

## Research paper

## Robust spin coating deposition process for paraffin phase-change films

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## ABSTRACT

In this work, for the first time, a cost-effective and resource-light spin coating process for the fabrication of the paraffin films on silicon and glass substrates is reported. Paraffin or alkane is a mechanical phase-change material (PCM) that undergoes approximately 15% reversible volumetric change through its solid-liquid transition. In addition, it is a low-loss dielectric with a loss tangent of  $6 \times 10^{-4}$  at 100 GHz which is a critical feature for certain radio frequency (RF) tunable devices. Traditionally physical vapor deposition techniques are used for paraffin PCM films. Here, an innovative and alternative process using spin coating is developed that overcomes the challenges imposed by the phase-change properties of the material. Spin coating of two types of paraffin—eicosane and hexatriacontane—on silicon, Pyrex and gold-coated substrates are studied. P-xylene is chosen as the solvent due to the solubility of eicosane and hexatriacontane at temperatures lower than its melting point. Effect of spin speed, spin time, substrate temperature, solution concentration, and substrate type on roughness and thickness of paraffin films are reported. Developed process offers a fast, repeatable, and resource-light deposition technique for hexatriacontane which can be utilized in developing several classes of new devices including low-loss RF architectures.

## 1. Introduction

Paraffin or alkane is a mechanical phase change material (PCM) that exhibits a large volumetric change (approximately 15%) during its solid-liquid transition [1–3]. Alkanes have a chemical form of  $C_nH_{2n+2}$  where  $n$  is typically a number between 10 and 50. The phase change temperature of paraffin is directly related to the number of carbon atoms; therefore, the melting temperatures of paraffin waxes that are solid in the room temperature can range from 37 °C ( $n = 20$ ) to 95 °C ( $n = 50$ ) [4].

Alkanes are of interest because they are shown to have a high capacity of thermal energy storage and constant phase change temperature [5]. Additionally, they are used in the heat management of the lithium-ion batteries [6]. They are also chemically inert, non-corrosive, and stable with reverse phase transition properties [7]. Due to its low chemical reactivity and high thermal expansion properties, paraffin can also be used as a support layer for the fabrication of wrinkle-free large area graphene [8]. Large volumetric change of paraffin creates a large force that can be used in micro-actuators [9–13]. In addition to unique mechanical properties, paraffin has a very low dielectric loss with a loss tangent of  $6.3 \times 10^{-4}$  at 110 GHz [14,15] making it attractive for RF

devices such as low-loss tunable variable capacitors [16,17].

Recently, our group reported paraffin-based millimeter wave (30–300 GHz) low-loss variable capacitors [16], which were used in the development of a reconfigurable millimeter wave antenna. As opposed to RF micro-electro-mechanical systems (MEMS) switches, these variable capacitors are capable of continuous tuning and can operate with low voltage bias.

Previously, paraffin-based MEMS devices have exploited the large force offered by the volumetric expansion of paraffin at its melting point. An example of such a device is a micro-scale paraffin actuator. Carlen et al. [10] reported a micro-valve based on surface micro-machined paraffin as an actuation layer by using physical vapor deposition (PVD). The concept of refreshable braille cells presented by Su et al. [12] is another example. These devices utilize the hydraulic pressure due to volumetric expansion in electro-thermally controlled micro-actuators achieved by injecting small volumes of paraffin into specially designed containers.

For the development of micron-scale devices, fabricating paraffin films (1–10  $\mu$ m in thickness) with low roughness ( $< 0.2$  m) is crucial. Even though the thermophysical [18], mechanical [13,19,20], and electrical [14,21] properties of paraffin have been studied, little work

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has been done on the development of an inexpensive and fast film deposition process. PVD process has been extensively reported in the past. Barberis et al. [22] studied PVD of paraffin on oxidized molybdenum substrates. A film thickness of 3–4 nm over a 5 s period was reported. Carlen et al. [10] used a similar thermal evaporation process for fabricating a micro-valve. Using a custom-made PVD chamber they achieved a deposition rate of 1000 Å/min. While physical vapor deposition can provide films with thicknesses of a few nanometers to a few microns, it is a slow and resource-heavy process. Firstly, custom-made deposition chambers need to be built for paraffin PVD to avoid contamination. Once solidified, paraffin can be difficult to remove at room temperatures and can be damaging to the chamber [23]. Additionally, Carlen [9] noted contamination of large solid spheres on the deposited paraffin films resulting from the splashing of boiling paraffin. For these reasons, spin coating process as an alternative to PVD methods is evaluated.

An analysis on the spin coating of paraffin for encapsulating biological samples was conducted by Jing et al. [24] It is reported that increase in spin speed and solvent concentration both lead to a decrease in film thickness. However, this analysis was limited to film thicknesses larger than 10 µm and did not characterize surface roughness. These are important variables in the fabrication of MEMS devices, which require a paraffin thickness of 0.1–10 µm with a roughness up to 0.2 µm [16].

Given the limitations of PVD for films, spin coating of paraffin on wafers was investigated because it was a resource light, low temperature, and batch-compatible process. The goal of this project was to develop a reliable spin coating process for achieving paraffin films with thicknesses of 1.5 µm with average roughness < 10% of the thickness or 150 nm.

This work facilitates the fabrication of a new class of low-loss RF MEMS devices with high actuation force and displacement. One of the key features of the paraffin that makes it attractive for this application is the low dielectric loss tangent of the paraffin which is measured as  $6.3 \times 10^{-4}$  at 100 GHz [14]. In addition, 15% volumetric change of the paraffin provides the displacement to create variable capacitors that can achieve tunability of approximately 15% [16,17].

The fabrication process for the paraffin MEMS device is illustrated in Fig. 1 [16]. First three layers, (a)–(c), are the integrated heater, insulator and the ground plane of the device, respectively. After deposition of paraffin through spin coating, the paraffin layer is patterned using reactive ion etching (RIE). To encapsulate the paraffin layer and contain it in its liquid phase, a layer of aluminum oxide ( $\text{Al}_2\text{O}_3$ ) is deposited using atomic layer deposition. This conformal layer of aluminum oxide ensures the shape stability of the device. Last metal layer is the top electrode of the device which is fabricated by sputter deposition of aluminum. A micrograph of the fabricated device is shown

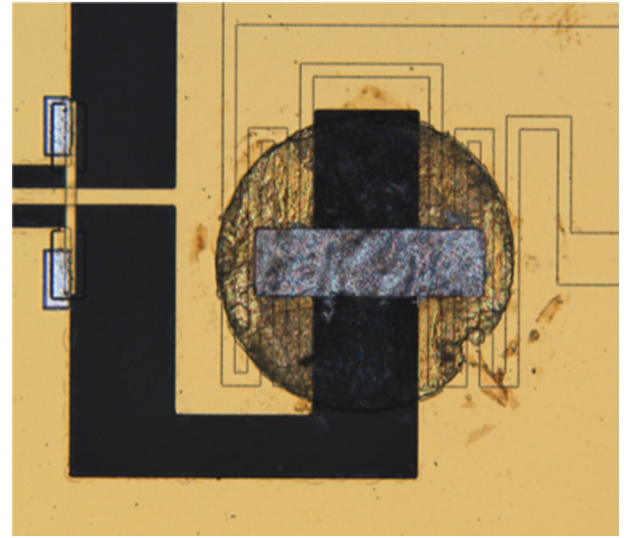


Fig. 2. Micrograph of the fabricated paraffin MEMS actuator.

in Fig. 2. Note that, in order to achieve high yield for the fabrication steps after deposition paraffin, paraffin layer needs to have minimum roughness. Adhesion of the top metal layer during the sputter deposition and etching is significantly affected by the increased roughness of the paraffin film. In addition, the losses associated with the device increases with increased roughness of the top metal layer which in turn is directly depends on the roughness of the paraffin layer.

A better understanding of the variables affecting the spin coating process allows for the development of highly tunable MEMS devices with low dielectric loss and high efficiency. This paper is organized as follows: Design of the experiment and the details of the spin coating process are described in Section 2. Experimental results for film thickness and roughness are presented in Section 3. Finally, discussion of the results and conclusion are presented in Section 4 and Section 5, respectively.

## 2. Experimental methods

The objective of this study is to develop a spin coating process to achieve paraffin films with a thickness of 1.5 µm and roughness < 10% of its thickness. Two types of paraffin were considered in this study, eicosane ( $\text{C}_{20}\text{H}_{42}$ , Sigma-Aldrich) and hexatriacontane ( $\text{C}_{36}\text{H}_{74}$ , Sigma-Aldrich). Properties of the eicosane and hexatriacontane are given in Table 1. For the first set of experiments, eicosane was used due to its

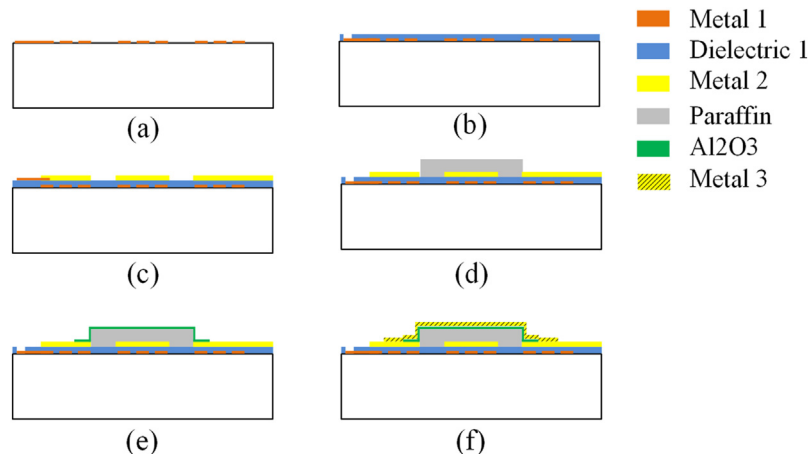


Fig. 1. Schematic of the fabrication process of the paraffin MEMS actuator; (a) heater (b) insulator (c) ground plane (d) paraffin (e) encapsulating layer (f) top metal electrode.

**Table 1**  
Physical properties of eicosane and hexatriacontane.

Material	Melting point (°C)	Molecular weight (g/mol)	Density (g/cm <sup>3</sup> )
Eicosane	35–37	282.55	0.788
Hexatriacontane	74–76	506.97	0.779

lower melting point (35–37 °C) relative to that of hexatriacontane (74–76 °C). This made it easier to maintain its liquid phase during the process and required lower operating temperatures (50–60 °C). This allowed us to develop a baseline spin coating process before moving on to hexatriacontane.

In a spin coating process, the choice of solvent plays a critical role. According to Eluru [25] and Faolin et al. [26], butanol, hexane, and p-xylene can be considered as solvents for the paraffin. In our experiments, it was observed that the two types of paraffin would not dissolve in butanol at a low temperature (< 85 °C). Also, eicosane dissolved in hexane only at low paraffin concentrations (< 10%). Additionally, it is observed that solubility of hexatriacontane in hexane is not significant. On the other hand, both paraffin types dissolved in xylene at temperatures lower than 85 °C making p-xylene the solvent of choice.

Paraffin samples are spin coated on three types of substrate, all 100-mm in diameter; Silicon, Pyrex and 500 nm-thick gold coated Pyrex wafers. For the spin coating process, a solution is prepared by mixing the paraffin flakes with p-xylene. The solution is stirred and heated up to 80 °C. The solution container is also capped with an aluminum foil to minimize the evaporation of p-xylene. Substrates are also heated to maintain paraffin in the liquid phase during the initial steps of spin coating. Paraffin solution is dispensed using glass pipettes which are kept at the same temperature as the paraffin solution. For the spin coating, substrate is manually transferred from hot plate to the spinner. Spin coating is performed in two steps; first, the solution is spread at 500 rpm. Then, the spin speed ranging from 800 to 6000 rpm with an acceleration of 5000 rpm/s is employed. Samples are inspected under microscope and average thickness and roughness of the samples are measured using a stylus surface profilometer (Veeco Dektak 3). For the thickness and roughness measurements, wafers are scratched in five locations to create a sharp step from the uncoated substrate to the surface of the paraffin layer.

To investigate the effects of the spin speed, substrate temperature, solution concentration, and substrate material, four sets of experiments are carried out which are detailed in Table 2.

### 3. Results

#### 3.1. Characterization of eicosane films

In this section, spin coating of eicosane onto 100-mm-diameter (4 in.) silicon substrate is investigated. The goal of this set of experiments is to determine the general trends associated with spin speed, substrate temperature. This allowed us to streamline the number of experiments conducted in spin coating hexatriacontane.

Fig. 3 shows the effect of spin speed on the average thickness and roughness of the paraffin film. The conditions of this set of experiments are listed in Table 2(a). As expected, the data indicates that increasing the spin speed results in lower thickness and roughness values. Measurement data is fitted to a power series using MATLAB Curve Fitting Toolbox. The thickness of the paraffin is proportional to  $1/\omega^{0.42}$  and the roughness varies as  $1/\omega^{0.75}$ .

The effect of the substrate temperature on the average film thickness and roughness at two different spin times can be seen in Fig. 4. The conditions of this set of experiments are listed in Table 2(b). The data indicates that increasing the substrate temperature will lead to a lower average film thickness and roughness. As shown in Fig. 4, the thickness

**Table 2**  
Experiment setup parameters.

(a) Effect of spin speed	
Variables	
Spin speed	800–600 rpm
Fixed parameters	
Parffin type	Eicosane
Wafer type	Silicon
Spin time	60 s
Solution temperature	≈ 50–60 °C
Substrate temperature	120 °C
Paraffin concentration	40% (v/v)
(b) Effect of substrate temperature	
Variables	
Substrate temperature	60–160 °C
Fixed parameters	
Parffin type	Eicosane
Wafer type	Silicon
Spin time	60 s
Solution temperature	≈ 50–60 °C
Paraffin concentration	40% (v/v)
Spin speed	4000 rpm
(c) Effect of hexatriacontane concentration	
Variables	
Paraffin concentration	10–40% (v/v)
Fixed parameters	
Parffin type	Hexatriacontane
Wafer type	Silicon
Spin time	60 s
Solution temperature	≈ 75–85 °C
Substrate temperature	160 °C
Spin speed	4000 rpm
(d) Effect of wafer type	
Variables	
Wafer type	Silicon, Gold, Pyre
Spin speed	2000–4000 rpm
Fixed parameters	
Parffin type	Hexatriacontane
Spin time	60 s
Solution temperature	≈ 75–85 °C
Substrate temperature	160 °C
Paraffin concentration	10% (v/v)

of the film decreases with exponential rate of  $7.1 \times 10^{-3}$  with respect to the substrate temperature. Thickness of the films ranged from 3 μm with a roughness of 0.4 μm at a substrate temperature of 50 °C to a thickness of 1 μm with a roughness of 0.1 μm at a substrate temperature of 150 °C. A paraffin solution would crystallize quicker on a substrate of a lower temperature which would give less time for the spin coater to remove excess paraffin and solvent. Hence lower thickness and roughness values are obtained. The data shows no significant effect on the thickness and the roughness when spin time is increased. This indicates that the paraffin solution is probably solidified before the 60 s mark, meaning that a spin time longer than 60 s would have no effect.

#### 3.2. Characterization of hexatriacontane films

Upon acquiring preliminary results from spin coating of eicosane, we conducted experiments on hexatriacontane. The variables tested were concentration, spin speed, and substrate type. Fig. 5 shows the effect of the paraffin concentration on the average film thickness. The conditions of this set of experiments are listed in Table 2(c). The data

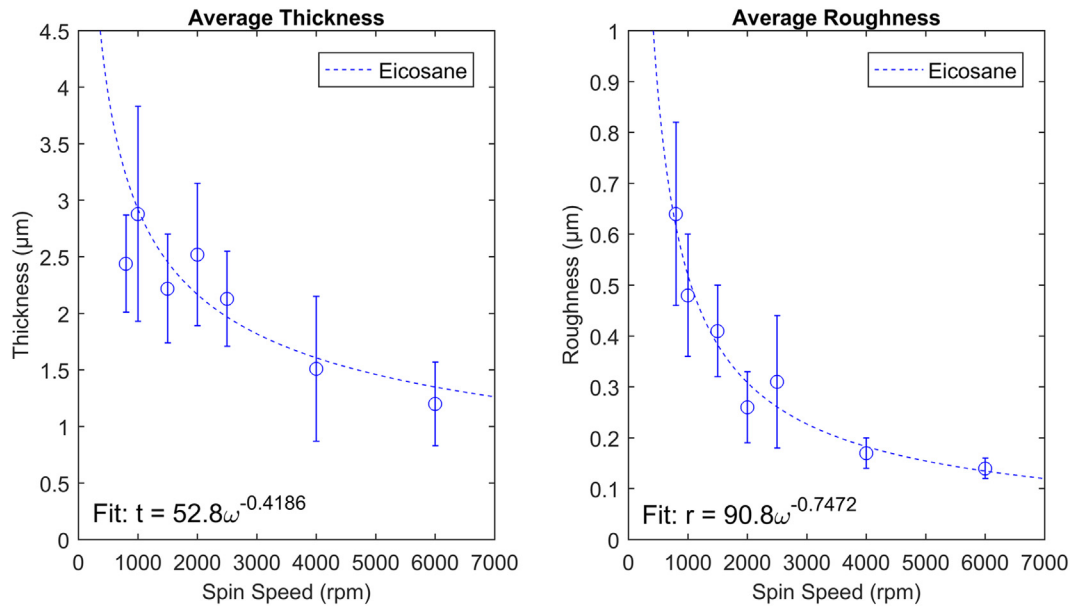


Fig. 3. Effect of spin speed on film thickness and uniformity of eicosane film on silicon substrates based on conditions listed in Table 2(a).

indicates that decreasing the paraffin concentration yields thinner and smoother films with a thickness of 2  $\mu\text{m}$  and a roughness of 0.25  $\mu\text{m}$ . It also shows that decreasing the paraffin concentration leads to less variation in film thickness. The standard deviation of the thickness as a percent of the film thickness is 27% at a 40% paraffin solution and 12% at a 10% paraffin solution. It also shows that decreasing the paraffin concentration leads to less variation in film roughness. The standard deviation of the roughness as a percent of the film roughness is 49% at a 40% paraffin solution and 27% at a 10% paraffin solution. The data is consistent with our expectations because the solution viscosity, which decreases with decreasing paraffin concentration, assists the spin coater in removing excess paraffin. Fitted thickness data shows that thickness is proportional to  $C^{1.35}$  where  $C$  is the volumetric concentration of the paraffin.

A paraffin concentration of 10% was chosen as the baseline for the next set of trials since values close to the desired film thickness and roughness were obtained at that concentration. Next, effect of the spin

speed and substrate type on the film thickness and roughness is investigated.

The effect that the choice of substrate has on film thickness and roughness can be seen in Fig. 6. The conditions of this set of experiments are listed in Table 2(d). The results show the correlation between substrate choice and film thickness and roughness. The data shows that at a spin speed of 5000 rpm, a thickness < 1.5  $\mu\text{m}$  and a roughness < 0.15  $\mu\text{m}$  have been achieved for all substrate types tested. While increasing the spin speed results in a lower film thickness and roughness values, the rates at which this occurs varies between substrate materials.

Overall thickness of the film on gold-coated Pyrex substrate is higher than the silicon and film on the bare Pyrex substrate has the lowest thickness. Gold coated substrate has the highest thermal conductivity, hence heat dissipation and paraffin crystallization are faster. As a result the paraffin thickness is higher. On the other hand, bare Pyrex substrate has the lowest thermal conductivity, therefore

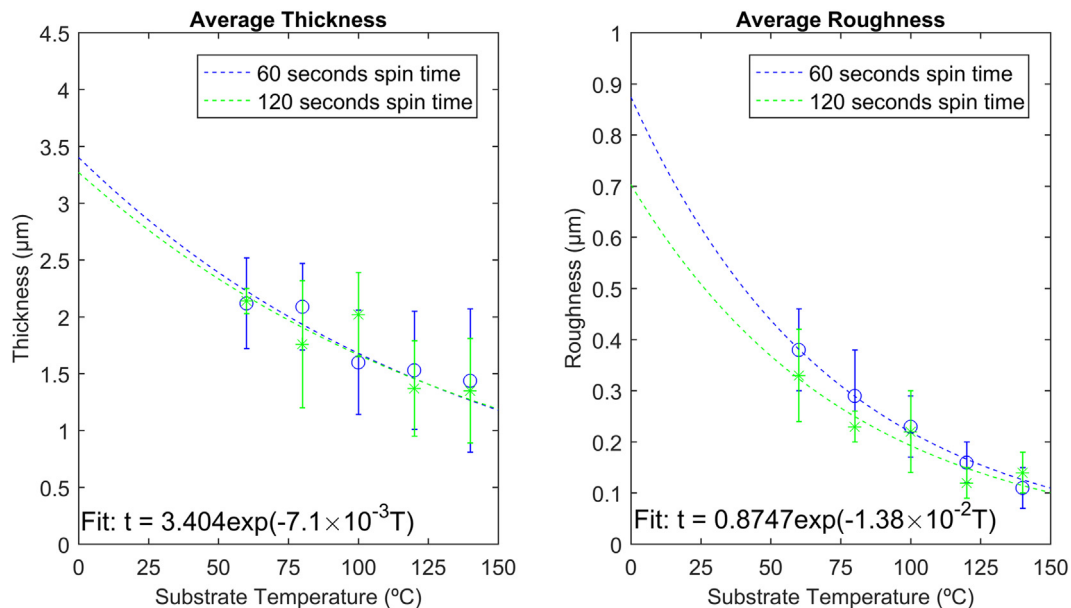


Fig. 4. Effect of substrate temperature and spin time on film thickness and uniformity of eicosane film on silicon substrates based on conditions listed in Table 2(b).

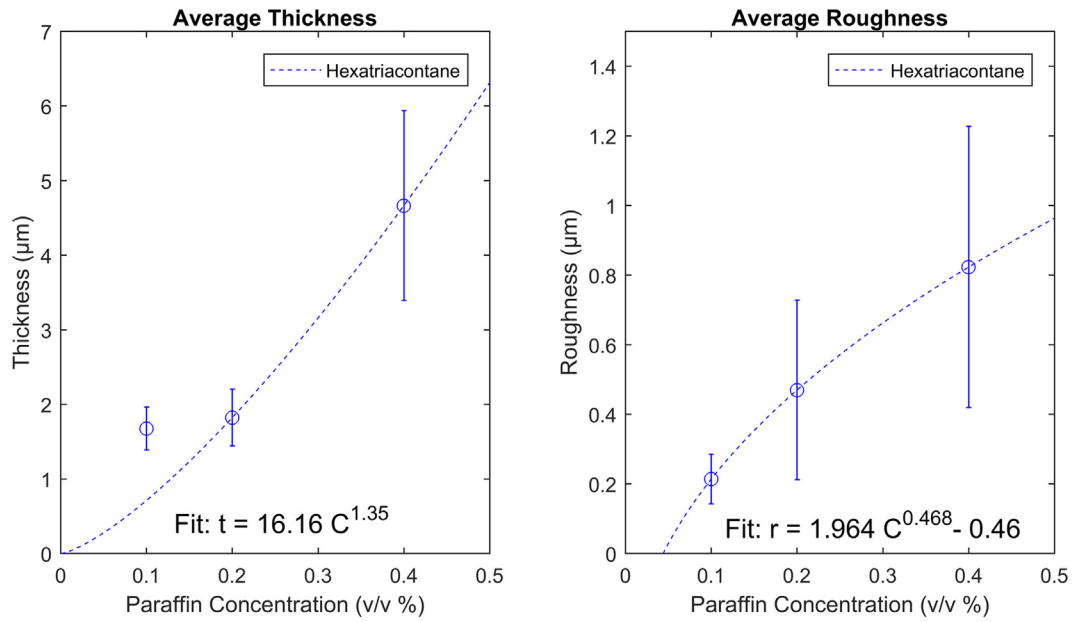


Fig. 5. Effect of paraffin concentration on film thickness and uniformity of hexatriacontane film on silicon substrates based on conditions listed in Table 2(c).

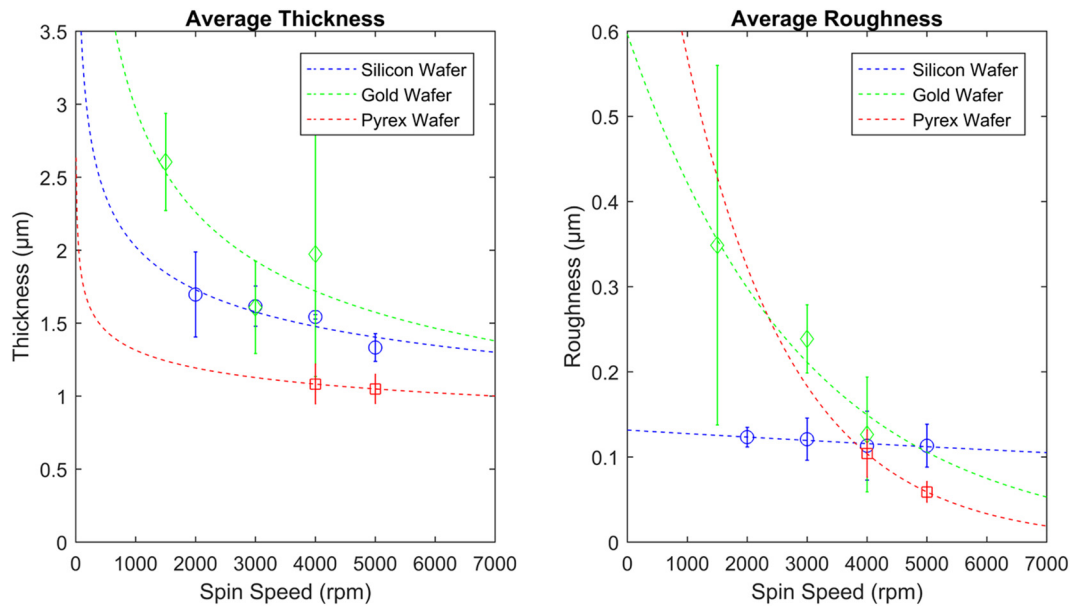


Fig. 6. Effect of substrate type on film thickness and uniformity of hexatriacontane film on silicon, gold-coated silicon, and Pyrex substrates based on conditions listed in Table 2(d).

maintains the heat for a longer period of time during spin coating. Consequently, paraffin film has a lower thickness.

Films deposited on silicon, gold, and Pyrex substrates at 4000 rpm were viewed under an optical microscope. Fig. 7 provides a qualitative view of the film from the center to the edge. The paraffin morphology appears to be consistent throughout each wafer but does appear to vary across different wafer types in terms of cracks and other imperfections.

#### 4. Discussion

The effect of multiple variables such as spin speed, spin time, substrate temperature, solution concentration, and substrate type on paraffin fabrication process allowing for control over desired film characteristics is studied. This lays a solid basis for the fabrication of a diverse range of novel paraffin-based MEMS devices. Two paraffin types, eicosane and hexatriacontane, were dissolved in a p-xylene and

were spin coated onto multiple wafers. Consequently, a paraffin film with the desired thickness of  $1.5 \mu\text{m}$  and a roughness of about 10% of the surface thickness is achieved. Moreover, paraffin films with a thickness as low as  $1 \mu\text{m}$  and roughness of  $0.1 \mu\text{m}$  are fabricated. Also, the effect of the substrate type on film thickness and roughness is characterized.

The surface roughness of bare silicon, Pyrex, and gold-coated substrates are  $< 2 \text{ nm}$  which is much smaller than the roughness values for the paraffin films and were ignored in the analysis of the data.

In this process, there were limiting factors that could have impacted the accuracy of our results. One of factors is the heat loss during the manual transfer of the substrate and the solution from hot plate to the spin coater. The transfer time is minimized as much as possible to keep the temperature of the substrate and the solution constant. Another limiting factor is the variation of the paraffin concentration due to the evaporation of p-xylene. This problem is addressed by making a fresh



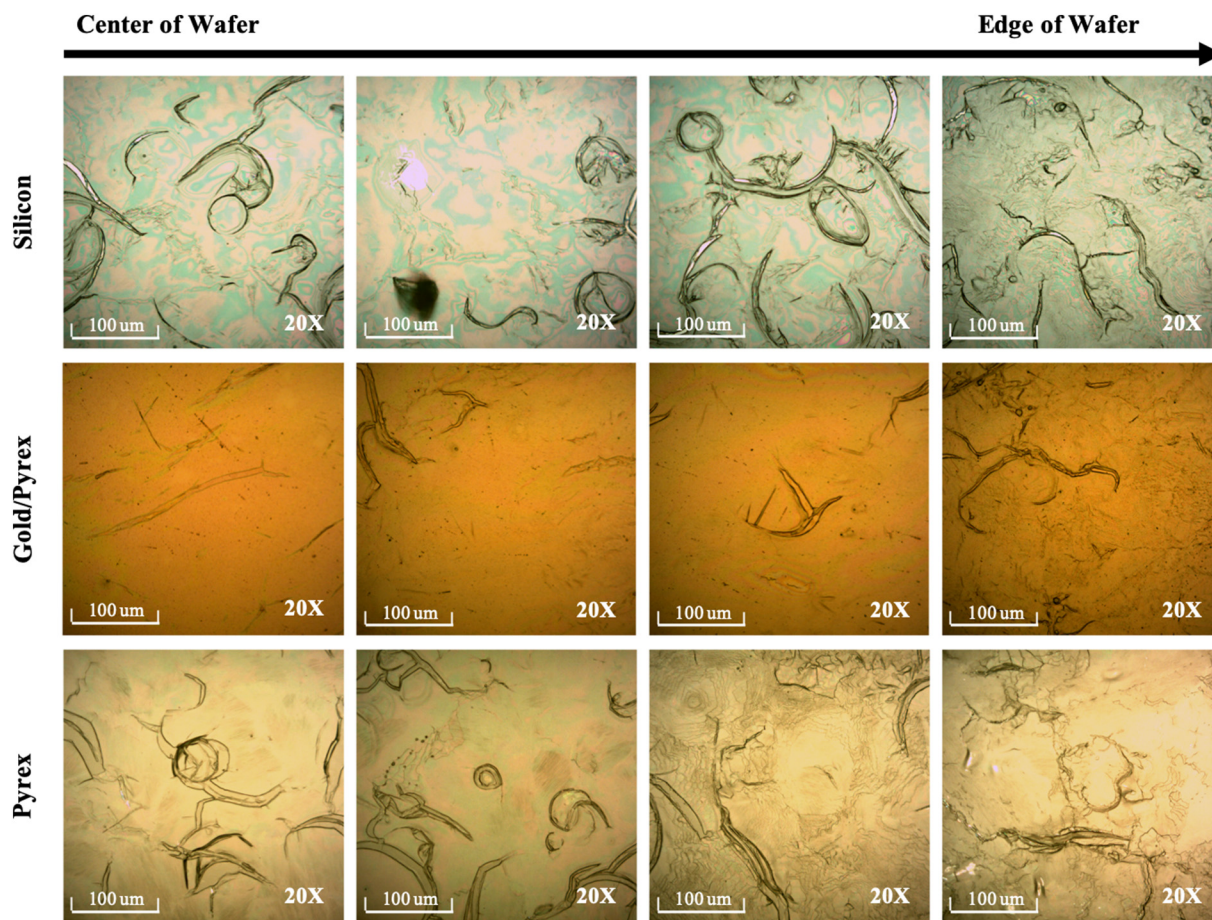


Fig. 7. Optical microscopy images of paraffin films on different substrates at 10% paraffin solution spun at 4000 rpm based on conditions listed in Table 2(d).

solution for every batch of samples. Additionally, it was observed that the center of the substrate cooled down faster than the outside of the substrate. This occurred because the center of the substrate was subject to a vacuum, which held it in place during the spin coating. This resulted in rougher films on the center of the wafer and smoother films on the outer edges. Lastly, imaging the film under a microscope revealed the presence of several cracks in the film. Cracks in the film are created due to the crystallization of paraffin and since the actuation force in MEMS devices is controlled by phase changes in the encapsulated paraffin layer, we do not expect the cracks to impact the functionality of the MEMS device.

## 5. Conclusion

A batch compatible, cost-effective, and resource-light spin coating process for the fabrication of paraffin films is presented. Paraffin is an attractive candidate for the design of MEMS devices due to its unique mechanical and electrical properties. Two types of paraffin, eicosane and hexatriacontane, were dissolved in p-xylene and deposited on silicon, Pyrex and gold-coated substrate. This study explored the effect of spin speed, spin time, substrate temperature, solution concentration, and substrate type on the thickness and roughness of the spin coated films.

A baseline spin coated process is developed for both paraffin types at 4000 rpm. For eicosane, a film thickness of  $1.6\ \mu\text{m}$  with a roughness of  $0.18\ \mu\text{m}$  is achieved at a paraffin concentration of 40%, substrate temperature of  $120\ ^\circ\text{C}$ , solution temperature around  $50\text{--}60\ ^\circ\text{C}$ , and spin time of 60 s. For hexatriacontane, a film thickness of  $1.5\ \mu\text{m}$  with a roughness of  $0.11\ \mu\text{m}$  is achieved at a paraffin concentration of 10%, substrate temperature of  $160\ ^\circ\text{C}$ , solution temperature in the range of

$75\text{--}85\ ^\circ\text{C}$ , and spin time of 60 s.

This process expands on earlier spin coating studies by outlining a process for achieving film characteristics suitable for MEMS devices that typically require micron-range thickness. Furthermore, this work provides a better understanding of variables affecting paraffin films and is important for the development of MEMS devices with low dielectric and insertion loss, and high actuation force and displacement.

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## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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