All-polymer based polymorph skin with controllable surface texture

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Abstract
Smart skins are integrating an increasing number of functionalities to improve the interactions between the equipped systems (robots or artificial systems) and their ambient environment. Here, we introduce a controllable texture as a new functionality, based on an innovative soft technology that leverages the strong electro-mechanical coupling of our all-polymer design, which can be easily embedded to a wide range of systems. The device comprises a polymer-based heating element [doped PEDOT:PSS (poly-(3,4 ethylenedioxythiophene): poly (styrene sulfonic acid)], a polymer-based soft actuator (Ecoflex 00–50/ethanol) and a polymer-based casing [PDMS (polydimethylsiloxane)]. We introduce a smart pipe prototype module and use our controllable polymorph skin to tailor the interaction between the pipe and the fluid. This allows us to obtain a 50% reduction of the friction coefficient in turbulent regime, between non-actuated and actuated configurations. This concept may find applications in engineering fields such as smart skin-based touch control and controllable friction coefficients.

Supplementary material for this article is available online

Keywords: flexible electronics, controllable texture, conducting polymer, drag, e-skin

(Some figures may appear in colour only in the online journal)

1. Introduction
Soft and flexible artificial skins play a major role as a functional interface between a system and its environment. Sensing is a major function and is nowadays integrated in smart skins for a wide range of measurements (pressure [1–4], temperature [5–7] and vibrations [8–10]). This is a growing field, and extensive research covering many aspects of multifunctional soft robotic skins is being carried. Recent innovations have been reported in the literature [11–15]. Controlling the macroscopic texture of soft skins is a new function that has been scarcely investigated. One recent study reported on the use of film patterning with nanoparticles, applied mostly to optical applications [16]. Yet, to our knowledge, controlling skin morphology, on a large scale, has not yet been investigated.

Such functionality could play a very important role in system/environment interactions, for several reasons. For example, texture primarily affects touch and contact behaviors. Contact behavior is most often quantified through macroscopic properties such as the Coulomb friction coefficient [17, 18]. Following the well-known Coulomb’s law, the slippage force between a robotic gripper and an object can be controlled either by changing the normal contact force or by controlling the friction coefficient, which largely depends on texture. Increasing the normal contact force is not desirable for fragile objects. In such cases, controlling the texture of artificial skins may be a way to safely improve the gripping efficiency. Texture is also an important factor in the interaction between fluids and surfaces. The texture of boundary walls can largely modify the drag coefficient [19–22].
Therefore, in this instance, modifying the texture is a way to control the interactions between the flow and its surrounding structure, and to create opportunities to design smart piping or hydro- and smart aerodynamic structures.

To the best of our knowledge, adaptive skins, for which the texture can change when subjected to an external electrical stimulus, have not been studied intensively. Some research studies have focused on the change in texture resulting from the mechanical loading of heterogeneous materials [23]. Other studies have used a difference in pressure [22], pneumatic actuation [24], origami-inspired structures [25] or laser ablation [26] to modify the texture.

We are proposing an original, all polymer-based, structure in which we take advantage of the strong electro-thermomechanical coupling of the constitutive behavior of the selected materials to create a responsive soft skin. To do so, we use a conductive polymer [PEDOT:PSS, i.e. (poly-(3,4 ethylenedioxythiophene): poly (styrene sulfonic acid))] to create a flexible heating network [27–29]. The generated heat induces the dilation of a material specifically designed to feature an extremely high coefficient of thermal expansion. We use an ethanol-doped Ecoflex 00–50 (demonstrated by Miriyev) [30] that combines the stretchability of a silicone-based elastomer with the liquid–vapor phase transition of ethanol to reach a high volumetric expansion. This material system enables much higher volumetric expansion compared to more classical conductive polymers that have been often used for soft actuators [31, 32]. This expanding material is then embedded in a much stiffer soft skin that restricts its in-plane expansion, resulting in the modification of the surface texture. A somewhat similar concept was introduced by Alt- muller [33], who used a gas pocket instead of a doped Ecoflex, and integrated it as an isolated actuator. Our technique makes it possible to use such a design for controlling the texture of large electronic skins.

We analyze the key parameters of our system in two steps. First, we analyze the volumetric expansion and examine how the texture gets modified, with varying time and temperature. We then use x-ray micro-computed tomography (XμCT) to verify the changes occurring in the internal structure, before and under actuation. Finally, we demonstrate the real actuation applications to show the capabilities of the developed smart structured skin for friction applications in smart piping technologies.

2. Experimental section

2.1. Sample preparation

The flexible electrodes and heaters were prepared as follows: an aqueous dispersion of PEDOT:PSS Clevios PH1000 (HC Starck) was mixed with 3% mass ratio of Ethylene glycol (EG) (Sigma-Aldrich) and magnetically stirred at 500 RPM, for 6 h. The solution was later on drop-casted on a petri dish base coated with a Teflon sheet and subsequently cured inside a fume hood, at room temperature, for 48 h, in order to obtain a 50 μm-thick film. To precisely cut the solution-casted PEDOT:PSS film, a CO₂ laser cutter PLS 6.75 (universal Laser Systems) was used, with the following optimized parameters: power 3%, speed 5%, pulse per inch 1000, and focus distance of Z-axis of 4 mm.

The soft actuator was prepared using an Ecoflex 00–50 (Smooth-On) silicone rubber and some ethanol at 96% (VWR Chemicals). In order to obtain a soft actuator, the preparation was optimized by mixing a 20% mass ratio of ethanol with Ecoflex. The silicone elastomer of Ecoflex (part A) and ethanol were first mixed for 2 min, the silicone crosslinker of Ecoflex (part B) was then added and mixed for another 2 min, at the recommended 1:1 mass ratio of part A and part B. The mixture was subsequently poured into a 3D-printed acrylonitrile butadiene styrene (ABS) mold and cured for 3 h, at room temperature.

To assemble the device, the Ecoflex based soft actuator was placed on a customized 3D-printed ABS mold. It was then covered with the PEDOT:PSS film to build the heating element. Contact electrodes were assembled on the PEDOT: PSS film, using copper wires fixed with colloidal silver liquid (Electron Microscopy Sciences). For the casing, PDMS was prepared using a silicon-based elastomer and curing agent Sylgard 184 (Dow Corning), with a 10:1 mass ratio. The mixture was degassed under vacuum for 30 min to remove air bubbles. Finally, PDMS was poured into the mold and cured at room temperature, for 48 h, to package all the components.

2.2. Temperature map

The evolution of temperature as a function of time was measured using a thermal camera (FLIR SC7000); the data were processed using the Altair software. The heating process was monitored for 4 min, with a supplied power of 5 V and 0.6 A. It was then turned off, and the data were collected during the cooling step.

To compare the temperature in function of time for ethanol and methanol, we used a Pico TC-08, as well as a thermocouple type K, which we placed over the soft actuator. We supplied the system with a power of 5 V and 0.6 A during 5 min.

2.3. Non-destructive x-ray micro-computed tomography

We imaged the sample, in situ, applying 3 W (to heat up the sample) using a Nikon XT H 225 (Nikon Metrology, Leuven, Belgium) XμCT device connected with a Paxscan 2520DX x-ray amorphous-Si flat panel detector (Varian Imaging Systems). Each sample had a nominal size of (5 × 2 × 40) mm (width, thickness, and length, respectively). The XμCT device was set to operate at a voltage of 60 kV and a current of 100 μA. The scanning of the sample was performed at a voxel size resolution of 16 μm, and with the specimen stage rotating through 360° in stepped increments of 0.115°. A total of 3141 projection images were obtained by averaging 8 frames for each rotation step, at an exposure of one second per frame. Reconstruction of the projected images was performed using the CT Pro 3D version 4.4.2 software (Nikon Metrology), resulting in 1524 slice images with a resolution...
of 1910 \times 1910 \text{pixels each. A volume rendering software (Avizo 9.2.0, FEI Company) was used to analyze the changes in the micro-structure of ethanol-filled bubbles.}

2.4. Device actuation

We actuated the device by applying a voltage of 5 V (0.6 A) for 4 min, it was then turned off. The actuation of the device was measured using a drop shape analyzer (Kruss), where an 

\textit{in situ} video was captured (as supplementary content), and image post-processing was carried out using ImageJ (an open source computer software).

To verify the lifetime and reliability of our device, we performed a cyclic test. Each cycle was composed of two periods: a heating period of 7 min in which the power was on, and a cooling period of 5 min with the power off. The voltage supplied during the heating period was 7 V, and a microscope (Leica S6D MC 190 HD) was used to capture images at the end of the 7 min period, to obtain the maximal actuation; image post-processing was once again carried out using ImageJ.

2.5. Smart pipe for friction coefficient reduction

We measured the output water flow in a smart pipe. The interior was equipped with six of our actuator devices systematically distributed along the flow in the streamwise direction. A 3D-printed polyactic acid (PLA) pipe was used as our main structure. The modules of the device were prepared using the protocol described in section 2.1, but with an adjustment of the dimensions, considering the volume of each actuator (5 \times 2 \times 100 \text{mm}^3). To constrain the ‘in-plane’ and ‘out-plane’ expansion (with a direction normal to the external surface of the pipe), a PDMS casing outside the pipe with a thickness of 3 mm was fabricated. The setup is composed of a reservoir fixed under a tap, with a drain that keeps the level of liquid constant to have a constant water column. The reservoir is connected, via a hose, to a valve, itself connected to the entrance pipe, in order to guarantee a fully developed flow at the entrance of the tested device.

In this experiment, the flow rate was measured under two different configurations: a first configuration in which the device was not actuated (as-prepared), and a second configuration in which the device was actuated with an applied voltage of 7 V, and held for 20 min when the valve was opened, to allow the flow of water. A chronometer was used to measure the flow, and a glass beaker was used to collect and quantify the total volume of water that flowed in 20 s. Additionally, in order to verify the friction coefficient modification as a function of the different Reynolds number, the height (vertical difference between the top of the reservoir and the pipe inlet) was modified in six several values from 0.14 to 0.64 m (steps of 0.1 m). From the obtained flow measurement, the Reynolds number was calculated, and the Colebrook equation [34] was used to calculate the friction factor. More details related to the entrance length, Reynolds number and Colebrook equation are provided in the supplementary information. A total of two pipes were fabricated, and for each pipe, measurements were made three times for the ‘as-prepared’ configuration, as well as for the actuated configuration.

3. Results and discussion

The device is fabricated using a thin film of ethylene glycol (EG) doped PEDOT:PSS as a heating element, Ecoflex 00–50/ethanol as a thermally-triggered actuator, and PDMS as a packaging case. Schematics of the soft actuator are shown in figure 1. An individual module (figures 1(a) and (b)) can be assembled, in an array, to create smart skins.

This device operates as follows: when the PEDOT:PSS element is subjected to the electrical current, the increase in temperature produced by the Joule effect heats up the Ecoflex 00–50/ethanol part. As the Ecoflex 00–50/ethanol part contains cavities partially filled with liquid ethanol, therefore, when the temperature reaches the ethanol boiling point the phase changes, resulting in a large dilatation of the Ecoflex. As this soft actuator is constrained laterally by a relatively stiff casing, an out-of-plane motion is generated, creating the apparent texture.

3.1. Electro-thermal response

The temperature field induced when applying a voltage (heating) or ceasing the voltage (cooling) in our actuator device is shown in figure 1(c). During the actuation, the center of the module (above the heating element) reaches the peak temperature (approximately 93 °C) within 2 min. The temperature field displays strong gradients during the first 2 min and then stabilizes. On the other hand, during the cooling process, the actuator reaches 50 °C after 2 min of cooling (figures 1(d) and (e)), and the surface of the device returns to its initial shape. The electro-thermal actuation behavior of the typical ethanol-based single module device is shown in figure 1(f) as-prepared module (0 s, 0 V); (g) as the actuated module (240 s, 5 V), and (h) as the module back to its original state when the voltage is turned off (120 s, 0 V). The current design is the result of an optimization process. For example, to reduce the surface temperature and the necessary power to have the actuation, we aimed to replace the ethanol with a different organic solvent that also had the chemical compatibility with the Ecoflex polymeric matrix. The potential alternative needed to have a lower boiling point, and/or lower latent heat, in order to reduce the necessary energy to obtain actuation. After multiple experimental trials of compatibility with different solvents (e.g. methanol, acetone, dichloromethane, hexane), methanol showed a positive and compatible behavior. It was expected that methanol would have a faster actuation, due to its lower boiling point in comparison to ethanol. The response curves for ethanol and methanol using a thermocouple are also shown in figure S1 is available online at stacks.iop.org/SMS/28/075011/mmedia (supplementary information). The methanol-based soft actuator also exhibits a better uniformity of bubble distribution in the Ecoflex, during the preparation, compared to that
of ethanol. Potentially, ethanol can be replaced by methanol without significant changes in the performance of the actuator. However, the latent heat of methanol is higher than that of ethanol. Consequently, all our studies were performed with the ethanol-based soft actuator.

3.2. Morphological studies with XμCT

The microstructure of the prepared individual module and its changes with the electro-thermal stimulus can be visualized in figure 2, which shows (a) the as-prepared module (control), (b) the actuated module, and (c) the module back to its original state when the voltage is turned off. Corresponding bubble mode appearances, through the XμCT scan, are also shown in figures 2(d)–(f). As expected, the increase in temperature above the vaporization temperature of the solvent resulted in an expansion of the trapped bubbles. Some of them seemed to merge during the expansion process, due to the development of cracks between them. Consequently, the total volume of the actuated module, i.e. the soft actuator component (Ecoflex + ethanol), increased by 62%, when compared to the ‘as-prepared’ module. A large ethanol cavity was observed at the interface between the PEDOT: PSS heater and the soft actuator. This cavity is due to the merging of bubbles next to the PEDOT: PSS layer and experiences large change in volume during the actuation (its volume increases threefold when the voltage is applied). This cavity provides the largest contribution to the total actuation. To better understand the change in morphology of the bubble cloud, in figure 2, a histogram was plotted for the bubble volume versus number of bubbles for three different stages: (g) when the voltage is switched-off, (h) during the applied voltage, and (i) before any applied voltage, respectively. The size distributions are very similar, before and after actuations, which demonstrates the good reversibility of the system. XμCT measurements on the as-prepared module showed an initial average bubble volume of 0.052 mm³, with a total porosity of 13.5%. In the actuated module, the bubble volume increased by approximately 20%, from its initial volume. These results show that the contribution of the small bubble to

Figure 1. Schematic of the soft actuator (a) individual module, (b) exploded view of the individual module. (c) Temperature profile (heating as well as cooling) of the typical individual module (5 V, 0.6 A), (d) image of individual module, and (e) change in temperature of the module surface at different locations, with respect to time. Electro-thermal actuation of the typical ethanol-based individual module, (f) before, (g) during the applied voltage, and (h) after the voltage is switched off. The sample measured 15 × 3 × 50 mm³ (width, thickness, and length, respectively).
the total expansion is of second order, compared with the main cavity.

During the segmentation, some bubbles appeared to be connected, however, in reality they should not be connected, as the size of the interface separating the bubbles were similar or smaller to the voxel size resolution (16 μm), hindering our ability to properly segment the bubbles because of the partial volume effect. By performing a separation of bubbles using a post-processing segmentation algorithm, we identified an overestimation of connected bubbles depicted in figure 2, in which the number of bubbles increased up to 44% after the artificial separation of bubbles (more details in the Supplementary Information). Nevertheless, the total volume of bubbles (porosity) remained virtually the same regardless the separation of bubbles.

3.3. Device actuation

When electrically stimulated, the device deformed in a pseudo-sinusoidal shape of maximum amplitude (∼1 mm) and then returned to its original position. The side view of the initial and final shapes of our device, when using ethanol as an organic solvent and an applied voltage of 5 V, is displayed in figures 1(f)–(h). The device showed a similar behavior when methanol was used as the organic solvent (image not shown). All actuator devices were completely reversible with a fast response, which is a critical aspect for any potential

Figure 2. XμCT imaging of the individual module, during ON (7 V) and OFF (0 V) periods of voltage (mentioned in the figure), after segmentation for (a)–(c) the bulk, and (d)–(f) the bubbles. A histogram of the volume of the bubbles versus number of bubbles is shown for the three different stages: (g) when the voltage is switched off, (h) during the applied voltage, and (i) before any applied voltage. The volume of interest measured 4.5 × 1.8 × 20 mm³ (width, thickness, and length, respectively).

a) b) c)
application. A controllable and reversible texture, therefore, was achieved.

Figure S2 shows the performance of our device actuator as a function of the number of cycles (Supplementary Information), with the maximum actuation measured in the first cycle as the reference point. As verified in figure S2, the device keeps 80% of the actuation, up to 50 cycles. We observed a reduction of the maximum actuation by 50%, after 80 cycles. Over the cycles, we observed a reduction of power consumption. While the voltage was kept fixed, the measured current through the sample was reduced from 0.43 A (1st cycle) to 0.28 A (80th cycle). The cyclic performance of the system was mainly governed by the integrity of the soft heater made of PEDOT:PSS, which broke after 96 cycles. With our design, we found the polymorph skin to be operational for approximately 50 cycles before its efficiency was reduced progressively to zero and breaking. A possible way to make the system more durable would be to add stretchability to the PEDOT:PSS electrode (by patterning, for example).

3.4. Application to smart pipe for friction reduction

One of the potential applications of our technology is in the field of smart pipes, more specifically for friction reduction.

In this case, a polymorph skin is introduced to control the boundary layer between the turbulent flow and the carrying pipe. A schematic displaying the components of the device and the assembly process for that purpose is shown in figure 3(a).

Riblets are well known to reduce the friction when positioned in the streamwise direction [34, 35]. These geometrical features modify the boundary layer by promoting vortices [34]. This effect can be observed only for the turbulent flow (Reynolds number, \( Re > 2400 \)). Indeed, the boundary layer in pipes is first formed of viscous sublayer in which vortices cannot be created. The thickness of this sub-layer is inversely proportional to the velocity [35]. Therefore, it is only when the velocity and the \( Re \) are high enough that the viscous sublayer becomes thin enough for the riblets to interact with the turbulent flow and start promoting vortices [35]. This is well summarized by the classical Moody chart, in the case of geometrical perturbations coming only from roughness modifications [36].

Figure 3(b) represents the schematic of the setup used to measure the performance of the device for friction reduction. In figure 3(c), we can see that the actuated pipe has a strongly reduced friction factor, compared to the non-actuated configuration. The decrease observed for \( \frac{f}{D} \) is between 45 to 65%, depending on the Reynolds number over the chosen experimental range. The higher standard deviation is originated mainly from three factors: hysteresis of our system, for a low Reynolds number we could be in a ‘transitional state’ where the vortex mechanism is randomly present, and also experimental error during the measurements.

4. Conclusion

We have fabricated an all-polymer-based electro-thermo-mechanical actuator device, using an ethylene glycol-doped PEDOT: PSS thin film (as a heating element), Ecoflex 00–50/ethanol (as a soft actuator) and PDMS (as packaging). The device successfully actuated and returned to its original position, achieving the main objective of our study. To validate our polymorph skin, a smart pipe was built. We observed that the device effectively reduced the friction factor by approximately 50%. We believe that our device provides a good foundation for the further development of devices for a wide range of applications, and for which morphing skins with controllable texture can be used for friction or flow control. As a key point of the developed device, when it is compared to the existent technologies it has the advantage of being a simplified control system (electrical), which make the device portable and easy to be embedded into any other system. To the best of our knowledge, this is the first time a complete concept of active and fully electrically controllable polymorph skin is proposed. The proposed technology can be merged with 3D printing approaches to design fully printable polymer-based active skins with optimized microstructures that will have wide applications in robotics and tailored structures/environments interactions.

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Author contributions

GL conceptualized and directed the study. NB and DS carried out the experiment. VL carried out the XCT measurements. All authors contributed to the writing of the manuscript.

Competing financial interests

We declare no competing financial or non-financial interest.

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Figure 3. (a) Components of our device and assembling steps: 1. Insertion of soft actuators in PLA pipe; 2. Placing the PEDOT: PSS heater on the soft actuators and making the electrical contacts; 3. Casing with PDMS and drying for 48 h. (b) Schematic of the setup to measure the performance of the device for friction reduction. (c) Averaged Ratio of Friction coefficient by Hydraulic diameter as a function of Reynolds number.

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