Electrospinning Thin Films of Stretchable and Self- Healing PDMS

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Abstract—

Recent advancements in space technology have prompted the exploration of extended human missions to celestial bodies like the Moon and Mars, posing new challenges in terms of material durability and reliability in harsh space environments. The study focuses on fabrication of the thin films of self-healing poly dimethyl siloxane (PDMS) employing electrospinning. Electrospinning is a promising technique that allows precise control over the properties of the spun material. The paper presents comprehensive research results, including thickness assessments, self-healing tests, and stretch tests on samples spun for different durations. Results indicate that thicker samples 201.2 µm tend to exhibit superior self-healing capabilities 35 % in three hours and 60 % healing after 95 hours at room temperature, with a linear relationship between spin duration and thickness. These findings offer valuable insights into the practical applications of self-healing materials in space technology, opening new avenues for the development of robust and reliable spacecraft components, and potential applications in aerospace and healthcare sectors.

TABLE OF CONTENTS

1. INTRODUCTION	I
2. MATERIALS AND METHODS	2
3. RESULTS	
5. REFERENCES.	
6. ACKNOWLEDGEMENTS	
7. BIOGRAPHY	
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1. Introduction

Recent advancements in space technology have opened up new possibilities for extended human exploration and colonization missions on celestial bodies like the Moon and Mars.[1-5] However, these exploration and colonization missions come with challenges, particularly in terms of the durability and reliability of materials and technologies in the harsh space environment [6, 7]. Future spacecraft must be long-lasting, highly reliable, autonomous, adaptable to various conditions, and require minimal maintenance. The experience gained over the last two decades on the International Space Station (ISS) has provided valuable insights into operational constraints and maintenance requirements. Instances of internal pressure loss due to breaches, cracks, or holes on the ISS have highlighted the need for robust maintenance procedures [8-11].

Space hazards such as micrometeoroids and orbital debris (MMOD), temperature fluctuations, radiation, and high vacuum conditions. MMOD can puncture or cut space structures, leading to depressurization and damage. Thermal variations can deform, crack, age, or detach materials [12, 13]. Radiation, including galactic cosmic rays and solar radiation, can damage electronics and materials, while high vacuum conditions cause material outgassing and chemical modification [14]. To address these challenges, self-healing materials have gained attention as potential solutions. These materials can autonomously repair damage caused by impacts or environmental factors, enhancing spacecraft reliability and safety while reducing maintenance costs[8]. Self-healing polymers find applications in the thin film coating for space electrical power systems insulation, integrated into the reinforced composites into thin films to improve specific properties of the composite material, such as strength, stiffness, durability, or other performance characteristics [15]. Reinforicable composites applications in a wide range of industries, including aerospace, automotive, construction, and sports equipment manufacturing, where lightweight, high-strength materials are often required [16-18]. Other applications of self-healing materials are in the space structural components of spacecraft

hulls, external instruments seals gaskets, and layers in the spacecraft structures to enhance their durability and ensuring continuous protection against the space environment. For long-duration space missions, the durability of electronics and sensors is vital[19]. Self-healing polymers can increase the lifespan of these components by repairing physical damages that might otherwise lead to failure[20, 21].

Thin polymer films have applications that can be extensively engineered and expanded for various applications, including filtration, composite manufacturing, energy systems, and microfluidics. Numerous methods exist for fabricating polymer fibers, including electrospinning, wet spinning, biospinning, interfacial complexation, microfluidic spinning, and melt spinning. Among these techniques, electrospinning emerges as the most promising and appealing choice not only for fiber but also for thin films.

Electrospinning is the process of applying a thin layer of a polymer on a surface in a fiber form. This process is performed by applying a high voltage to the nozzle or needle and the spinning metal drum collector. The fundamental operation revolves around electrohydrodynamic principles, where ultra-thin fibers are generated in response to a highvoltage electrical supply. In the electrospinning process, a liquid droplet containing polymer or emulsion is subjected to a potent electric field, instigating the formation of a jet. Subsequently, a sequential process of stretching and elongation occurs, leading to the hyperstretching of the jet and ultimately yielding fibers. This method stands out due to its potential for effective control over attributes, including thickness, composition, and porosity, all within a relatively straightforward protocol. Additionally, electrospinning boasts notable advantages in scaffold fabrication, such as its simplicity, the precision it affords in regulating flow rate and voltage, and its suitability for scaling up the production process. Various materials, including organic polymers, small molecules, colloidal particles, composites, and combinations of organic and inorganic or organic and organic systems, have been successfully transformed into nonwoven mats using modified electrospinning techniques. These advancements have expanded the scope of electrospinning applications, particularly in the realm of flexible and stretchable electronic devices such as flexible and stretchable conductors, transparent electrodes, strain sensors, pressure sensors, energy harvesting and storage devices, transistors, and optoelectronics.

This report provides a comprehensive account of the synthesis of a self-healing polymer known as PDMS (Polydimethylsiloxane) using a one-pot condensation method. Subsequently, the synthesized PDMS was subjected to electrospinning, a technique facilitated by an in-house setup. The objective of this study was to create thin films of PDMS, which were meticulously characterized through tests, including profilometry, self-healing assessments, and stretch tests. These tests were essential in assessing the properties and performance of the electrospun thin films.

2. MATERIALS AND METHODS

Bis (3-aminopropyl) terminated poly (dimethylsiloxane), H₂N-PDMS-NH₂, Mn = ~2,500, Triethylamine, ≥99.5%, anhydrous Chloroform, Methylenebis (phenyl isocyanate), Isophorone diisocyanate, and Methanol were purchased from Sigma Aldrich and used without modification.

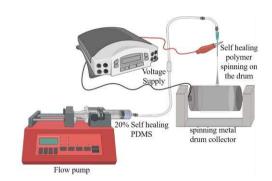


Figure 1: Schematic image of the electrospinning process of the self healing polymer.

Methods:

To a flame-dried round bottom flask containing bis(3aminopropyl) terminated poly (dimethyl siloxane) (15.0 g, M_n~2500 g/mol, 1 equiv.) at 0 °C in 60 mL anhydrous CHCl₃, was added Et₃N (1.5 mL, 1.8 equiv.) slowly. A solution of 4,4'-methylenebis(phenyl isocyanate (0.6 g, 0.4 equiv.) and isophorone diisocyanate (0.8 g, 0.6 equiv.) in 30 mL anhydrous CHCl₃ was added dropwise over 15 minutes to the reaction mixture while maintaining a temperature of 0 °C. The reaction was allowed to slowly warm to room temperature and stir overnight. The reaction was quenched with excess MeOH (2.3 mL) and concentrated by rotary evaporation to a 20% w/w solution of polymer to CHCl₃. To obtain an optimal viscosity for electrospinning, additional MeOH (9 mL) was added, and the solution was allowed to stir for several minutes to ensure homogeneity[15, 22]. Characterization of the self-healing PDMS was conducted by Fourier-transform infrared (FTIR) spectroscopy using the attenuated total reflectance (ATR)-Fourier transform infrared (ATR-FTIR). Analyses were performed with an Agilent Cary 630 FTIR spectrometer, equipped with a Golden Gate Single Reflection Diamond ATR attachment.

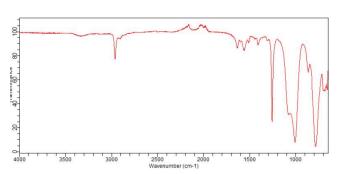


Figure 2: Fourier-transform infrared spectroscopy (FTIR) plot showing the complete reaction of the copolymers in synthesis of self-healing PDMS polymer.

Spectra were averaged over 128 scans at a resolution of 4.0 cm⁻¹

Experiment Set-Up: The electrospinning setup, as illustrated in figure 1, involves several steps. Initially, a 10.0 ml syringe was filled with the SHP-chloroform solution and connected to an 18-gauge needle using appropriate connectors and tubing with dimensions of 1/16" ID \times 1/8" OD. This syringe was then placed within the NE-4000 Two Channel syringe pump, with the pump settings adjusted to a syringe diameter of 14.43 mm and a flow rate of 99.0 µL/min. The needle was positioned in a downward direction, facing the spinning collector. Two alligator clips connect the voltage supply to both the needle and the collector. The surface of the collector was covered with aluminum foil, serving as the substrate for the electrospinning process. Once a consistent flow of solution was observed, typically after about five drips, the DC high voltage supply was activated and set to 18.0 kV. The formation of a Taylor cone was monitored, and simultaneously, the spinning collector is initiated at 250 rpm, with a timer set for the desired spinning duration. The solution was spun for various durations, such as 15.0 minutes, 30.0 minutes, 60.0 minutes, 2.0 hours, and 3.0 hours. Upon completion of the designated time period, the instrument was shut down carefully to prevent dripping of the solution over thin films.

3. RESULTS

Fourier-transform infrared spectroscopy (FTIR):

The disappearance of the MPU and IU peaks containing the N=C=O end-groups around 2300cm⁻¹ confirms that the material synthesis has been completed, and all isocyanate groups have been consumed and reside within the new polymer chain as shown in figure 2.

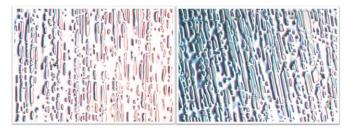


Figure 3: Optical microscope images depict electro-spun polymers subjected to varying spin durations: 30 minutes on the left and 60 minutes on the right.

Microscopic Analysis:

The microscopic images were taken using the Axioscioe 7, Carl Zeiss, of the electro spun self-healing polymer as shown in figure 3. In the 30-minute spin sample, fibers exhibit a tendency to cluster, leading to reduced fiber count and increased gaps. Conversely, the 60-minute spin sample displays a denser fiber distribution.

Thickness Assessment:

To assess thickness of the spun samples, thickness tests were conducted on specimens spun for different durations: 15 minutes, 30 minutes, 1 hour, 2 hours, and 3 hours. Silica wafers were chosen as the substrate for these samples due to

their ability to provide adequate contrast between the upper and lower surfaces of the spun polymer, facilitating accurate thickness measurements. The thickness evaluation was carried out using a profilometer, a specialized instrument capable of scanning the lowest and highest points of a

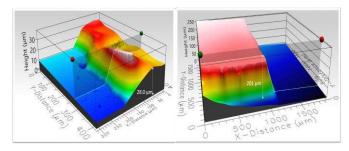


Figure 4: Profilometer scan results for 15-minute electro-spin (left) and 3-hour electro-spin (right). 15-minute spin was found to have a thickness of about 28.0 µm. 3-hour spin was found to have a thickness of about 201 µm.

surface, thereby providing valuable insights into its topography and thickness distribution. This test aids in understanding how the thickness of the spun polymer varies with the duration of the electrospinning process. The sample was measured using Filmetrics Profilm 3D, a 3D optical profilometer and the results are illustrated in figure 4. The profilometer works on the principle of White Light Interferometry (WLI) (FILMETRICS, A KLA Company, 2023). In WLI, a white light source illuminates the test object and the reference. The reflection from the reference and the test object forms a constructive or destructive interference pattern on the Charge Coupled Device (CCD) image sensor that is then spatially sampled by individual CCD pixels[23]. The Profilm 3D calculates 47 standard ASME/EUR/ISO roughness parameters and is capable of measuring surface feature in the range of 50 nm - 10mm (FILMETRICS, A KLA Company, 2023). The thickness measurements for the different samples are as follows: 15 minutes resulted in a

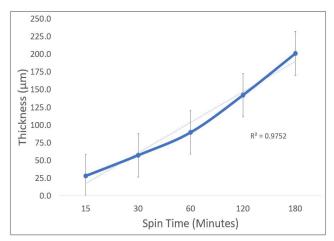


Figure 5: The figure illustrates the variation in thickness with respect to spin time. The Profilometer scan results indicate a nearly linear correlation between each spin duration and the resulting thickness.

thickness of 28.2 μ m, 30 minutes yielded 57.3 μ m, 1 hour exhibited 89.6 μ m, 2 hours recorded 142.3 μ m, and 3 hours showed 201.2 μ m. It is noteworthy that the relationship

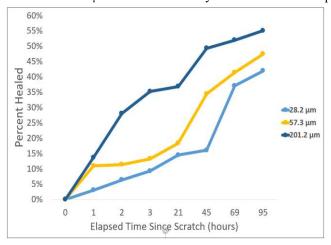


Figure 6: Graph of the self-heal tests compared to its thickness.

between spin time and thickness is nearly linear, with a high R^2 value of 0.9752 as shown in figure 5. This data enables the calculation of the coating rate, which under the given conditions, was approximately $1.50~\mu m/\text{min}.$ With this information, one can determine the required spinning time to achieve a specific thickness. For instance, if a thickness of $15.0~\mu m$ is desired, the polymer can be electrospun for 10~minutes using the provided conditions. The other two tests compare the thicknesses of the samples rather than the spin times.

Self-healing assessments:

These were carried out on spun samples with varying spin durations, including 15 minutes, 30 minutes, 1 hour, 2 hours, and 3 hours. These samples were electrospun onto PET plastic substrates. The procedure involved deliberately scratching the surface with a fine needle of gauge 2. Following the scratching, images were captured at hourly intervals for the initial three hours, and subsequently, once

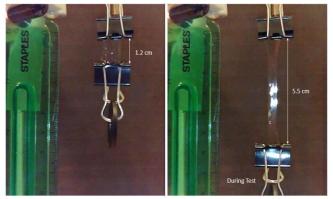


Figure 7: Images of the beginning (left) and during (right) of the stretch tests. Samples were cut to 1.2 cm × 1.1 cm and stretched by hanging a binder clip and 5 g weight and then with 10 g.

daily for the following four days, all using an optical microscope. These tests were conducted at room temperature. To analyze the results, ImageJ, an image processing software, was employed. It was utilized to determine both the area of the scratch and the percentage reduction in the scratch area. The calculations were performed using the following formula

$$\frac{initial\ area-current\ area}{initial\ area}\times 100\%$$

The figure 6 shows thicker samples 201.2 µm were able to heal at room temperatures to 50 % with 24 hours and 60 % within 95 hours. The thinner samples 28.2 μm were able to heal at room temperature to 15% in 24 hours and 50% within 95 hours. The self-healing capabilities are attributed to the intermolecular interactions between the urea functional groups within the self-healing polymer. The strongest of these interactions is proposed to involve MPU units, which are capable of forming quadruple hydrogen bonds in a cooperative manner with opposing MPU units. In contrast, IU units can only establish a maximum of dual hydrogen bonds with another IU unit, hindered by the isophorone moieties. Consequently, the multivalent effect leads to significantly stronger MPU-MPU interactions compared to IU-IU interactions. This enhanced MPU-MPU crosslinking plays a crucial role in maintaining the elastomer's structural integrity. Insert reference: ACS Appl. Polym. Mater. 2020, 2, 9, 4127-4139

Stretch Tests:

The investigation on stretch tests were conducted for two weight conditions, specifically 7.7 grams (including the clip

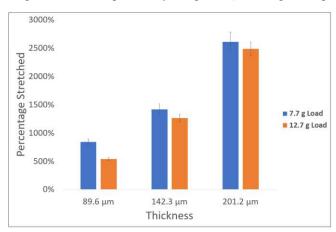


Figure 8: Bar graph of the stretch tests for 89.6 μm, 142.3 μm, and 201.2 μm with 7.7 gram and 12.7 g

and 5 grams of additional weight) and 12.7 grams (comprising the clip and 10 grams of additional weight) as shown in figure 7. All samples were cut to 1.2 cm × 1.1 cm. As illustrated in figure 8, it becomes evident that thicker samples tend to exhibit better performance compared to their thinner counterparts. This phenomenon is likely attributed to the presence of a greater number of molecular polymer chains in thicker samples. These additional chains contribute to a more robust network, enhancing the material's self-healing capabilities. However, it is worth noting that there were slight declines in performance when a larger load was applied, as

seen in the 12.7 grams condition. This decline may be attributed to the reduced amount of time available for the molecular polymer chains to retract and reform their bonds before encountering additional stress-induced breakages.

4. CONCLUSIONS

In summary, electrospun self-healing polymer thin films show great promise for enhancing the reliability and durability of materials used in aerospace and beyond. These materials offer innovative solutions to challenges posed by the harsh space environment and can find applications in various industries, ultimately contributing to advancements in technology and engineering. Further research and development in this field hold the potential to unlock even more remarkable properties and applications for self-healing materials, paving the way for safer and more efficient space exploration and technological advancements.

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6. ACKNOWLEDGEMENTS

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