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PARYLENE CAPPING LAYER FOR EMBEDDED LIQUID MASS FOR MEMS PACKAGING

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ABSTRACT
Microelectromechanical Systems (MEMS) packaging is over 80% of the cost of a typical MEMS device because there are no standard packaging methods, and each device requires unique packaging. Recently several MEMS devices have illustrated the desire to have a liquid filled cavity within the MEMS device for applications such as biomedical sensors, tunable energy harvesters, or liquid cooling microelectronics. However, embedded liquids in silicon pose a challenge when it comes to packaging. This paper illustrates a novel concept of using a conformal parylene coating to cap or encapsulate the liquid. The concept is validated using various liquids such as various viscosity silicone oils as well as Galinstan a Ga-based liquid metal. The study investigates the packaging reliability through a series of systematic accelerated life-time testing, elevated temperature testing, accelerated soak testing, and mechanical testing (shock and resonant frequency testing). Mass changes were monitored and compared to control (no capping), glass epoxy bonded packaging, and silicone spray coating encapsulation. The results demonstrate the superior mean-time-to-failure of the parylene capping method compared to the other methods. The results confirm that parylene can be used to package embedded liquids in silicon or 3D printed structures.

Keywords: Microelectronics, MEMS, Packaging, Parylene, Embedded Liquid

1. INTRODUCTION
Microelectromechanical Systems (MEMS) packaging along with microelectronics packaging is a critical component to the performance of the device, and for MEMS packaging there are no standard packaging techniques as each device has specific packaging needs [1]. Some MEMS and microelectronics devices could benefit from the potential to embed liquid inside of the device, but to date this concept has been limited by the high packaging challenges associated with embedded liquids. Applications including microfluidics such as lab on chip [2], microelectronic cooling using flowing liquids [2, 3], microthrusters [4], atomizers [5, 6] all require a packaging method that allows liquid to be embedded in micro-channels without leakage. Recently energy harvesters have also demonstrated advantages when integrating embedded liquids to increase bandwidth [7-10] or tuning resonant frequencies [11-13]. However, so far these types of devices are limited to research labs as packaging embedded liquids is challenging as leakage can lead to catastrophic failure for the device and its electronics. These applications could be used for anything from implants to consumer electronics. Therefore, the packaging needs to be able to withstand leak testing but also it must be able to withstand vibrations (especially for kinetic energy harvesters).

Typical, packaging for these types of devices includes glass or polymer cap bonding or silicone encapsulation packaging but these are not ideal as they often require thick layers adding to the mass of the system, which for energy harvesters can significantly alter the resonant frequency and affect performance.

Parylene is a conformal chemical vapor deposited polymer that is deposited at room temperature. It has numerous advantages over other polymers such as: pinhole free, low water absorption, relatively good thermal properties (dependent on type of parylene), thickness range from 200 nm to 50 µm, transparency etc… It has also been widely used previously in MEMS packaging [14-16] as well as encapsulation layers [17-19]. It has also been used previously as a capping layer for embedded nanomagnets in silicon [20-24]. Recent efforts have demonstrated its potential use in packaging implantable pressure sensors covered in high viscosity silicone oil [19, 25, 26].

This study investigates the feasibility of creating a thin-film parylene-based capping layer for embedded liquids. The study investigates various liquids from silicone oil with varying viscosities to gallium-based liquid metals. We investigated the effects of concavity and filling ratios. The study within the paper also investigates the reliability of the packaging by putting various test devices under various accelerated air evaporation testing, soak testing, as well as mechanical vibration testing.

2. MATERIALS AND METHODS

2.1 Concept

The concept of using a conformal coating of parylene to encapsulate a pressure sensor soaked in silicone oil was previously investigated for implantable applications [25, 26]. However, this study involves embedding liquids into a silicon cavity, and capping the entire cavity in parylene to prevent liquid leakage. The basic concept is illustrated in Figure 1.

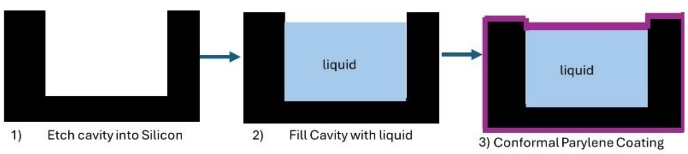

FIGURE 1: SCHEMATIC OF THE CONCEPT OF FILLING A CAVITY WITH LIQUID AND USING PARYLENE AS A CAPPING OR ENCAPSULATION LAYER FOR EMBEDDED LIQUID PACKAGING.

2.2 Fabrication Process

Silicon wafers were used to create the test structures, but 3D printed structures could also be used. Oxide was grown on the wafers and patterned to have 2 x 2 x 0.05 cm square cavities. The cavities were created using Deep Reactive Ion etching (DRIE) using the parameters previously described [27] and shown in Fig. 1a. Then liquid was dispensed into the cavity. Various liquids were investigated, consisting of four different viscosities of silicone oil (Sigma Aldrich) (100, 1k, 10k, and 100k cP). In addition to silicone oil Galinstan a Ga-based liquid metal was also used to illustrate the potential to encapsulate an electrically conducting liquid with large density (>6 g cm^{-3}) and low viscosity (~ 2 cP). The cavities were filled to varying thicknesses ranging from 20-100% fill volume of the cavity.

Parylene-C was deposited using the Gorham methods using an (SCS Labcoater) as previously described [28]. Parylene-C was selected as it is the most common type of parylene and the deposition rate is higher than other parylenes allowing for thicker layers. Thickness of 20 µm was deposited. A silane (A-174) adhesion layer was coated on the substrates. A pressure of 25 mT was used to deposit the Parylene.

Control samples consisted of no packaging or capping layer, only liquid filled cavities. To compare the reliability of the parylene encapsulation two other methods were investigated. The first method used glass slides that were bonded to the liquid filled cavities using a room temperature cured epoxy to bond the glass to the silicon cavity. The other method investigated was the use of silicone encapsulation (Q-ballz Smooth On), which was spray coated on the liquid filled cavities in n=5 layers for a total thickness of approximately 100 µm.

2.3 Reliability Testing

2.3.1 Evaporation Testing

Air evaporation testing was performed at three different temperatures (room temperature (~20°C, 50°C, and 80°C). The elevated temperature of 50°C was selected as it is approximately the glass transition temperature of Parylene-C. However, since silicone oil has a very slow evaporation rate at room temperature accelerated testing was performed up to 80°C [29]. The evaporation testing was performed in an oven with an air environment.

Twelve cavities of each liquid were filled (4 x 3 array) (cavities were filled between 20-100%). The reason for the variation of filling was to determine if the parylene capping layer would create a solid capping film on various amounts of filling. Liquids investigated were silicone oil (100, 1,000, 10,000, and 100,000 cP) were filled and coated with parylene as described above. Other test devices consisted of a control (no capping layer filled with each type of silicone oil (3 cavities each). A test structure with a silicone encapsulation (spray deposited), and a glass slide epoxy bonded capping layer was also tested. Testing was performed by weighing the test structures and determining any change in weight due to evaporation.

Accelerated temperature testing was performed to calculate the acceleration factor using the Arrhenius equation (1):

$$AF = e^{\left[\frac{E_a}{k}\left(\frac{1}{T_1}-\frac{1}{T_2}\right)\right]} \quad (1)$$

Where AF is the acceleration factor, k is the Boltzman constant, T_1 and T_2 are the lower and higher temperatures in K, and E_a is the activation energy, which for the evaporation study was -0.7 eV [30]. The AF was used to determine the mean time to failure (MTTF).

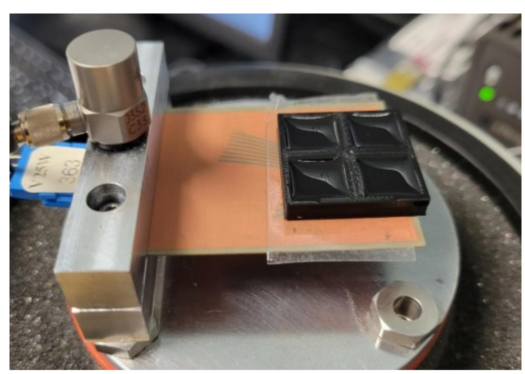

FIGURE 2: MECHANICAL RESONANT FREQUENCY TESTING IMAGE SHOWING ACCELEROMETER, CANTILEVER, AND 2 X

2 CAVITY TEST STRUCTURE WITH SILICONE OIL AND PARYLENE CAPPING LAYER.

2.3.2 Soak Testing

Soak testing was performed by submerging the test structures in water that was heated to 80°C for accelerated testing. Testing structures consisted of the same type as used in air evaporation testing. However, we also investigated parylene capping of liquid metal (Galinstan) as this has been used in numerous electronic applications as it is liquid at room temperature. Test structures were weighed to determine any change in weight caused from leaking or absorption.

2.3.3 Mechanical Testing

Mechanical reliability testing was performed by putting a 2 x 2 cavity array test structure with silicone oils and various capping methods on the end of a piezoelectric cantilever as shown in Figure 2. Two different tests were performed using an electrodynamic vibration shaker. The first test consisted of shaking the test structure and cantilever at resonant frequency for 1 day at various accelerations and then measuring their change in mass to determine if there was any leakage. The second test performed was a shock test which consisted of impulse shocks from 0-50 g.

3. RESULTS AND DISCUSSION

Figure 3 illustrates the ability to create a parylene-C capping layer on various liquids from low viscosity silicone oil (100-1000 cP) (Fig. 3a), which illustrates a smooth parylene layer that is near wrinkle free. The figure shows capping of convex and concave films for various filling ratios. Figure 3b illustrates the ability to create a parylene capping film on top of a viscous 100k cP silicone oil. The image shows significant wrinkling due to stress of the film [25, 31], but a solid parylene film was still created. Figure 3c illustrates the ability to create a parylene capping layer on Galinstan (liquid metal), even when the liquid metal did not completely fill the cavity the parylene was conformally coated to encapsulate the metal, thus reducing the parylene volume. Figure 3d shows the silicone spray encapsulation film covering the silicone oil.

Room temperature evaporation testing results are demonstrated in Figure 4. The results demonstrate that parylene capping test structures had no significant change in mass over the 50-day testing period indicating there was no significant evaporation of the liquids. Although silicone oil has a low evaporation rate the control with no capping layer demonstrated significant decrease in mass (>5%) after 16 days, and it continued to decrease over time reaching nearly 13% decrease over the 50 days. The spray on silicone encapsulation film test devices also demonstrated a significant decrease of up to 4.2% over 50 days. The glass packaging films had similar results as the parylene capping method.

The elevated temperature air evaporation testing results are demonstrated in Figure 5. The results show a significant decrease in mass (~30% at 80°C (18 days) and 15% at 50°C (30 days)). The silicone encapsulation and glass packages also had significant decrease in mass. The glass test structures were bonded with low temperature epoxy which likely melted at high temperatures, which could potentially be resolved by using higher temperature grade epoxy. However, the parylene-capped test structures showed no significant change in mass. The 100 cP silicone oil did show a 0.9% decrease in mass at 18 days of 80°C whereas the 100 kcP oil only showed 0.5% decrease in mass under the same test. This demonstrates that high viscosity liquids with parylene capping are likely to have a longer lifetime.

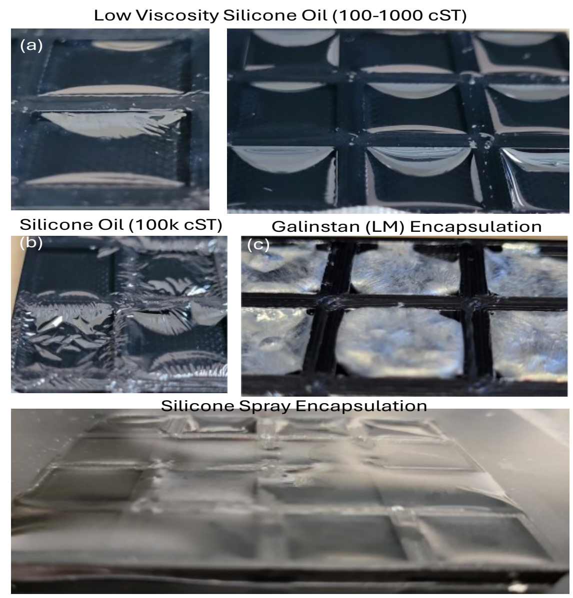

FIGURE 3: IMAGE ILLUSTRATING PARYLENE-C CAPPING LAYER CAPABILITY ON VARIOUS LIQUIDS AND VISCOSITIES.

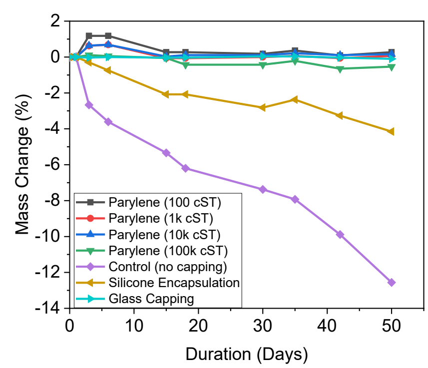

FIGURE 4: AIR EVAPORATION RESULTS FOR 20°C TESTING ILLUSTRATING MASS CHANGES AS A FUNCTION OF TIME.

Using the Arrhenius equation and the criteria that a 5% loss is considered failure, we predict the MTTF to be approximately 51 years at room temperature. More samples and longer testing periods are required to get a more accurate estimation. Images of the testing structures after 80°C air testing are shown in Figure 6. The parylene capped structures look similar to the initial images, but the glass capped packages demonstrate air bubbles indicating leaks in the bonds, and the silicone spray encapsulation packaging likely dissolved which led to leakage.

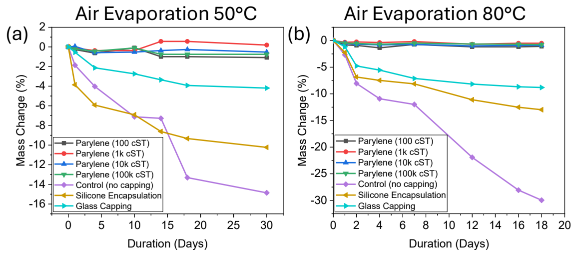

FIGURE 5: EVAPORATION RESULTS FOR ELEVATED TEMPERATURE TESTING ILLUSTRATING MASS CHANGES AS A FUNCTION OF TIME.

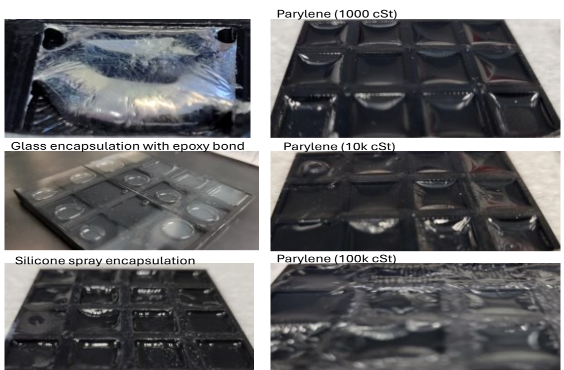

FIGURE 6: IMAGES OF THE TEST STRUCTURES AFTER 80°C TESTING.

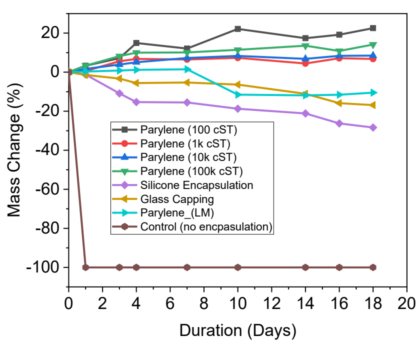

FIGURE 7: SOAK TESTING AT 80°C WITH MASS CHANGES AS A FUNCTION OF TIME.

Elevated temperature soak testing results are demonstrated in Figure 7. The control cavities had catastrophic failures in all of the cavities as expected as there was no protection layer to prevent the leakage of the silicone oil. As with the elevated temperature air testing the glass and silicone encapsulation demonstrated significant decrease leading to failures, which is likely due to the heat. The Liquid metal with parylene coating had 1 of the 12 cavities failed at day 9 which shows a significant loss in mass, but afterwards the mass held constant. The cavity that failed had a tear in the parylene which caused the LM to leak out completely but only from 1 cavity. Most of the parylene-capping test structures demonstrated an increase in mass by up to 21% for 100 cP and 6% for higher viscosity silicone oils. The reason for the increase in mass is likely due to water absorption of the parylene, as the mass increase happened early in the testing and then saturated. However, there was no significant decrease in mass, this means that the parylene capping did not show any signs of silicone oil leakage during the test.

Images of the capping layers after the 18-day soak test are shown in Figure 8. The image shows significant warpage and wrinkles in all the films which is likely due to the coefficient of thermal expansion mismatch between the parylene (35 ppm/°C) compared to silicone oil (1% /°C). However, since parylene-C has an elongation to break of approximately 200% the film can stretch as illustrated in the image. However, the wrinkles in the parylene-capping layer did not seem to affect its leakage performance in silicone oil and only 1 cavity failure occurred for liquid metal.

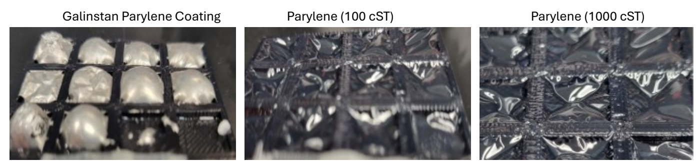

FIGURE 8: IMAGE OF PACKAGES AFTER 80°C SOAK TEST (18 DAYS).

Mechanical testing results are important to determine if the parylene capping film can withstand vibrations and shocks without delaminating or tearing. The results for both tests are shown in Figure 9. Resonant frequency testing at various accelerations illustrates that the control sample without capping showed a gradual decrease in mass at various accelerations, and above 1.75 g's the silicone oil was completely removed from the cavity, but significant leakage (>5% in mass) was demonstrated at 0.4 g. The viscosity of the liquid does have a significant influence as the 100 cP silicone oil failed at lower acceleration than the 100k cP (>5% at 1 g). The spray on silicone encapsulation test structures also failed at 1.5 g. However, the parylene-capped structures with 100 and 100k cP silicone oil demonstrated no significant change in mass or resonant frequency up to 2 g. Higher accelerations were not possible as it would have broken the cantilever.

Shock testing of the 100 cP silicone oil and parylene-capping film demonstrated no significant leakage with only 0.53% loss in mass at 50g. Higher acceleration testing was not possible on our system.

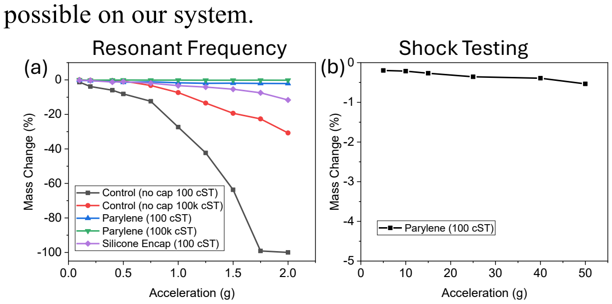

FIGURE 9: RESULTS OF THE MECHANICAL RELIABILITY TESTING AT RESONANT FREQUENCY (A) AND SHOCK (B).

4. CONCLUSION

This study investigated a novel method of embedding liquids into cavities while protecting them from leakage using a parylene capping film. Parylene-C was used in this study due to the ability to apply a thicker layer, but varying thickness was not investigated in this study. In addition, higher temperature parylene could be used in the future to avoid wrinkles and thermal mismatches, which could be necessary for high-temperature operations. However, based on the results parylene-C should be a good capping layer for room-temperature applications. In summary a parylene capping film was successfully deposited onto various liquids and liquids with various viscosities. The capping layer held up to accelerated temperature testing in air and submerged soaking as well as mechanical testing. This indicates that this could be a very effective packaging method for preventing liquid leakage in MEMS packaging.

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REFERENCES

[1] K. Najafi, "Micropackaging technologies for integrated microsystems: Applications to MEMS and MOEMS," in *Micromachining and Microfabrication Process Technology VIII*, 2003, vol. 4979: SPIE, pp. 1-19.

[2] H. Mansoorzare *et al.*, "A microfluidic MEMS-microbalance platform with minimized acoustic radiation in liquid," *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control,* vol. 67, no. 6, pp. 1210-1218, 2019.

[3] R. Li *et al.*, "Enhancement of cooling performance in MEMS by modifying 3D packaging structure: A design, integration, analysis and test study," *Applied Thermal Engineering,* vol. 237, p. 121758, 2024.

[4] D. Fontanarosa, C. De Pascali, M. G. De Giorgi, P. Siciliano, A. Ficarella, and L. Francioso, "Fabrication and embedded sensors characterization of a micromachined water-propellant vaporizing liquid microthruster," *Applied Thermal Engineering,* vol. 188, p. 116625, 2021.

[5] P. Sharma, M. Quazi, I. R. Vazquez, and N. Jackson, "Investigation of droplet size distribution for vibrating mesh atomizers," *Journal of Aerosol Science,* vol. 166, p. 106072, 2022.

[6] P. Sharma, I. R. Vazquez, and N. Jackson, "Atomization of High Viscous Liquids Using a MEMS Vibrating Mesh With Integrated Microheater," *Journal of Microelectromechanical Systems,* 2023.

[7] R. Somkuwar, J. Chandwani, and R. Deshmukh, "Bandwidth widening of piezoelectric energy harvester by free moving cylinders in liquid medium," *Microsystem Technologies,* vol. 27, pp. 1959-1970, 2021.

[8] N. Jackson and F. Stam, "Sloshing liquid-metal mass for widening the bandwidth of a vibration energy harvester," *Sensors and Actuators A: Physical,* vol. 284, pp. 17-21, 2018.

[9] N. Jackson, F. Stam, O. Z. Olszewski, H. Doyle, A. Quinn, and A. Mathewson, "Widening the bandwidth of vibration energy harvesters using a liquid-based non-uniform load distribution," *Sensors and Actuators A: Physical,* vol. 246, pp. 170-179, 2016.

[10] K. J. Kim, J. Kim, and D. Kim, "Slosh-induced piezoelectric energy harvesting in a liquid tank," *Renewable Energy,* vol. 206, pp. 409-417, 2023.

[11] A. Doria, E. Marconi, and F. Moro, "Tuning of vibration energy harvesters by means of liquid masses," in *2021 Sixteenth International Conference on Ecological Vehicles and Renewable Energies (EVER)*, 2021: IEEE, pp. 1-7.

[12] N. Jackson, "Tuning and widening the bandwidth of vibration energy harvesters using a ferrofluid embedded mass," *Microsystem Technologies,* vol. 26, no. 6, pp. 2043-2051, 2020.

[13] R. Adhikari, V. Karimi, and N. Jackson, "Passive Frequency Tuning Using Liquid Distributed Load," in *ASME International Mechanical Engineering Congress and Exposition*, 2023, vol. 87691: American Society of Mechanical Engineers, p. V012T13A024.

[14] J. H.-C. Chang, Y. Liu, D. Kang, and Y.-C. Tai, "Reliable packaging for parylene-based flexible retinal implant," in *2013 Transducers & Eurosensors XXVII: The 17th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS & EUROSENSORS XXVII)*, 2013: IEEE, pp. 2612-2615.

[15] J. Ortigoza-Diaz *et al.*, "Techniques and considerations in the microfabrication of Parylene C microelectromechanical systems," *Micromachines,* vol. 9, no. 9, p. 422, 2018.

[16] H.-s. Noh, K.-s. Moon, A. Cannon, P. J. Hesketh, and C. Wong, "Wafer bonding using microwave heating of parylene for MEMS packaging," in *2004 Proceedings. 54th Electronic Components and Technology Conference (IEEE Cat. No. 04CH37546)*, 2004, vol. 1: Ieee, pp. 924-930.

[17] S. Buchwalder, F. Bourgeois, J. J. D. Leon, A. Hogg, and J. Burger, "Parylene-AlOx Stacks for Improved 3D Encapsulation Solutions," *Coatings,* vol. 13, no. 11, p. 1942, 2023.

[18] J. Maeng, B. Kim, D. Ha, and W. J. Chappell, "Parylene interposer as thin flexible 3-D packaging enabler for wireless applications," *IEEE transactions on microwave theory and techniques,* vol. 59, no. 12, pp. 3410-3418, 2011.

[19] A. Shapero, Y. Liu, and Y.-C. Tai, "Parylene-on-oil packaging for implantable pressure sensors," in *2016 IEEE 29th International Conference on Micro Electro Mechanical Systems (MEMS)*, 2016: IEEE, pp. 403-406.

[20] N. Jackson, "PiezoMEMS Nonlinear Low Acceleration Energy Harvester with an Embedded Permanent Magnet," *Micromachines,* vol. 11, no. 5, p. 500, 2020.
[21] O. D. Oniku, B. J. Bowers, S. B. Shetye, N. Wang, and D. P. Arnold, "Permanent magnet microstructures using dry-pressed magnetic powders," *Journal of Micromechanics and Microengineering,* vol. 23, no. 7, p. 075027, 2013.
[22] B. Ma, A. Sun, X. Gao, X. Bao, and J. Li, "Preparation of parylene-coated bonded NdFeB magnets," *Journal of Magnetism and Magnetic Materials,* vol. 467, pp. 114-119, 2018.
[23] Z. Guler and N. Jackson, "Magnetic Proof Mass Configurations to Reduce Spatial Dependency of Piezomagnetic AC Current Sensors," *IEEE Sensors J.,* 2024.
[24] N. Jackson, F. J. Pedrosa, A. Bollero, A. Mathewson, and O. Z. Olszewski, "Integration of thick-film permanent magnets for MEMS applications," *Journal of Microelectromechanical Systems,* vol. 25, no. 4, pp. 716-724, 2016.
[25] A. Shapero and Y.-C. Tai, "Parylene-oil-encapsulated low-drift implantable pressure sensors," in *2018 IEEE Micro Electro Mechanical Systems (MEMS)*, 2018: IEEE, pp. 47-50.
[26] A. M. Shapero, Y. Liu, and Y.-C. Tai, "Parylene-on-oil packaging for long-term implantable pressure sensors," *Biomedical microdevices,* vol. 18, pp. 1-10, 2016.
[27] P. Sharma, M. Pleil, and N. Jackson, "Effect of SF6 and C4F8 Flow Rate on Etched Surface Profile and Grass Formation in Deep Reactive Ion Etching Process," in *ASME International Mechanical Engineering Congress and Exposition*, 2022, vol. 86717: American Society of Mechanical Engineers, p. V009T13A012.
[28] N. Jackson, F. Stam, J. O'Brien, L. Kailas, A. Mathewson, and C. O'Murchu, "Crystallinity and mechanical effects from annealing Parylene thin films," *Thin Solid Films,* vol. 603, pp. 371-376, 2016.
[29] W. Zhang, R. Shen, K. Lu, A. Ji, and Z. Cao, "Nanoparticle enhanced evaporation of liquids: A case study of silicone oil and water," *AIP Advances,* vol. 2, no. 4, 2012.
[30] N. Jackson and J. Muthuswamy, "Flexible chip-scale package and interconnect for implantable MEMS movable microelectrodes for the brain," *Journal of microelectromechanical systems,* vol. 18, no. 2, pp. 396-404, 2009.
[31] N. Binh-Khiem, K. Matsumoto, and I. Shimoyama, "Tensile film stress of parylene deposited on liquid," *Langmuir,* vol. 26, no. 24, pp. 18771-18775, 2010.