NDnano
Center for Nano Science and Technology

NDnano Summer Undergraduate Research 2018 Project Summary

1. Student name & home university: Paula Murphy, Trinity College Dublin

2. ND faculty name & department: Department of Aerospace and Mechanical Engineering

3. Summer project title: Stretchable Polymer Composite Film with Extreme Ductility and Toughness

4. Briefly describe new skills you acquired during your summer research: This project taught me how to think outside the box as a considerable amount of innovation was required to overcome the problems encountered. In addition, I gained teamwork and communication skills due to the fact that I worked on this project with another student. Finally, I gained experience using instruments and equipment that I would never have had the opportunity to work with before, such as the corona treatment and the Discovery HR-2 Hybrid Rheometer.

5. Briefly share a practical application/end use of your research: This aim of this project was to develop an extremely ductile yet tough material, however the combination of these two properties is quite rare. If successful, this project could be used to advance the flexible electronics industry as it could lead to the development of inexpensive reusable devices.

6. 50- to 75-word abstract of your project:

The purpose of this project was to successfully create a highly ductile polymer composite with great tensile strength and toughness, which would be capable of undergoing significant strain without fracture failure. The polymer composite consisted of a matrix of polydimethylsiloxane (PDMS) and reinforcing ribbons of molecularly aligned polyethylene. The results proved successful, whereby the ultimate tensile strength of the final polymer composite was approximately four times that of pure PDMS while maintaining its ductility.

7. References for papers, posters, or presentations of your research:

[1] Li, Xinming , et al. (2016), Large-Area Ultrathin Graphene Films by Single-Step Marangoni Self-Assembly for Highly Sensitive Strain Sensing Application. Adv. Funct. Mater., 26: 1322-1329. doi:10.1002/adfm.201504717Advanced Functional Materials. 26. 1322-1329. 10.1002/adfm.201504717.

[2] Pang, Yunsong, et al. (2018) Exfoliated Graphene Leads to Exceptional Mechanical Properties of Composite Polymer Films. under review, ACS Nano (2018)

[3] Yang, Chenying, Wang, Wei & Li, Zhihong. (2009). Optimization of corona-triggered PDMS-PDMS bonding method. 319 - 322. 10.1109/NEMS.2009.5068586.

The purpose of this project was to develop a ductile polymer composite with great toughness and optimise the manufacturing procedure. A material which exhibits ductility, toughness and high strength simultaneously is uncommon. However, such a material would be desirable as it could undergo significant strain with a low risk of failure. If successful, this project could greatly improve the reusability of flexible, stretchable and wearable electronic devices at a relatively low cost, which as a result could aid with much advancement in the biomedical industry [1].

The polymer composite was to consist of a polydimethylsiloxane (PDMS) matrix and reinforcing ribbons of molecularly-aligned polyethylene (PE). PDMS is a silicone polymer with great ductility, biocompatibility and durability. However, it has extremely low toughness and tensile strength. On the contrary, prior research has shown that aligning the PE molecular structure has the ability to increase its ultimate tensile strength beyond that of steel in the chain direction, while maintaining its bending flexibility. However, the PE produced is not ductile [2]. Since both PDMS and PE are widely available and inexpensive, they were the ideal materials to be used for the polymer composite.

The original concept for the polymer composite was to sandwich ribbons of the PE between two stretched films of PDMS. Upon removing the force from the PDMS causing the distortion, it would return to its original length and the PE ribbons would form wrinkles, as shown in figure 1. This would allow for the PDMS to maintain its ductility up to a certain point and, when stretched beyond this limit, the PDMS would be reinforced by the high-strength PE ribbons. Thus there were three areas of focus for this project: 1) To develop and optimise a means to fabricate the thin PDMS films required; 2) To design a device which could be used to stretch and align the PDMS films when making the polymer composite; 3) To optimise the bond between the two PDMS films and the PE ribbon.

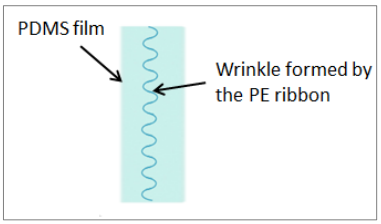

Figure 1: The Polymer Composite

When manufacturing the PDMS films, the aim was to ensure the films were as thin as possible, ideally less than 100µm. However at this scale, difficulties arose from the fact that the PDMS was highly susceptible to failure when loaded if any imperfections are present in the film, such as air bubbles or tiny edge tears. In addition, PDMS was very sticky and tended to curl up on itself, meaning failure was likely when unravelling the film. After much experimentation, it was found that the most effective way to fabricate the PDMS films was as follows:

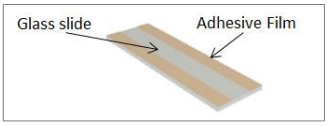

Figure 2: Glass slide lined with adhesive film to create a 100µm crevice

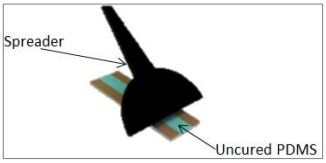

Figure 3: A spreader was use to ensure the surface of the uncured PDMS was even and in line with the adhesive film

1) A glass slide was cleaned with ethanol to remove any residue. Using two layers of a 50µm adhesive film, the two long edges of the glass slide were lined creating a 100µm deep rectangular crevice in the centre of the slide, as seen in figure 2. This crevice had the same dimensions as that of the final PDMS film to be manufactured.
2) The uncured PDMS was prepared with a 10:1 ratio of the polymer base to the curing agent. This was then placed in a vacuum chamber to allow for the PDMS to de-gas.
3) The uncured PDMS was filled into the crevice created on the glass slide. Using a spreader, it was ensured that the surface of the PDMS layer was the correct depth and even, as seen in figure 3.The glass slide then was placed back into the vacuum chamber to remove any air bubbles that may have formed.

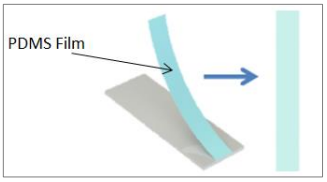

Figure 5: The cured PDMS was carefully removed from the slide to produce a 100µm PDMS film

4) When removed from the vacuum chamber, the glass slide was heated to 85°C for 20 minutes to allow for the PDMS to cure. Once cured, then PDMS film was carefully removed from the glass slide using a tweezers (figure 4). This produced the final 100µm PDMS film required for the composite.

It should be noted that although this was found to be the most effective procedure, there were still many difficulties faced. For example, removing the PDMS film from the glass slide proved to be quite a challenge and it was at this point the PDMS film was most likely to tear if there were any imperfections present. In addition, when the film was removed, the PDMS tended to curl up and adhere to itself. These difficulties were reduced by handling the PDMS with great care and soaking the film in ethanol to temporarily relax it and prevent it from sticking to itself.

Following the production of the PDMS films, it was necessary to design a device which could stretch two of these films by the same amount and allow for the easy insertion of the PE ribbons. In addition, it must allow for the two stretched films to be aligned with each other easily when assembling the polymer composite and hold the PDMS films stretched for the entire curing process. The final stretching device can be seen in figure 5, which was designed in Solidworks and manufactured using a 3D polymer printer.

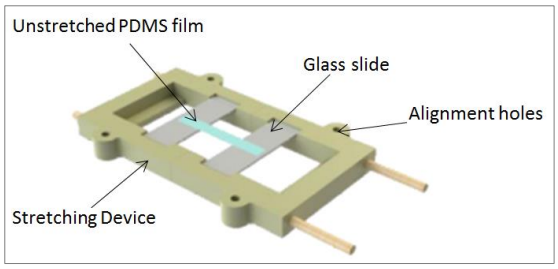

Figure 6: Stretched PDMS film

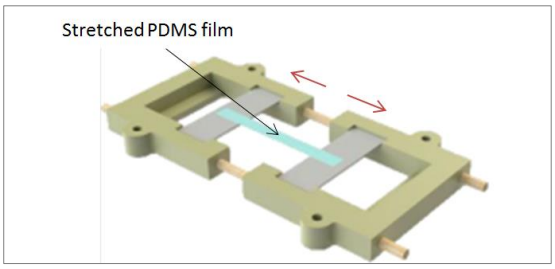

Figure 7: Stretched PDMS film

The stretching device was operated by first setting up the PDMS as shown in figure 7. In this image, a single PDMS film was bonded to two glass slides using the corona treatment [3], which was subsequently set up in the stretching device as shown in figure 5. From here, the PDMS was simply stretched by the desired amount (figure 6). Finally, figure 8 shows how the device was used to align the two stretched PDMS films for the polymer composite. It should be noted that in this image, there are two identical stretching devices, where one is inverted. The two PDMS films were aligned using the alignment pins.

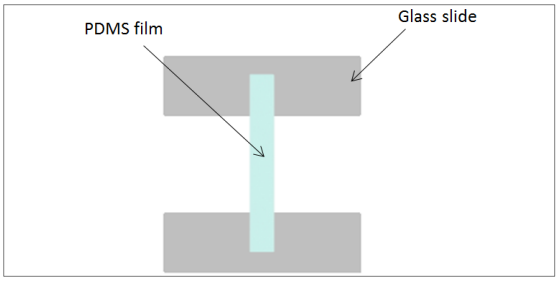

Figure 8: The PDMS film setup for the stretching device

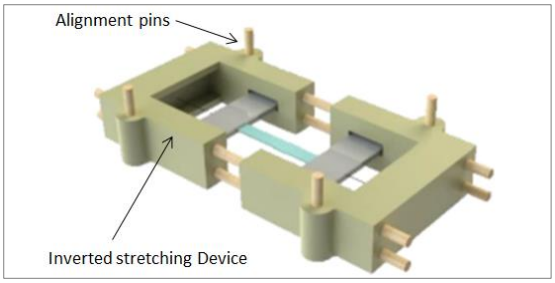

Figure 9: Aligning the two stretched PDMS films

The final stage of the project was to manufacture the polymer composite and optimise the bond between the PDMS films and the PE ribbons. The original manufacturing process involved using the corona treatment separately on the two PDMS films in their stretched state before placing a PE ribbon on one of the films and finally aligning the films as shown in figure 8. The films were subsequently cured at 85^{o}C for 30 minutes in this state. However, there were initially many issues encountered with the bond that formed between the layers when using this process, as well as the bond that formed between the PDMS and the glass slide when setting up the films as shown in figure 7.

After much experimentation, it was found that the following greatly improved the strength of the bond formed using the corona treatment:

- Ensuring the glass slides are cleaned with ethanol prior to bonding.
- Applying a weight helped improve the PDMS to glass bond.
- Using the corona treatment separately on both layers to be bonded when bonding PDMS to PDMS or PDMS to glass.
- Coating the PE ribbon in a very thin layer of uncured PDMS before placing it between the two stretched PDMS films, which have previously been subject to the corona treatment.
- Firmly pressing the stretched PDMS films together with the PE ribbon between them prior to curing to remove air bubbles and to ensure good contact between the layers.

Following the procedure outlined, the polymer composite produced can be seen in figure 9. In the image, the wrinkle formed by the PE ribbon is clearly defined and it is clear to see that there are no air bubbles present between the layers of the polymer composite. When tested, the polymer composite acted as one material, meaning the bond between each of the layers was good. In addition, the final polymer composite appeared to have maintained its bending flexibility.

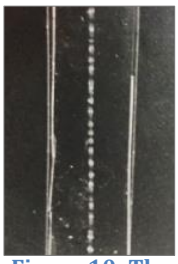

Figure 10: The final polymer composite

Following the visual inspection, the strength of the polymer composite was tested using a Discovery HR-2 Hybrid Rheometer with the ETC Tensile attachment, as shown in figure 10. The rheometer subjected the material being tested to a constant strain rate and the corresponding load experienced by the test material was recorded at certain intervals. The device was used to first determine the stress-strain data associated with a pure PDMS film and then that of the polymer composite containing a single PE ribbon, where both samples had a cross-sectional thickness of 200μm. A graph of the data produced can be seen in figure 11.

Figure 11: Discovery HR-2 Hybrid Rheometer

From figure 11, it is clear to see that the addition of a single PE ribbon to the PDMS film greatly increased the ultimate tensile strength in the direction of the PE ribbon. The strength of the material was approximately quadrupled, where the pure PDMS failed at 1.6MPa and the polymer composite could reach up to 7MPa. From the graph in figure 11, the slightly steeper slope associated with the polymer composite when compared to that of the pure PDMS indicates a slight decrease in ductility, as a greater force was required for the same extension. However, the polymer composite was still extremely ductile and, due to its increased strength, had a much greater strain to failure than the pure PDMS sample.

Overall, the project was a success as a polymer composite was created using the desired materials which had superior strength while maintaining ductility.

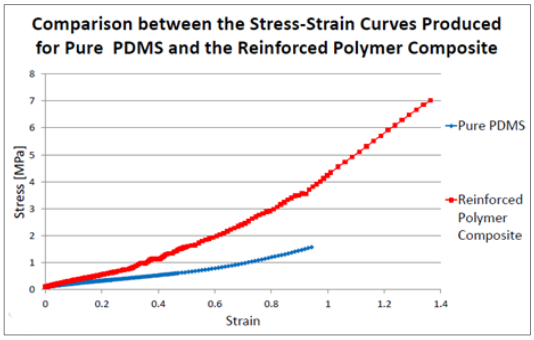

Figure 12: Graph comparing the stress-strain curve produced for a pure PDMS film and that of the polymer composite

Following the successful creation of the polymer composite, where the PDMS film was reinforced in a single direction, it was decided to create a sample where the PDMS film was reinforced in two perpendicular directions. The initial attempt involved manufacturing a biaxial stretching device and following the same procedure as before, however this method proved unsuccessful. It was subsequently decided to manufacture the biaxial sample in two separate stages, where three PDMS film layers were used and the PE ribbons sandwiched between the first and second PDMS film layers were at 90^{o} to the PE ribbons between the second and the third film layers. This method proved to be the most effective and the polymer composite produced can be seen in figure 12.

In conclusion, a manufacturing method was successfully developed and optimised as part of this project to produce a polymer composite with high strength, toughness and ductility. The polymer composite performed as predicted and the PE ribbon formed the intended wrinkle in the PDMS film. Further testing would need to be carried out in order to determine the response of the polymer composite when subject to dynamic loading conditions or to characterise its creep behaviour. In addition, more research could help to further improve the quality and consistency of the polymer composites produced, as well as determining a means to automate the manufacturing process or increase the scale of the samples produced.

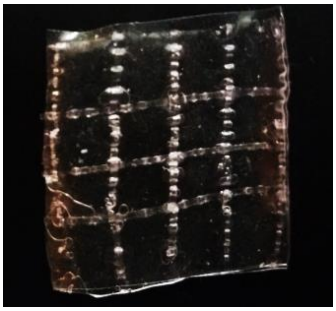

Figure 13: Biaxial polymer composite