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MECHANICAL CHARACTERIZATION OF STRETCHABLE ECOFLEX THIN FILMS FOR MICROENGINEERING APPLICATIONS

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ABSTRACT

Use of elastomers in microelectromechanical systems (MEMS) is increasing especially in wearable technology. Ecoflex is a silicone-based elastomer that has demonstrated significant interest in a variety of MEMS applications including wearables, stretchable electronics, and microfluidics. However, the manufacturing methods and their impact on their mechanical properties have not been systematically investigated. This paper investigates three different types of Ecoflex material (Gel, 00-10, 00-30) and methods of how to manufacture the material at the micro-scale and the impact that the manufacturing methods have on the material's mechanical properties. The results demonstrate that the method of mixing and the curing temperature and procedure used significantly affect the mechanical properties. Spin mixed films had less variation and higher elastic modulus compared to hand mixed due to gas entrapment in the film, especially for higher viscosity Ecoflex. The paper also investigated mixing of multiple variations of Ecoflex materials to alter the mechanical properties which can be used to fine tune mechanical features. Thin films of Ecoflex were fabricated using spin coating techniques and patterned using O_2 plasma. This paper describes manufacturing methods of making thin film Ecoflex samples for MEMS applications.

Keywords: Ecoflex, elastomer, stretchable electronics, microengineering, Elastic Modulus, silicone

1. INTRODUCTION

Stretchable elastomers are growing in demand with applications in soft robotics, wearable technology, electronic skin, and stretchable electronics [1-5]. With the growing demand there is also a keen interest in integrating these materials into microfluidics or other microelectromechanical systems (MEMS) applications [6, 7]. The most commonly used elastomer in MEMS is polydimethylsiloxane (PDMS) which has been extensively investigated in bioMEMS and microfluidic applications [8-10]. However, PDMS has a relatively high elastic modulus (MPa) range and has a low elongation to break <200% [11-14], which is a poor match for tissue. Previous research demonstrated that the manufacturing methods used to fabricate thin-film PDMS had a significant effect on the mechanics of the material [14], but there is limited research on mechanics of other silicone elastomers.

Due to the high elastic modulus of PDMS it is not suitable for e-skin applications or applications that attempt to mimic biological tissue mechanical properties, as most biological tissue has elastic modulus in the kPa range [15, 16]. Recently there has been significant interest in other silicone-based elastomers such as Ecoflex. Ecoflex is a series of silicone-based elastomers with a lower elastic modulus (5-100 kPa range) and increased elongation to break (300-1000%) compared to PDMS which makes it ideal for e-skin and soft actuator technology. Ecoflex has been integrated into various devices including tactile sensors [17, 18], stretchable antenna and phase shifters [19-21], and lab on chip microfluidics [22, 23]. Research has also investigated methods of creating nanocomposites of Ecoflex to enhance functionality such as making it electrically conducting [24-27].

The mechanical properties of Ecoflex depends significantly on the type of Ecoflex which are typically named based on their shore hardness (00-XX) [28]. The most common Ecoflex are 00-

30 and 00-50 but some of the Gel versions have shore hardness in the 000 range. However, most papers do not describe in detail the methods by which they manufacture the material including mixing methods and curing methods. Most researchers simply follow the manufacturers' recommended methods which include mixing in a 1:1 ratio and curing at room temperature for period of hours (exact time depends on type of Ecoflex). It has been reported that applied temperature after curing can significantly affect the mechanical properties [29, 30]. There is also limited research on manufacturing Ecoflex for MEMS applications and their impact on the mechanical properties [31]. PDMS results have indicated that mixing and curing temperatures have a significant impact on the mechanical properties [14], and therefore it is believed that Ecoflex will have a similar impact, and thus it is necessary to investigate the effects caused from manufacturing.

This paper investigates various manufacturing techniques (mixing and curing temperature) on multiple types of Ecoflex materials to determine the impact they have on mechanical properties. In addition, we investigate methods of creating a thin film of Ecoflex and methods of patterning the material which can then be integrated into a MEMS device. Mixing Ecoflex with other elastomers can affect their mechanical properties as well, and to demonstrate fine tuning of the Ecoflex mechanics we mixed various types of Ecoflex at various ratios.

2. MATERIALS AND METHODS

2.1 Sample Preparation and Design of Experiments

Platinum-catalyzed silicone elastomers were used in this experiment selected from the Ecoflex series from (Smooth-On, USA). The three different Ecoflex elastomers that were investigated include (00-10, 00-30, and Gel), where the 00-10 and 00-30 represent the shore hardness. The Gel material had a hardness of 000-35 according to the manufacturer's specification sheet. All of the materials consisted of two parts (A and B) that were mixed at a 1:1 ratio by weight (50/50 wt%). The viscosity and density of the 1:1 mixed solutions are given in Table 1.

Table 1: Mixed Ecoflex Properties

Ecoflex Type	Viscosity (cps)	Density (g cm^{-3})
00-30	3,000	1.07
00-10	14,000	1.04
Gel	9,300	0.98

The generic method of making the films consisted of: i) measuring parts A and B , ii) mixing parts A and B (various methods investigated below), iii) pouring the mixture into a mold that was sprayed with release agent (Mann Ease Release 200) and using doctor blade techniques to create smooth uniform films [32], iv) curing the material (various methods investigated below). The overall procedure is shown in Figure 1.

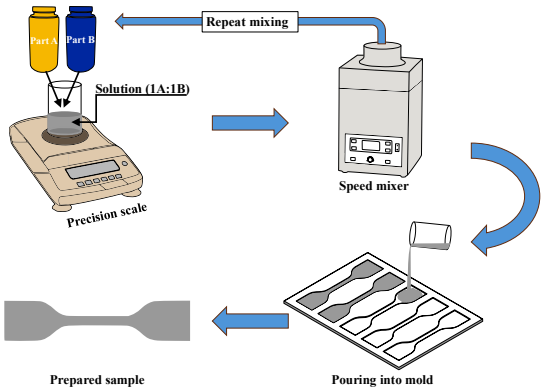

FIGURE 1: SCHEMATIC OF PREPARING ECOFLEX SAMPLES FOR MECHANICAL TESTING [33]

Molds were designed and fabricated using a 3D printer (Ultimaker S5). The molds used were designed using ASTM D412 C as shown in Figure 2. To measure micro-scale versions micro-sized molds using S1 dimensions were used. The molds allowed us to replicate structures while providing smooth sides to the material instead of using a puncher to cut the structures into the mold shape.

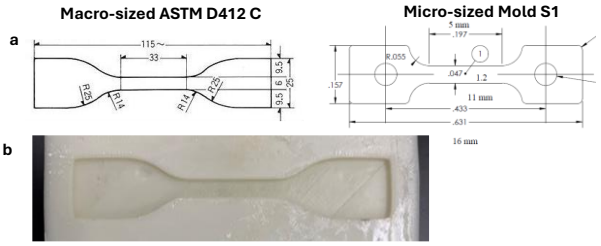

FIGURE 2: (a) DRAWING OF MACRO AND MICRO SIZED MOLDS WITH DIMENSIONS AND (B) A 3D PRINTED MOLD USED TO CREATE DOGBONE SHAPED TESTING STRUCTURE.

The first manufacturing step necessary to make the material is to mix Parts (A and B). Parts A and B were measured on a scale and mixed (50/50 wt%). The samples were mixed using various techniques including hand mixed with a stirring stick ("hand mixed"). Hand mixed samples were mixed for 5 minutes. A speed mixer from Flacktek (DAC 150.1 FVZ-K) was used to mix the solutions together. The speed mixed parameters were set at 3000 rpm for 1 minute. The speed mixer is typically used to mix solutions without significant heat and was designed to reduce air bubbles in the mixture to create a homogenous mixture. The third method investigated included hand mixing followed by a 10-20 minute degassing procedure which was performed in an vacuum desiccator until the bubbles were no longer visible [28].

After mixing the solutions were poured slowly into the molds. During elevated temperature curing methods, the 00-10 and 00-30 solutions remained at room temperature for 10 minutes prior to being heated, this was performed to allow any bubbles from the pouring process to escape.

Curing procedure is very important to MEMS applications. The manufacturer provides recommendations for curing including a room temperature cure followed by a heat cure. For most MEMS applications faster curing of the material is beneficial as it saves time. PDMS is often cured at elevated temperatures but research has shown that the curing procedure affects the mechanical properties [14]. A pre-test was performed to determine the curing time at various temperatures. The results of the initial test were then used in the design of experiments shown in Table 2, to determine effects of curing temperature on the mechanical properties of the films. Five different curing methods were investigated including: Room temperature (RT) at the recommended manufacturers time, 80 °C at the time shown in Table 2, 100 °C, and 125°C, a 5th method included the extended cure method which consisted of a room temperature cure followed by several heat cured methods. Overall, the extended cure for 00-30 took 7 hours whereas the room temperature took 4 hours and the 125°C took 15 minutes. The Gel films had a lower pot life so curing at 125°C was not performed as the 100°C cure only required 15 minutes.

Table 2: DESIGN OF EXPERIMENTS FOR INVESTIGATING EFFECTS OF CURING TEMPERATURE

Ecoflex	00-30	00-10	GEL
Mixing Types	Speed Mix Hand Mix	Speed Mix Hand Mix	Speed Mix Hand Mix
Cure #1	Cure RT 4h	Cure RT 4h	Cure RT 2h
Cure #2	Cure 80°C 2h	Cure 80°C 2h	Cure 80°C 30m
Cure #3	Cure 100°C 1h	Cure 100°C 1h	Cure 100°C 15m
Cure #4	Cure 125°C 15m	Cure 125°C 15m	n/a
Cure #5 (Extended Cure)	Cure RT 4h 80°C 2h 100°C 1h	Cure RT 4h 80°C 2h 100°C 1h	n/a

2.2 Mechanical Testing

The samples were tested using a stress-strain testing procedure which was previously described [32]. The testing was performed using a TA.XTplus100C Texture Analyzer (Stable Micro Systems, USA). The samples were clamped and subject to a uniaxial tensile test to evaluate the Elastic modulus which was taken as the linear slope of the stress-strain curve up to 100% strain. The strain rate was 3 mm/s.

2.3 Microfabrication of Ecoflex

Spin coating was used to create thin films of Ecoflex after mixing. Samples were spun at varying final speeds from 1000-6000 rpm. Due to the high viscosity of the solutions the initial spreading speed was at 500 rpm with an acceleration of 100 rpm/s. The acceleration to the final speed was 300 rpm/s and the final spin speed was 60s in duration. A Si wafer was used as the substrate which was cleaned using O_2 plasma. The liquid Ecoflex solution was mixed using the speed mixer and then poured onto the wafer. The material was then room temperature cured as well as elevated temperatures.

To pattern the Ecoflex, a metal (Ti) layer was deposited and patterned using a liftoff technique. The Ti layer acted as a hard mask during the etching process. The Ecoflex samples were etched in a plasma asher using O_2 plasma (Tergeo-Plus, Pie Scientific, USA) at 50 mTorr and 150 W. The etch rate was determined by etching for 1 hour and then measuring the thickness variation.

3. RESULTS AND DISCUSSION

A typical stress-strain curve for the 00-30 and 00-10 Ecoflex material cured at room temperature and mixed using speed mixer is illustrated in Figure 3. The results indicate that 00-10 has a higher ultimate tensile strength compared to 00-30 (697 kPa compared to 572 kPa. The elastic modulus was calculated by measuring the slope of the linear section from 0-100% strain.

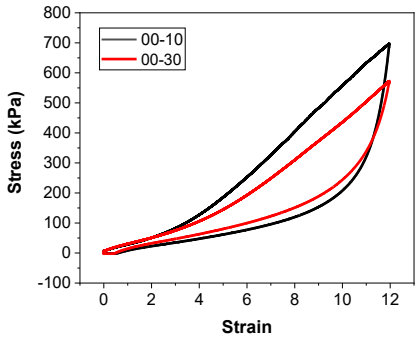

FIGURE 3: STRESS STRAIN CURVE FOR 00-30 AND 00-10 ECOFLEX.

To determine the effects of mixing, samples were mixed using three methods and cured at both 100°C and room temperature. Figure 4 illustrates the visual effects on the samples when mixed using the speed mixer or by hand. The Speed mixer 00-30 sample had no visual bubbles in the films after curing at 100°C, whereas the 00-10 samples had a couple bubbles. On the other hand, the hand mixed samples for both 00-30 and 00-10 had an abundance of bubbles. The 00-10 sample had more bubbles or gas entrapments than 00-30, which is believed to be due to the higher viscosity of 00-10 which slows down the ability of the gas to be released from the sample. If the top layer of the Ecoflex is cured before the gas can escape it will become entrapped in the film. This entrapment is believed to cause significant variations in mechanical properties as they act as voids. The impact of bubbles or voids has been previously illustrated in polymers and porous materials [34]. Since the curing process at 100°C occurred within 1 hour, the gas did not have enough time to diffuse out of the film before the top layer was cured. The speed mixer mixing method produced significantly less gas entrapments compared to hand mixing. Even when cured at room temperature the hand mixed samples had a significant number of bubbles within the film. The random distribution of bubbles can affect the mechanical properties as they are leaving defects or gas entrapment within the films. All elevated temperature tests were performed in a convection oven, a hot plate might help cure the material from the bottom up which could help to alleviate gas entrapment.

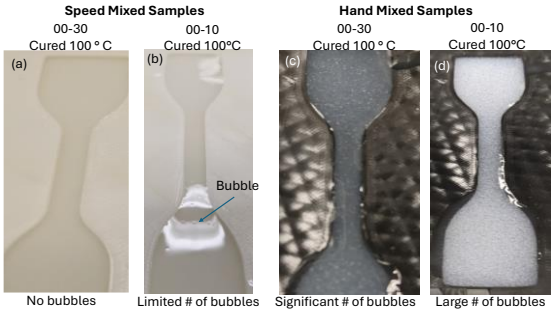

FIGURE 4: CURED ECOFLEX AT 100°C WITH DIFFERENT MIXING METHODS

Mechanical characterization of the films as a function of mixing methods is shown in Figure 5. The elastic modulus of 00-10 was higher than 00-30 which was higher than the Gel. However, in all three materials the elastic modulus was significantly reduced, and the variation (standard deviation) was increased for hand mixed material compared to speed mixed. The mechanical results agree with the results from Figure 4, the entrapment of gas caused a significant decrease in the elastic modulus as it creates voids in the film which can be easily stretched. Also, the variation increases as distribution of the bubbles will alter the mechanical properties and having random locations of the bubbles will lead to higher variations in elastic modulus. However, samples that were hand mixed and degassed had similar elastic modulus compared to the speed mixer. The speed mixer method saves manufacturing time as it only required 1 minute of mixing versus hand mixed/degas which took 15-25 minutes. The hand mixed method is therefore time consuming and can have a significant factor in lower pot life elastomers. Therefore, the speed mixing method was used throughout the rest of the paper.

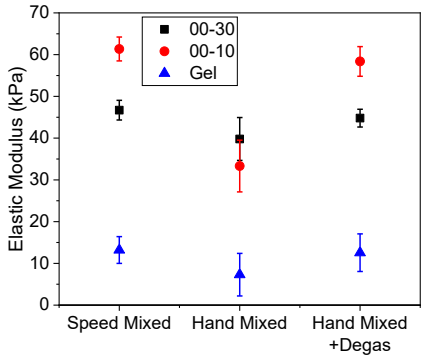

FIGURE 5: ELASTIC MODULUS AS A FUNCTION OF MIXING METHODS AND CURED AT ROOM TEMPERATURE.

The results of the elastic modulus as a function of curing temperature and procedure are demonstrated in Figure 6 for the three different Ecoflex materials. The results for the 00-30 Ecoflex illustrate a slight decrease in elastic modulus for 80°C and 100°C, but a significant decrease from 46.7 kPa (RT) to 38.2 kPa for (125°C) and the standard deviation was increased this was believed to be due to the fast-curing time which does not allow enough time for gas to escape. Similar results were found for other elastomers such as PDMS [14]. The extended cure which was cured at room temperature and then followed by elevated curing did not show a decrease but instead illustrated a slight increase to 49.1 kPa. The 00-10 material illustrated a significant decrease in elastic modulus even at the 80°C cure temperature which was due to the higher viscosity of 00-10 which requires more time for gas to escape. The 00-10 cured at RT had an average elastic modulus of 61.25 kPa but when cured at 125°C the average elastic modulus decreased to 30.9 kPa, and the standard deviations increased curing temperature. The Gel samples had a significantly lower elastic modulus of 14.4 kPa at RT and demonstrated a similar reduction of elastic modulus with increasing cure temperature. The results indicate that the 00-30 can be cured at temperatures of up to 100°C without significantly changing the elastic modulus but the other Ecoflex materials were significantly affected. However, in some applications even a 50% reduction in elastic modulus might not be significant but the time saving aspect might be beneficial. The curing procedures could also be used as methods to fine tune the elastic modulus as some applications might want lower elastic modulus.

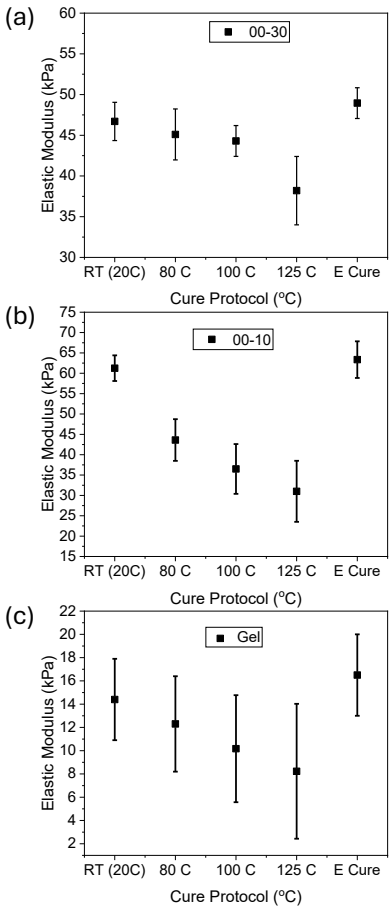

FIGURE 6: ELASTIC MODULUS AS A FUNCTION OF CURING TEMPERATURE PROCEDURE FOR (A) 00-30, (B) 00-10, AND (C) GEL.

MEMS researchers are often interested in fine tuning the material properties as the mechanical properties can affect things such as dynamics, frequency, acoustic velocity etc. To investigate methods of altering the elastic modulus we investigated creating compositions of three Ecoflex materials (ie. 00-10 mixed with Gel, 00-30 mixed with Gel, and 00-10 mixed with 00-30) and cured at room temperature. The results of the elastic modulus for these mixtures from 0,25, 50, 75, and 100% are illustrated in Figure 7. Mixing 00-30 and 00-10 resulted in a decrease in elastic modulus as the 00-30 percent was increased which had a slope of -0.143 kPa/00-30%. Mixing the Gel with any of the Ecoflex significantly altered the elastic modulus where the 00-10 to Gel had a slope of -0.43 kPa/Gel%. The slopes can be used to allow researchers to tune the elastic modulus to fit their specific application.

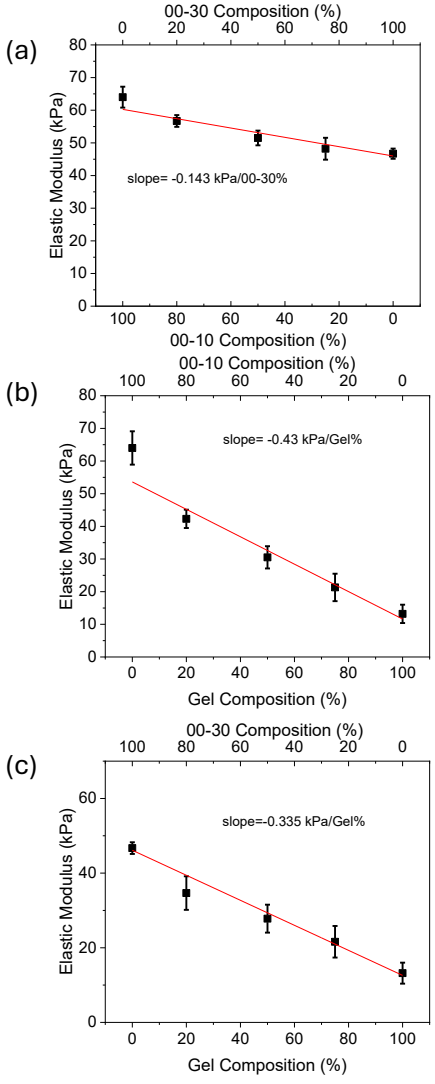

FIGURE 7: ELASTIC MODULUS AS A FUNCTION OF COMPOSITION FOR (A) 00-30 AND 00-10, (B) 00-10 AND GEL, AND (C) 00-30 AND GEL.

After mixing Ecoflex it can be spin coated onto a substrate to be used in MEMS applications. Figure 8 illustrates the thickness as a function of the final spin speed. The 00-10 results in a thicker layer which is expected given the higher viscosity, where a 1000 rpm resulted in a thickness of 152 µm whereas spinning at 5000 rpm resulted in 18.6 µm. The 00-30 films were slightly lower with a thickness of 79.7 and 6.58 µm at 1000 and 5000 rpm respectively. Thin films were able to be cured at elevated temperatures with more ease. This is because the films were thinner so gas bubbles diffused through the film faster than with thick films.

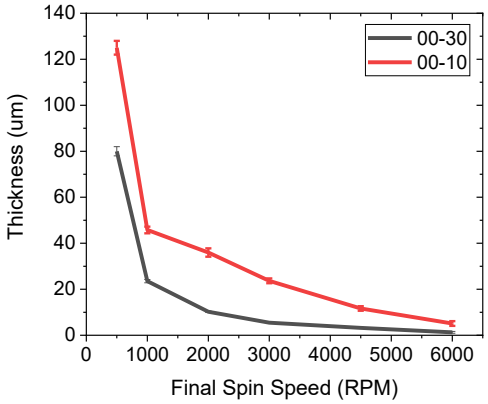

FIGURE 8: SPIN SPEED CURVES FOR 00-30 AND 00-10 AS FUNCTION OF THICKNESS AND FINAL SPIN SPEED.

Being able to pattern the material is critical to be integrated into a microfabrication process. Since Ecoflex is a silicone-based elastomer the etching process was expected to be similar to PDMS. The Ecoflex was etched using a plasma asher with O_2 plasma. The etch rates were 8.20 µm/hr and 6.78 µm/hr for the 00-10 and 00-30 films. The slower etch rate for 00-30 is likely due to the higher density of 1.07 g cm^{-3} compared to 1.04 g cm^{-3} for 00-10. The etching process was slow but probably could be increased by using reactive ion etching (RIE). However, we were able to successfully pattern a structure as shown in Figure 9. This demonstrates the potential capability of integrating Ecoflex films into a MEMS device.

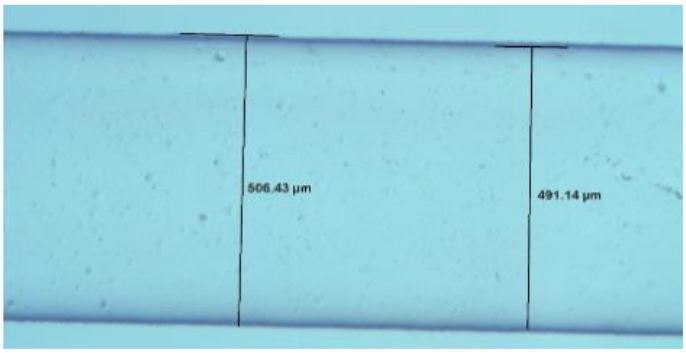

FIGURE 9: PATTERNED RECTANGULAR TRACE OF ECOFLEX ON A SILICON SUBSTRATE.

4. CONCLUSION

We investigated the curing temperature and mixing effects on the mechanical properties of three different types of Ecoflex. In conclusion, the method used to manufacture Ecoflex can have a significant impact on the mechanical properties of the film. The method of mixing the two parts was critical as well as the curing procedure. The gas entrapment (bubbles) appears to have a significant role and thus must be avoided to create repeatable mechanical properties. By using the speed mixer instead of the degassing method, it results in reduced manufacturing time with similar mechanical results. In addition, low viscosity Ecoflex materials can be cured at elevated temperatures without significant change in elastic modulus. However, increasing the cure temperature to significantly reduce the curing time to less than an hour can have a significant impact.

In addition, we demonstrated that Ecoflex materials can be combined to alter the elastic modulus to match specifications needed for the application. This combined with adding nanoparticles to the Ecoflex can be used to alter the mechanical properties, as well as add functionality to the material [27]. Lastly, we demonstrated that Ecoflex can be deposited in micro-thin films and etched using standard microfabrication techniques. Combining these manufacturing methods will allow future researchers to integrate Ecoflex materials to create unique polymer MEMS devices and could potentially replace PDMS in numerous applications as the elastic modulus of Ecoflex is orders of magnitude lower than PDMS.

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