chip. Two commonly employed methods for sealing open channels are adhesive bonding and solvent bonding. In both approaches, an adhesive or solvent layer is needed to be created between the two substrate surfaces to enable bonding through chemical and physical reactions. Hence, controlling the process of forming this layer assumes critical importance. Spin coating, a technique frequently utilized to swiftly and easily produce thin coating layers due to centrifugal force, is employed to achieve this objective [1,2]. Nevertheless, this method also presents several limitations, including restricted thickness control, uniformity issues, limited material compatibility, and inadequate control over film structure [3].

In this study, we investigate factors affecting the channel sealing processing mode of microfluidic chips using spin-coating technology and propose a new way to reduce the thickness at the center of the spin-coated layer [4].

METHOD

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Mathematical Modeling of Thin Film Formation in Traditional Spin Coating

A typical spin coating process is depicted in Figure 1 and commonly undergoes the following steps: **(1) Preparation of Substrate:** The substrate, typically a flat surface such as a silicon wafer or glass slide, is cleaned thoroughly to remove any contaminants; **(2) Dispensing Liquid**: A small amount of liquid coating material, often a solution or dispersion, is dispensed onto the center of the substrate surface; (**3) Spinning**: The substrate is rapidly rotated (spun) around its central axis. The centrifugal force generated by the spinning causes the liquid to spread outwards from the center towards the edges of the substrate; **(4) Film Formation**: As the liquid spreads, it forms a thin, uniform film across the substrate surface due to the combination of centrifugal force and surface tension; **(5) Evaporation or Curing**: Depending on the nature of the coating material, the solvent may evaporate, leaving behind a solid film, or the material may undergo curing or crosslinking to form a solid film; **(6) Final Film Thickness**: The final thickness of the film is influenced by various parameters including the viscosity of the coating material, spinning speed, and duration of spinning. Furthermore, a previous study demonstrated that the thickness of the film is inversely proportional to the distance from the center raised to the power of 1/3, indicating that the greater the distance from the center, the thinner the film [4].

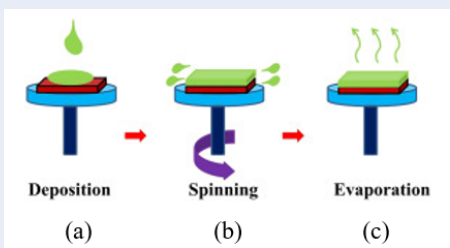

Figure 1: A typical spin coating procedure [5]

Examining a liquid droplet positioned on the flat surface of a rotating disk, the droplet's location can be determined using cylindrical coordinates (r, θ, z), where r denotes the distance from the center of rotation to the droplet, θ represents the angle relative to the reference position, and z signifies the droplet's height. The rotating plane possesses an angular velocity ω. Two forces act upon the rotating droplet: viscous drag force and centrifugal force. To induce thin film motion, the minimum centrifugal force must surpass the droplet's inherent viscous drag force. As the flat disk's rotational velocity escalates, the centrifugal inertial force acting on the droplet increases, facilitating the droplet's motion and film formation on the disk's surface. Nevertheless, excessive rotational speed diminishes the droplet's adhesion to the disk surface. Furthermore, the disk's imbalance ensues due to substantial uncontrolled vibration effects, leading to system instability and safety concerns. Thus, constraining the flat disk's rotational speed to the lowest feasible level is imperative.

To determine the minimum centrifugal force under equilibrium conditions of the system consisting of a rotating flat disk and a droplet, both forces are balanced, and the force equilibrium equation can be expressed as in equation (1). Here, η, u, and ρ represent the absolute viscosity, radial velocity, and density of the fluid, respectively.

$$0 = \mu\left(\frac{\partial^2 u}{\partial z^2}\right) + \rho\omega^2 r \quad (1)$$

Since u is only a function of z, equation (1) can be written as

$$-\left(\frac{\partial^2 u}{\partial z^2}\right) = \frac{\rho\omega^2 r}{\mu} \quad (2)$$

Let h_f be the final film thickness at the spinning time t, and let double integration concerning z direction, the force-balance equation (2) becomes equation (3).

$$u = -\frac{1}{2}\left(\frac{\rho\omega^2 r}{\mu}\right)z^2 + c_1 z + c_2 \quad (3)$$

Applying the boundary conditions. There is no shear force at the top surface of the film ($z = h_f$) then $\partial u/\partial z = 0$, and no-slip at the bottom of the film (z = 0) then u = 0. With $c_1 = \left(\rho\omega^2 r h_f\right)/\mu$, $c_2 = 0$, the equation (3) becomes equation (4).

$$u = -\frac{1}{2}\left(\frac{\rho\omega^2 r}{\mu}\right)z^2 + \left(\frac{\rho\omega^2 r h_f}{\mu}\right)z \quad (4)$$

To find the radial volumetric flow rate of the drop, the integration of the liquid velocity u concerning the vertical direction z from 0 to final film height h_f. The equation (4) becomes equation (5).

$$q = \int_0^{h_f} u(z)\,dz = \left[\left(-\frac{1}{6}\right)\left(\frac{\rho\omega^2 r_f}{\mu}\right)z^3 + \frac{1}{2}\left(\frac{\rho\omega^2 r_f h_f}{\mu}\right)z^2\right]_0^{h_f}$$

$$= \left(\frac{1}{3}\right)\left(\frac{\rho\omega^2 r_f}{\mu}\right)h_f^3 = \left(\frac{\rho\omega^2}{3\mu}\right)r_f h_f^3 \quad (5)$$

For a spin coating with a uniform rotating speed, the variables that affect h_f are the spinning time t and the distance r_f from the center with $h_f = h\left(r_f(t)\right)$. To obtain a differential equation for h_f, the equation (6) of continuity is applied, where $Q = uA = qr_f$ and $A = h_f r_f$.

$$0 = \frac{\partial(\rho u A)}{\partial r_f} + \frac{\partial(\rho A)}{\partial t} = \frac{\partial\left(\rho q r_f\right)}{\partial r_f} + \frac{\partial\left(\rho r_f h_f\right)}{\partial t} \quad (6)$$

$$0 = \frac{\partial\left(q r_f\right)}{\partial r_f} + r_f\left(\frac{\partial h_f}{\partial t}\right) \quad (7)$$

$$-\frac{\partial\left(q r_f\right)}{\partial r_f} = r_f\left(\frac{\partial h_f}{\partial t}\right) \quad (8)$$

The equation (9) is the use of equation (6) in (8) yields with $k = \left(\rho\omega^2/3\mu\right)$

$$\left(\frac{\partial h_f}{\partial t}\right) = -k\left(\frac{1}{r_f}\right)\frac{\partial\left(r_f^2 h_f^3\right)}{\partial r_f} \quad (9)$$

Before seeking general solution of equation (9), the special solution is considered, which depends only on time t.

$$\left(\frac{\partial h_f}{\partial t}\right) = -k\left(\frac{1}{r_f}\right)\left(2 r_f h_f^3\right) + k\left(\frac{1}{r_f}\right)\left(3 r_f^2 h_f^2\right)\left(\frac{\partial h_f}{\partial r_f}\right) \quad (10)$$

$$\left(\frac{\partial h_f}{\partial t}\right) = -\left(2 k h_f^3\right) + \left(3 k r_f h_f^2\right)\left(\frac{\partial h_f}{\partial r_f}\right) \quad (11)$$

Since the film is uniform at the beginning, h is independent of r and hence $\left(\frac{\partial h_f}{\partial r_f}\right) = 0$ which gives equation (12).

$$\left(\frac{d h_f}{dt}\right) = \left(\frac{\partial h_f}{\partial t}\right) = -\left(2 k h_f^3\right) \quad (12)$$

or

$$\left(\frac{\partial h_f}{h_f^3}\right) = -(2k)\,\partial t \quad (13)$$

Integrating both sides of equation (13) with the limits at $t = 0$, $h_f = h_{fo}$ and at $t = t_f$, h_f, the equation (14) is written out. The equation (14) is the solution corresponding to an initially uniform distribution with $h_f = h_{fo}$ at $t = 0$. Thus, if the initial distribution of fluid is everywhere uniform, it will remain so with time, as the thickness of the fluid film is decreased by continuing application of centrifugal force. This conclusion immediately tells us that ultimate uniformity in thin films is assured if an initial thick fluid distribution, before centrifugation, can somehow be made uniform. However, it does not tell us whether uniformity can be expected in the more practical case of an initial distribution that is irregular[6]. Equation (14) shows that the fluid layer decreases in thickness by a factor $1/\sqrt{2}$ in a time which shows that a thick layer thins out much more rapidly than a thin one. This suggests, in turn, that a no uniform layer should become increasingly more uniform as centrifugation continues.

$$h_f = \frac{h_{fo}}{\sqrt{1 + 4 h_{f0}^2 k t}} = \frac{h_{fo}}{\left(1 + 4 h_{f0}^2 k t\right)^{1/2}} \quad (14)$$

In general solution, similarly from equation of continuity (8) and the expression for $\partial h_f/\partial t$ is equation (11). Relating partial and total derivatives as equation (15).

$$\left(\frac{d h_f}{dt}\right) = \left(\frac{\partial h_f}{\partial t}\right) + \left(\frac{\partial h_f}{\partial r_f}\right)\left(\frac{d r_f}{dt}\right) \quad (15)$$

and

$$\left(\frac{d r_f}{dt}\right) = 3 k r_f h_f^2 \quad (16)$$

On substituting equation (14) into equation (16) obtained equation (17). The equation (18) is found out by the integral of equation (17).

$$\left(\frac{d r_f}{dt}\right) = \left(\frac{3 k r_f h_f^2}{1 + \left(4 k h_{f0}^2\right) t}\right) \quad (17)$$

$$r_f = r_{f0}\left(1 + \left(4 k h_{f0}^2\right) t\right)^{3/4} \quad (18)$$

Equations (14) and (18) give the coordinates $\left(r_f, h_f\right)$ after time t_f of a point on the surface in terms of its original coordinates $\left(r_{f0}, h_{f0}\right)$.

The spinning speed is one of the most crucial factors in the spin coating process. The centrifugal force depends on this factor and causes turbulence in the air immediately above the coating layer. Even small changes in speed at this stage can lead to significant variations in thickness, as shown in Figure 1(c). When increasing the spinning speed of the disk, the thickness of the film is largely achieved by balancing the forces acting to push the liquid droplet towards the edge of the substrate. As the droplet evaporates, it becomes more viscous until the centrifugal force can no longer move the droplet on the surface. At this point, the film thickness will not decrease significantly as the spinning time increases. Besides spinning speed, the ability to accelerate can also affect the coating layer. Because droplets begin to dry in the early part of the cycle, it is important to control acceleration accurately. According to some studies, in certain processes, nearly 50% of the solvent in the droplets will evaporate in the first few seconds of the process. Acceleration also significantly influences the characteristics of the coating layer on substrates with patterns or grooves. In many cases, the film will replicate the shape of the terrain from previous processes. It is crucial for the coating layer to be uniform and pass through these features. When centrifugal force directs and energizes the droplet, acceleration assists in dispersing the droplet around the terrain and may fill or miss portions of the substrate material from the droplet forming the film.

Based on the literature review, some criteria for selecting the appropriate spin coating mode are as follows:

a. The more uniform the film and the better control over the desired thickness

b. The lower the spinning speed, the better to ensure adhesion and filling of the coating material onto the substrate.

c. The faster the processing time, the better to avoid issues with solvent evaporation control of the coating material.

Here, we can see that criteria a contradicts b, and b contradicts c because the centrifugal force (F_{cen}) necessary for the coating process depends significantly (quadratic) on the spinning speed of the flat disk and the radius of the coating position. The resolution of these contradictions will be addressed in the following section.

Parametric Study of Spin Coating for Microchannel Sealing

To investigate and assess the selection of appropriate coating modes for creating adhesive layers or solvent layers to support chemical bonding between substrates in the fabrication process of microfluidic chips using traditional spin coating methods, the mathematical model will rely on equations (14) and equation (18). The evaporation of solvents into the environment will not be addressed here to simplify the investigation process.

To prevent the risk of excessive adhesive layer coating, which could potentially lead to channel clogging, the spinning speed and processing time must be appropriately matched to the employed droplet volume. In the case where the coating material is a mixture of Glycerin-Water, with a desired thickness of approximately $h_f \approx 0.001mm$ for the processing area with a radius of $r_f = R_{max} = 75\ mm$ for a droplet volume V = 20 μL, the survey parameters for the spin coating process are presented in Table 1.

Table 1: The parameters investigated for Glycerin-Water mixtures droplets

Symbol	Description	Value
μ	absolute viscosity	16×10^{-3} (Pa.s)
r	fluid density	1.169×10^{3} (kg/m^3)
r_0	The radius of the droplet when it contacts the surface of the rotating disk	2.75×10^{-3} (m)
V	Droplet volume	2.25×10^{-8} (m^3)
n	Spinning speed of the rotating disk	From 100 to 1000 (rpm)

s = second, m = meter, kg = kilogram, N = Newton, rpm = round per minutes.

RESULTS AND DISCUSSION

The investigation results indicate the relationship between the radius of the coated region r_f and the thickness of the coating layer h_f at different spinning speeds of the flat disk, as described in Figure 2 .

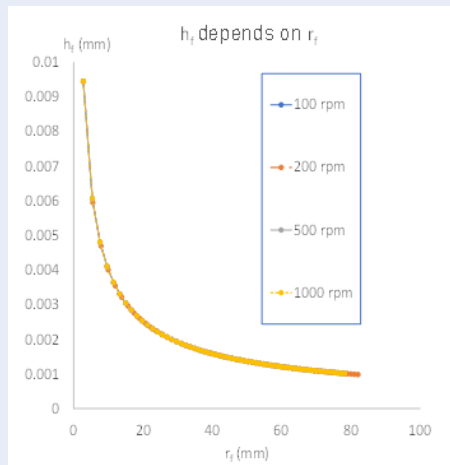

Figure 2: The relationship between the radius of the coated region and the thickness of the coating layer atdifferent spinning speeds of the flat disk.

Positioning the kit test at the center of the flat disk confers several advantages for device design, such as facilitating a compact structure, ensuring stable operation, and enhancing safety measures. However, a notable drawback of this configuration is the necessity for an exceedingly high spinning speed to initiate membrane formation. Consequently, achieving dynamic equilibrium within the system becomes considerably more challenging.

Placing the test kit at a distance from the center of the disk, typically r_f > 25 mm as observed in the study, serves as an example of achieving a more uniform thickness for the membrane layer. Nevertheless, this arrangement also introduces instability to the system due to uneven mass distribution on the spinning disk and increases the consumption of coating material. The influence of spinning speed on the ability to uniformly thin the surface of the coating layer is depicted in Figure 3 .

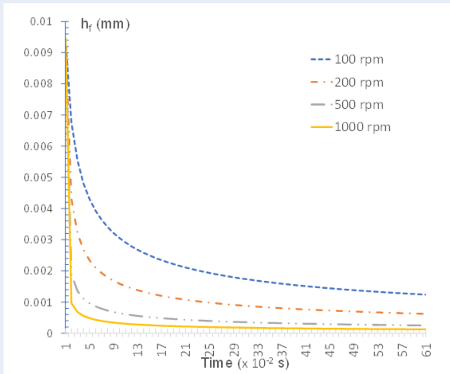

Figure 3: The influence of different spinning speeds on the ability to uniformly thin the surface of the coating layer over time.

As the spinning speed increases, the coating layer becomes easier to form. To achieve an adhesive layer with a thickness of approximately $h_f \approx 0.001mm$, the coating modes are presented in Table 2. Additionally, the formation region of the coating layer r_f over time at different spinning speeds of the flat disk is illustrated in Figure 4. With increasing speed, the region of coating formation is also expanded. In Figure 5 (a), (b), (c), and (d), the trends in coating formation and corresponding thickness at various r_f regions for different processing speeds from 100 rpm to 1000 rpm are shown.

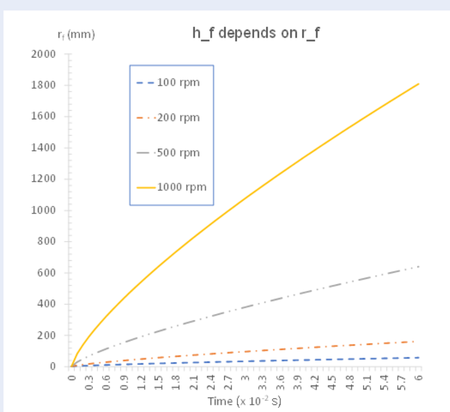

Figure 4: The formation of the coating layer region over time at various spinning speeds of the flat disk.

Table 2: Results of Processing Modes for Glycerin-Water Mixtures Droplets with $h_f \approx 0.001mm$, and $R_{max} \approx 75\ mm$

No.	Rotational Speed- n	Time
1	100	8.55 s
2	200	2.12 s
3	500	0.342 s
4	1000	0.0855 s

s = second, rpm = round per minutes.

The survey results indicate the difficulty in selecting the substrate placement position ($r_f \approx 25$ mm) and choosing the appropriate spinning speed for the coating material. This is a consequence of the crucial external force required for the coating process, namely the centrifugal force F_{cen}, which is highly dependent (quadratically) on the spinning speed of the flat disk and the radius of the substrate placement position. The centrifugal force F_{cen} decreases as the disk's center is approached. To overcome this problem, a suggestion is to introduce an extra force on the spinning disk to enhance the dispersal capability of the coating material in the central region. The direction of this supplementary force component aligns with the centrifugal force F_{cen} and diminishes in magnitude as the radius of the spinning disk increases.

To balance the centrifugal inertial force F_{cen} , a supplement force F_{sup} should be enhanced for the system. This will help the spin coating system achieve better efficiency and become less dependent on the substrate placement and selection of spinning speed. Figure 6 illustrates the relationship between the centrifugal inertial force F_{cen} the supplementary force F_{sup} , and the total force F_{total}

The force F_{sup} can be generated in various ways, such as the suggested vibration force in previous related studies[7]. Within the scope of this study, the force F_{sup} component is only proposed to be added to generate the necessary total forceF_{total} , and its determination is done in the simplest way. There are several methods to create the force component F_{sup} and will be addressed in another study. The total forceF_{total} can be find out in equation (19) and (20).

$$F_{total} = F_{sup} + F_{cen} \tag{19}$$

$$0 = \mu \left(\frac{\partial^2 u}{\partial z^2} \right) + \rho \omega^2 r + \frac{\partial F_{sup}(r)}{\partial V} \tag{20}$$

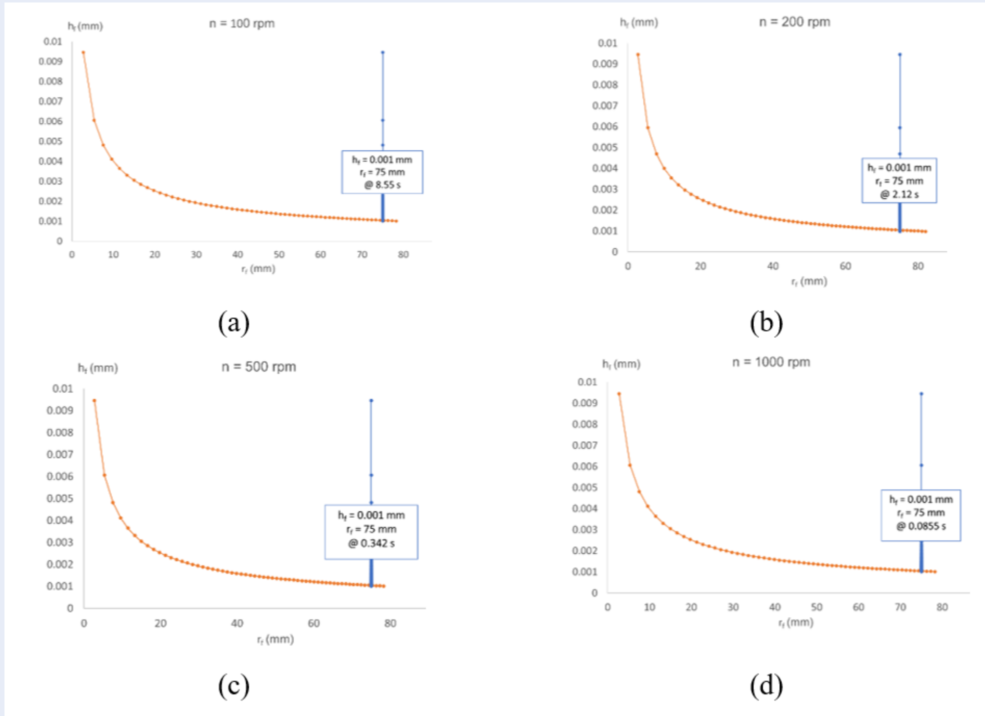

Figure 5: The formation of the coating layer region overtime at different spinning speeds of the flat disk. (a) at 100 rpm, (b) at 200rpm, (c) at 500 rpm, and (d) at 1000 rpm.

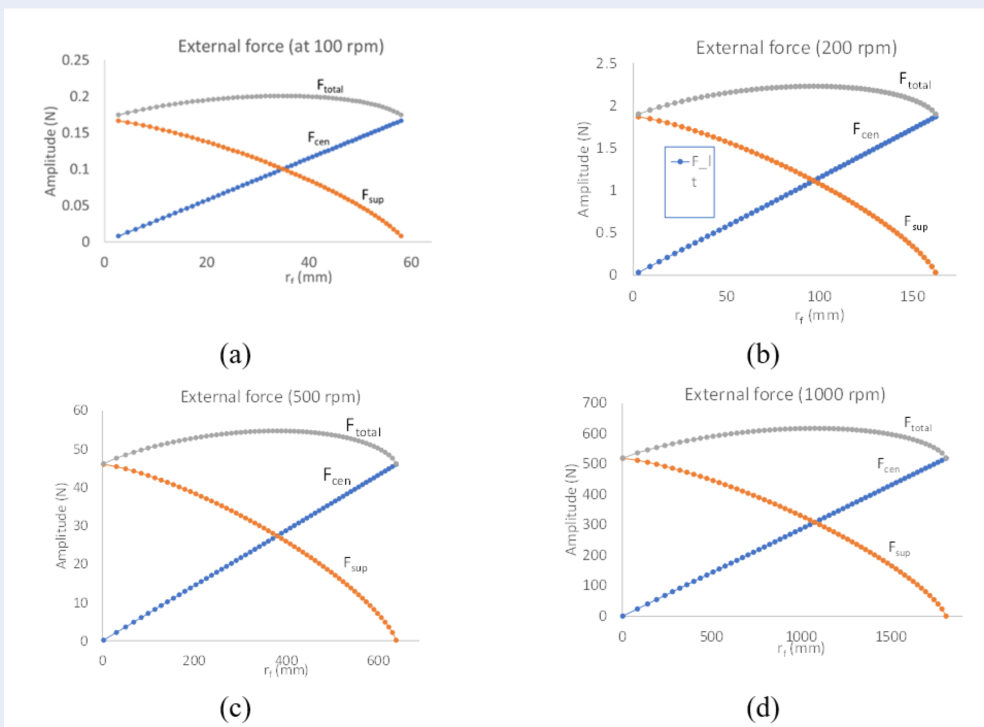

Figure 6: The relationship between the centrifugal inertia force F_{cen}, the additional force F_{sup} and the total forceF_{total}.

CONCLUSION

The investigation results indicate difficulties in selecting the substrate placement position and determining the appropriate spinning speed for the coating material. This is a consequence of the crucial external force needed for the film formation process, namely the centrifugal force F_{cen}, which is highly dependent on the spinning speed of the flat disk and the radius of the coating position.

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CONFICTS OF INTEREST

The author group confirms that there are no conflicts of interest related to the research work.

AUTHORS' CONTRIBUTION

Authors Lynh Huyen Duong and Nam Hai Tran are accountable for the conceptualization, model selection, computational methodology, data analysis, manuscript refinement, and finalization. Students Tuong Chi Vu and Khang Minh Ngo contributed to computational tasks, and data analysis.

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Bài nghiên cứu

Khảo sát các yếu tố ảnh hưởng đến quá trình niêm phong kênh dẫn của chip vi lỏng dùng công nghệ tạo màng niêm bằng phương pháp phủ quay

Dương Huyền Lynh[1,2], Trần Hải Nam[1,2,*], Vũ Chí Tường[1,2], Ngô Minh Khang[1,2]

TÓM TẮT
Ngày nay, việc nghiên cứu, chế tạo các chip vi lỏng ứng dụng trong các lĩnh vực sinh học, hóa học đang thu hút sự quan tâm mạnh mẽ của các nhà khoa học trên toàn thế giới. Để sản xuất các chip lỏng, hầu hết đều cần thực hiện hai bước, bao gồm: gia công tạo vi kênh dẫn mở, và niêm phong các vi kênh dẫn mở đó. Việc niêm phong các vi kênh dẫn mở là một bước vô cùng quan trọng quyết định đến chất lượng cuối cùng của sản phẩm. ``Adhesive bonding''(liên kết dựa vào chất kết dính), và ``Solvent bonding'' (liên kết dựa trên dung môi) là hai hướng tiếp cận thường được sử dụng để niêm phong các kênh dẫn mở. Hai hướng tiếp cận này đều yêu cầu việc tạo ra một lớp phủ vật liệu kết dính, hoặc lớp dung môi giữa hai bề mặt chất nền để tiến hành việc liên kết dưới tác dụng của các phản ứng hóa, lý kết hợp. Việc kiểm soát tốt quá trình tạo lớp phủ vì thế đóng một vai trò quan trọng. Để làm được việc đó, ``spin coating'' (phủ quay) là một kỹ thuật thường được sử dụng để nhanh chóng và đơn giản tạo ra lớp phủ mỏng dựa trên tác dụng của lực ly tâm. Tuy nhiên, phương pháp này cũng tồn tại rất nhiều hạn chế như: việc kiểm soát độ dày lớp phủ, các vấn đề liên quan đến độ đồng nhất của lớp phủ, hạn chế về loại vật liệu phủ, và hạn chế trong việc tạo lớp màng phủ trên bề mặt chất nền có cấu trúc phức tạp. Trong nghiên cứu này, nhóm tác giả tiến hành khảo sát các thông số có ảnh hưởng đến việc tạo thành lớp phủ bằng kỹ thuật phủ quay. Kết quả khảo sát cho thấy việc khó khăn khi chọn vị trí đặt chất nền và chọn tốc độ quay cho phù hợp với chất tạo màng (chất phủ). Một vấn đề nữa là việc đề xuất thêm vào một thành phần lực mới kết hợp cùng với lực ly tâm trong quá trình phủ cho thấy tiềm năng trong việc hạn chế sự tạo thành đặc điểm độ dày đặc trưng của lớp phủ ``distinctive thickness profile'' thông qua việc làm giảm độ dày ở khu vực trung tâm của lớp phủ
Từ khoá: Phủ, Phủ Quay, Niêm phong chip vi lỏng

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