Impact of electrode preparation on the bending of asymmetric planar electro-active polymer microstructures

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ABSTRACT

Compliant electrodes of microstructures have been a research topic for many years because of the increasing interest in consumer electronics, robotics, and medical applications. This interest includes electrically activated polymers (EAP), mainly applied in robotics, lens systems, haptics and foreseen in a variety of medical devices. Here, the electrodes consist of metals such as gold, graphite, conductive polymers or certain composites. The common metal electrodes have been magnetron sputtered, thermally evaporated or prepared using ion implantation. In order to compare the functionality of planar metal electrodes in EAP microstructures, we have investigated the mechanical properties of magnetron sputtered and thermally evaporated electrodes taking advantage of cantilever bending of the asymmetric, rectangular microstructures. We demonstrate that the deflection of the sputtered electrodes is up to 39 % larger than that of thermally evaporated nanometer-thin film on a single silicone film. This difference has even more impact on nanometer-thin, multi-stack, low-voltage EAP actuators. The stiffening effect of many metallic electrode layers is expected to be one of the greatest drawbacks in the multi-stack approaches, which will be even more pronounced if the elastomer layer thickness will be in the sub-micrometer range. Additionally, an improvement in voltage and strain resolution is presented, which is as low as 2 V or 5×10^{-5} above 10 V applied.

Keywords: Compliant electrodes, asymmetric planar electro active polymers, Young's modulus, thermal evaporation, magnetron sputtering, cantilever bending, polyetheretherketone, polydimethylsiloxane.

1. INTRODUCTION

Metal coatings of low weight, low cost and compliant materials gain increasing interests in areas as packaging industry and microelectronics [1-4]. Thus, metal deposition onto polymer materials has been a subject of research. Investigations concerning the bi-layer systems of relatively soft polymer bulk material and rather rigid thin-layered coatings have been published. Here, the diffusion of metals into the bulk [5-7] and barrier materials [7], the wettability [8], as well as the conductivity have been considered [1-4, 9-13]. Further, investigations on the surface reactivity, activation and modification were conducted suggesting the formation of a third interfacial metal/polymer composite layer, when subject to cold ion plasma sputtering [2, 4, 9, 10, 12, 13].

For dielectric elastomer actuators (DEA) the choice of the electrode material including the preparation is of key importance. Especially for multi-stack DEA's compliant electrodes, which do not dominate the stiffness of the entire structure, have to be identified. The use of nanometer-thin metallic films as electrodes is a widespread approach. It is well known that the compliance of metals is usually not given and only allows for strains between 1 and 2 % [14]. There are examples of ultra-thin noble metal films on polymers including polydimethylsiloxane (PDMS), which give rise to strains well above 10 % [14]. Nevertheless, the impact of the Young's modulus from the metal onto the effective modulus of the entire EAP structure cannot be neglected, although the electrodes are much thinner than the polymer layer. The stiffening effect is pronounced in multi-stack actuators [14, 15].

For the optimization of ultra-thin metal layer electrodes in DEA's we hypothesize that the film preparation, for example by thermal evaporation and by magnetron sputtering, has a significant impact on the actuation of DEA-microstructures. Our instrument to evaluate this impact is based on a matured technique used in a broad range of applications [16-22] and has been introduced as a method to evaluate EAP microstructures with high resolution at low voltages [23, 24].

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2. EXPERIMENTAL

2.1 Electrically activated polymer microstructures

Figure 1 displays the procedure for preparing the asymmetric planar structure. The measured asymmetric EAP microstructures were built on polyetheretherketone (PEEK) substrates (APTIV 2000, Victrex, Lancashire, UK) with a thickness of 25 µm. The PEEK films were cut to the shape of 3-inch wafers. These substrates were rinsed in acetone (Merck KGaA, Darmstadt, Germany) to clean the surface. Subsequently, gold (Lesker, East Sussex, United Kingdom) was either thermally deposited or magnetron sputtered on the rough side of the PEEK film, mean surface roughness about 0.8 µm, under vacuum conditions at room temperature. The related sputtering conditions in the Balzers Union SCD 040 system (Balzers, Lichtenstein) corresponded to 0.05 mbar Ar-atmosphere (Carbagas AG, Gümligen, Switzerland) and a constant working current of 30 mA. The thickness was measured using a quartz crystal microbalance (QSG 301, Balzers, Balzers, Lichtenstein). A Baltec MED 020 (Leica Microsystems, Wetzlar, Germany) was used to thermally evaporate the gold at a pressure of 2×10^{-4} mbar and a current of 40 mA. The gold was evaporated from a tungsten boat 32 mm × 7 mm × 0.1 mm (Umicore, Bösingen, Switzerland) at a rate of 0.45 nm/s according to the inbuilt quartz crystal microbalance. In a next step the substrates covered by gold were spin-coated using PDMS (Elastosil 745 A/B, Wacker Chemie AG, Munich, Germany) mixed in a volume ratio 1:1 (component A and B) with rotation speeds to obtain layer thicknesses ranging from 2 to 5 µm. Before crosslinking the PDMS films at a temperature of 120 °C for a duration of 60 minutes, the coated substrates were partly submerged into ethyl acetate (Fisher Scientific, Reinach, Switzerland) to dissolve and wash off the PDMS to become access to the lower electrode (cp. scheme in Figure 1). After PDMS-crosslinking, a mask to obtain two electrodes with a step-like profile as displayed in Figure 1 covered some parts of the structure. The uncovered PDMS surfaces were then either sputter or thermally coated with a gold film of the same thickness to form the second electrode.

2.2 Cantilever preparation

The 3-inch substrates were cut into rectangular cantilevers with an active area of 3 mm × 15 mm and the step-like profile at one end to attach the contacts. The cantilevers were fixed using photo-curable resin (Sensationail, Fing'rs, Dübendorf, Switzerland) cured with an adequate UV-light source providing radiation with a wavelength of 400 to 505 nm. Mounted on a PTFE support, the sample including support was put into an airtight box with feed through for wiring the DEA-structure as presented in Figure 2.

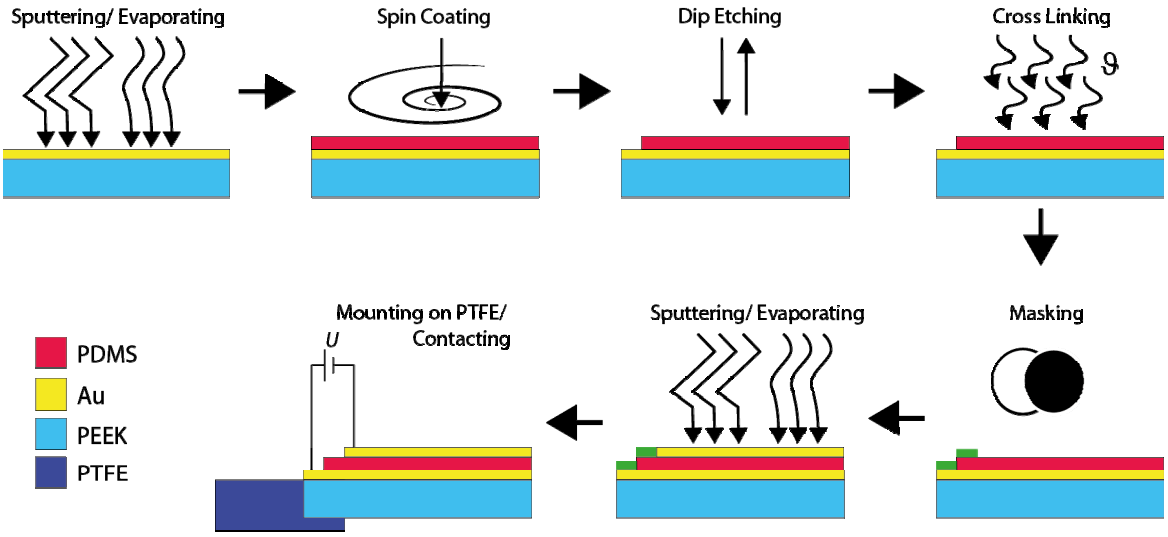

Figure 1. Schematic representation of the manufacturing steps of an asymmetric planar EAP structure. In a first step, the 3-inch PEEK substrates are coated with gold by either magnetron sputtering or thermal evaporation followed by spin-coating of the PDMS. Subsequently the layered structure is submerged into ethyl acetate in order to obtain silicone free access to the first layer of gold. Next the PDMS is thermally polymerized and covered with a second Au layer. The picture on the bottom left shows the asymmetric EAP cantilever structure.

2.3 Cantilever bending measurement

The system to measure the cantilever bending is built on a rotatable frame that holds the laser (He-Ne, 10 mW, Uniphase, Mantca, CA, USA), the cantilever on its holder and the position-sensitive detection unit (PSD, Spotcom, Duma Optronics Ltd, Nesher, Israel). The entire system is placed on an air-damped table (Stada, Vilnius, Lithuania) to reduce the impact of vibrations. To keep the measured samples from moving induced by air convolution the PTFE holder is placed in an air-tight box containing a window transparent to the incident and reflected laser beam as shown in Figure 2. The deflection of the vertically oriented cantilever was measured during the step-wise increase of the applied voltage provided from a Stanford Research System high-voltage power supply (PS310, GMP SA, Lausanne, Switzerland). Vertical arrangement was chosen to avoid cantilever bending by gravity.

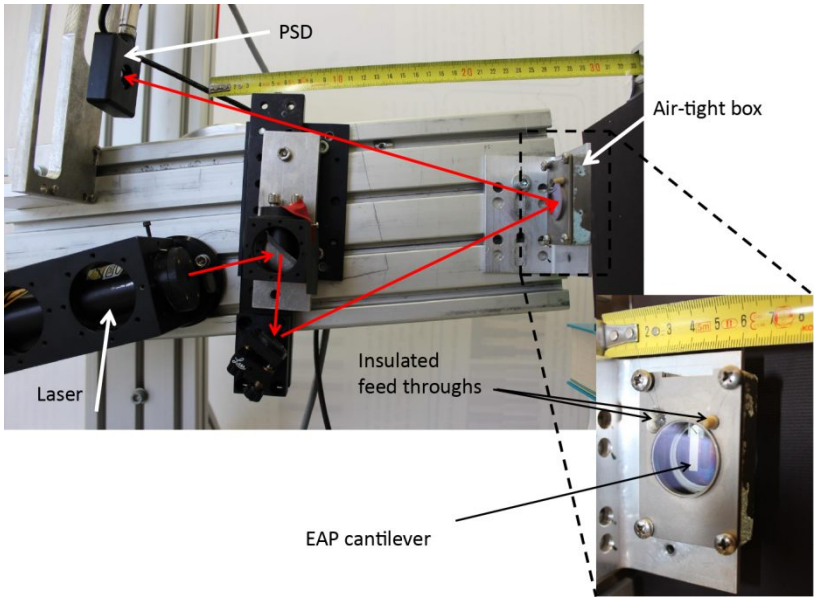

Figure 2. Image of the set-up to measure the bending of asymmetric planar EAP structures. The inset shows the air-tight box containing the EAP cantilever with a window for the incident and reflected laser beams. The electrical contacts are made wiring the gold electrodes.

2.4 Imaging the morphology of the prepared electrodes

The atomic force microscope used was a Bruker Dimension 3100 (Karlsruhe, Germany) operating in tapping mode with a Si cantilever. This cantilever (BudgetSensors, Tap190Al-G) had a spring constant of 190 N/m and a 10 nm tip radius. We applied a scanning speed of 1.19 µm/s.

In addition to the atomic force microscope, a scanning electron microscope (Zeiss, Leo Supra 35, Kloten, Switzerland) provided images of the electrode's morphology.

3. RESULTS

3.1 Performance of DEA's with thermally evaporated and magnetron sputtered Au electrodes

Figure 3 shows the experimental data, meaning the bending of the asymmetric EAP microstructures as the function of the applied voltage. Here, the thermal and sputtered electrodes each with the nominal thicknesses of 25 and 50 nm were selected. The PDMS thickness was set to a value of 5 µm, i.e. it is a factor of 100 or 200 thicker than the electrodes. We normalized the displacement of the laser spot on the PSD d by the cantilever length L. The strain in z direction s_z results from the Maxwell pressure p divided by the Young's modulus E, which can be written as

$$s_z = -\frac{p}{E} = -\frac{1}{E}\varepsilon_r\varepsilon_0\left(\frac{U}{z}\right)^2 \qquad (1)$$

with the applied voltage U, the layer thickness of the elastomer z and the relative dielectric constants of the elastomer and of the vacuum ε_r and ε_0, respectively [25].

Figure 3 demonstrates the impact of electrode preparation on the actuation. We observe that the thermally evaporated electrodes are less compliant. As a consequence the actuators with thermal electrodes exhibit smaller bending than the sputtered ones at the same voltage.

For voltages below 100 V the actuators show a linear behavior for the bending-strain relation, see fitted curves in Figure 3, as expected according to Equation 1.

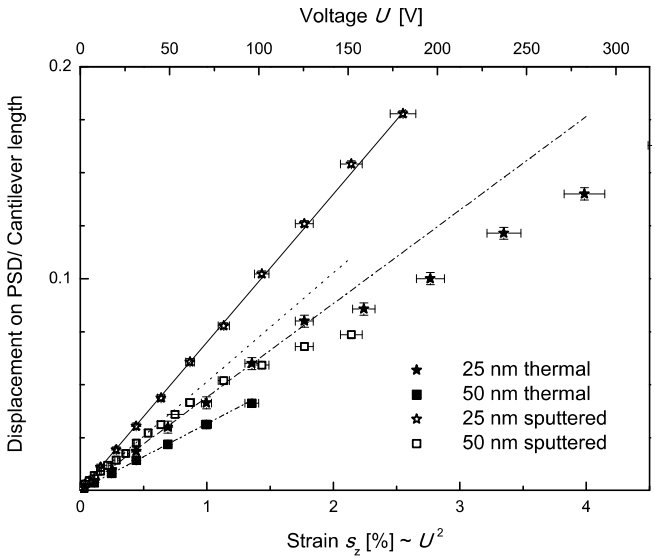

Figure 3. The relation between the applied voltage and the bending of the asymmetric EAP structure demonstrates that the sputtered gold electrodes are better suited than the thermal ones. The solid lines correspond to the fits of the displacement on the PSD as the function of the square of the applied voltages.

3.2 Morphology of the thermally evaporated and magnetron sputtered Au electrodes

Since one can reasonably expect that the defect structures of the electrodes influences the bending behavior of the actuators, the morphology of the thermally evaporated and sputtered gold electrodes was investigated using atomic force microscopy (AFM), see Figure 4, and scanning electron microscopy, data not shown.

The left image of Figure 4 shows the surface of a 15 nm-thin Au layer on a 5 µm-thin PDMS film. Its morphology notably differs from the morphology of the 15 nm-thin sputtered Au film shown in the right image of Figure 4. The AFM image of the thermally prepared Au electrode consists of circularly shaped clusters several 10 nm in diameter, which gives rise to a root mean square roughness of 3.4 nm. The magnetron sputtered Au films are rather flat with a root mean square roughness of 2.2 nm. They show a characteristic defect structure consisting of a few 100 nm-long and 10 to 20 nm-wide cracks. Around each crack one finds an increased height (protrusions), which indicates materials transport from the crack to the surrounding area. Nevertheless, these magnetron sputtered Au films are much more homogeneous than the thermally evaporated ones.

To approve the morphologies of the electrodes prepared by thermal evaporation and magnetron sputtering visualized by AFM, scanning electron microscopy was performed. Both techniques reveal the same morphological features.

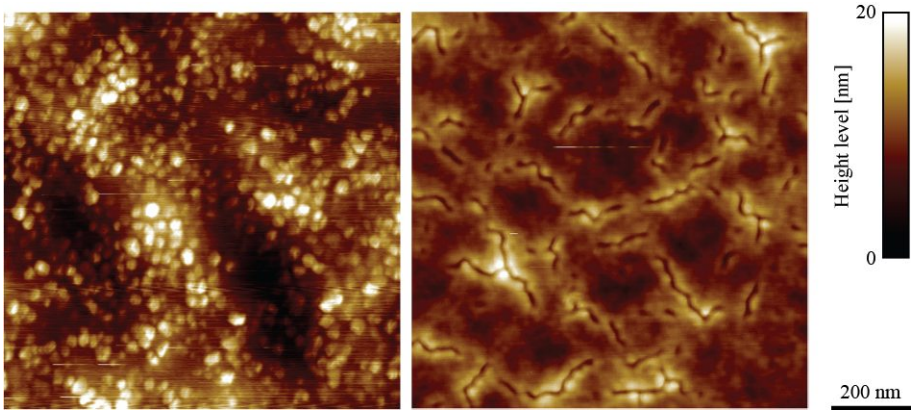

Figure 4. The AFM images of 15 nm-thin thermally evaporated Au films (a) and 15 nm-thin magnetron sputtered Au films (b) deposited on a 5 µm-thick PDMS layer exhibit distinct morphologies on the nanometer scale. Because the thermally evaporated films are coarse with respect to the sputtered ones it is not surprising that the related actuators show minor bending. The root mean square roughness of the thermally evaporated Au film corresponds to 3.4 nm. The related value for the sputtered Au film is 2.2 nm.

3.3 Actuator performance versus film thickness of magnetron sputtered Au electrodes

Figure 5 displays the experimental results of the actuation by the change of the magnetron sputtered Au film thickness from 5 to 70 nm. Again for voltages U below 100 V, one finds the behaviour predicted in Equation (1). For the film thicknesses of 5, 25 and 35 nm the differences are marginal, whereas the thicker films clearly show the expected stiffness increase of the entire DEA structure.

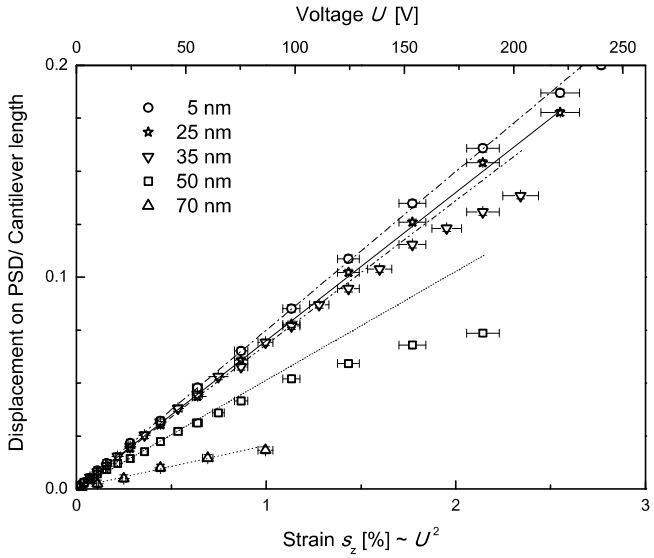

Figure 5. The actuation of an asymmetric DEA structure, which consists of a 25 µm-thick PEEK substrate and a 5 µm-thick PDMS layer with magnetron sputtered Au electrodes with the thicknesses indicated on both sides, shows a behavior as predicted by Equation (1). The diagram also demonstrates that the film thickness of the Au electrodes significantly determines the stiffness of the asymmetric DEA.

In order to evaluate the influence of PDMS-layer thickness on the actuation we prepared further DEA structures. Figure 6 contains data of DEA structures of PDMS layers with thicknesses of 2.1, 2.9, and 5.0 µm sandwiched between 35 and 70 nm-thin Au electrodes, respectively. Figure 6 consists of two parts to present the voltage dependence as well as the strain dependence of actuation. The fits included demonstrate that the data can reasonably be described using Equation 1. The actuators with the 35 nm-thin electrodes exhibit a better performance than the ones with 70 nm-thin Au layers. This phenomenon is more pronounced for the asymmetric structures with 5 µm-thick PDMS films than that with 2.9 µm-thick PDMS layers.

Plotting the normalized bending as the function of the strain s_z, as given in Figure 6(b), one realizes for the measured ranges that the impact of the electrode thickness is dominating the impact of the elastomer thickness. These normalized experimental data further indicate that the thickness ratio between electrode and elastomer is important, which is reflected in the effective Young's modulus of the EAP structure. The data further show the linear dependence up to 2 % strain with slopes depending on PDMS- and Au-layer thicknesses.

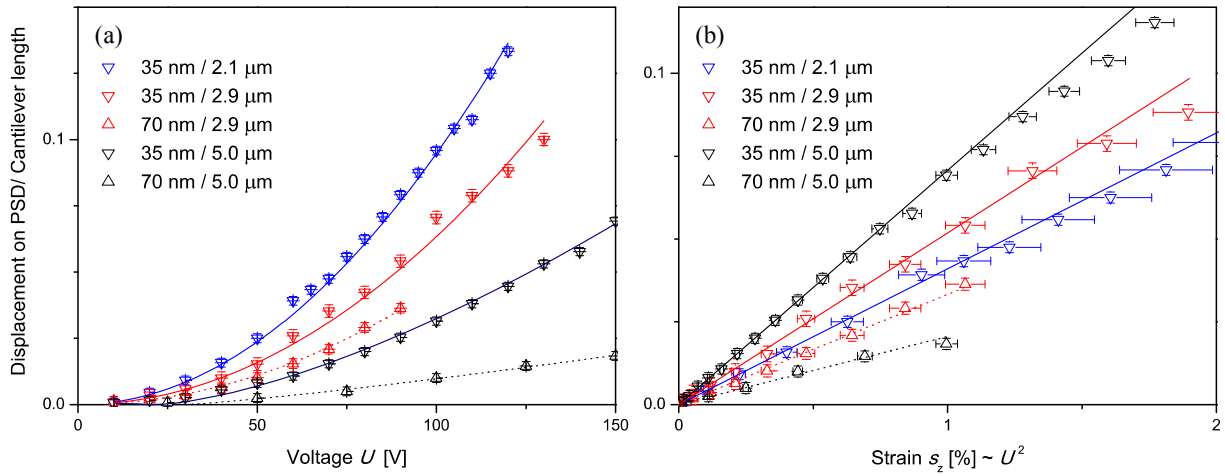

Figure 6. The behavior as predicted by Equation (1) is also found for asymmetric EAP-structures with 2 to 5 µm-thick PDMS layers. The data are represented as the function of applied voltage U in the left diagram (a) and as the function of strain s_z in the right diagram (b).

3.4 Low-voltage behavior and accuracy of the cantilever bending method

In order to demonstrate the improvement of the experimental setup used in previous studies [23, 24], we focus on the low voltage behavior. Figure 7 elucidates first that Equation 1 perfectly predicts the bending behavior as the function of the applied voltage between 10 and 50 V and second that changes in the applied voltages as low as 2 V and the related strain can be reliably detected by means of the current experimental bending measurement. For this purpose, we used again a 25 µm-thick PEEK substrate to build an asymmetric structure. After sputtering a 35 nm-thin Au layer, the 2.1 and 2.9 µm-thick PDMS film, respectively, was spin-coated before the 35 nm-thin counter electrode was sputtered.

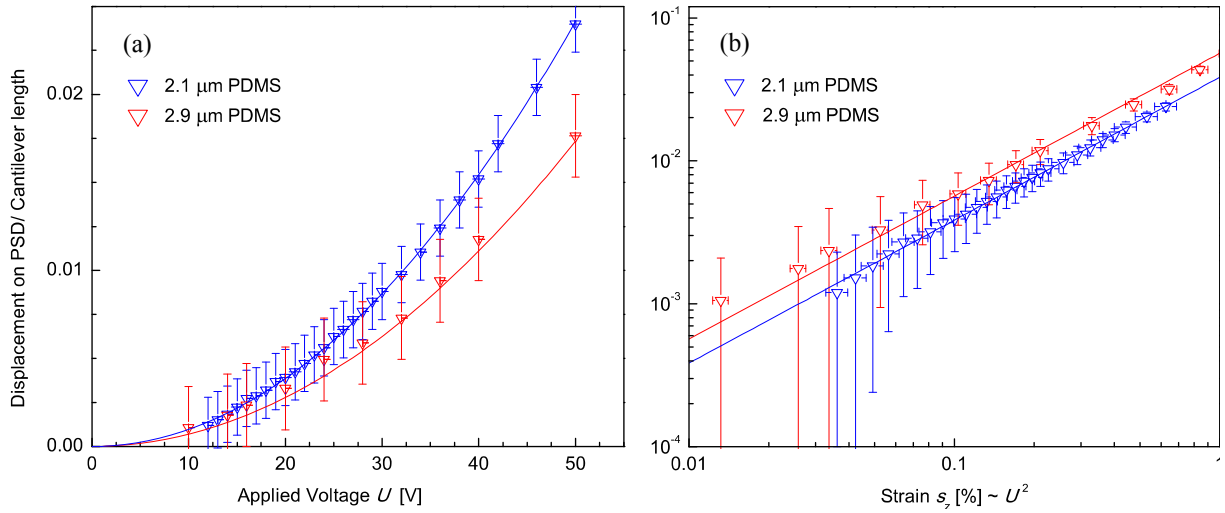

Figure 7. Equation 1 perfectly describes the bending of asymmetric structures consisting of a 25 µm-thick PEEK substrate with 2.1 and 2.5 µm-thick PDMS sandwiched between 35 nm-thin Au films on top, if voltages from 10 to 50 V are applied. In this range, the current experimental setup allow detecting changes of the bending radius through voltage increases as low as 2 V and through strains as low as 10^{-5}.

4. DISCUSSION

The preparation of the electrodes determines the performance of the anisotropic actuator structure. As for both the magnetron sputtering and the thermal evaporation Au has been used, we have to conclude that the defect structure characteristic for the electrode preparation causes the difference represented in Figure 3. The related surface morphology presented in the AFM images of Figure 4 depicts these characteristics. These characteristics have been shown [3]. V. Švorčík *et al.* [3] claimed that the mechanisms of sputtering and evaporation caused the differences in morphology. Apparently the sputtering produces single atoms deposited on to the polymer surface, whereas the thermal evaporation produces atomic clusters leading to the formation of rounded Au islands. This explanation, however, is somehow questionable, as the atomic cluster formation is not clearly demonstrated. In all probability, the three-dimensional islands are the result of the restricted migration length of Au at substrate temperatures of about 20 °C [26]. As the sputtered Au carries higher kinetic energies, they might heat the substrate surface leading to improved surface migration. The cracks are an obvious indication, because one may interpret them as the result of cooling-down process of materials with different thermal expansion.

Nevertheless, one can only speculate about the physical reason behind the performance differences between the thermally evaporated Au and the magnetron sputtered Au electrodes. The experimental data on the bending of the asymmetric structure, however, allow a detailed comparison and thereby provide a method to optimize the electrode preparation.

If asymmetric EAP structures are stretched or bent the characteristic defects in the electrodes with nanometer extensions can significantly reduce the conductivity of the nanometer-thin layers. It seems to be that the defect structure of the thermally evaporated film causes a gradual reduction of charging, whereas for the sputtered Au films the phenomenon is much less pronounced at the same strain. The related dependence of the electrical conductance on the grain and crack size, respectively, was for example described by F. Habrard *et al.* [14].

It should be noted and not surprising that the defect-morphology-related performance difference is more distinctive for thinner Au layers. The deviation of the experimental data from the prediction given by Equation 1 starting at certain strain thresholds is attributed to the defects within the rigid electrodes. Therefore, Au is suboptimal and maybe replaced by softer electrodes or even liquid metals [27]. The thickness of the electrodes by L. R. Finkenauer and C. Majidi is with 60 µm, however, by far too thick for the foreseen application and to be reduced by three to four orders of magnitude. The electrode film, however, has to be thick enough to realize a confluent layer with reasonable conductivity along the entire actuator structure.

Besides the defect structure, the mechanical properties of the entire structure determine the performance of the actuator. There is a significant stiffening of the EAP structure arising from the comprised materials easily derived from the thickness ratios of PDMS and Au layers. Their Young's moduli differ by five orders of magnitude: E_{PDMS} = 200 kPa and E_{Au} = 78 GPa [28]. For example, S. Rosset *et al.* [15] described the stiffening and estimated the mechanical properties for a single polymer layer sandwich structure applying the Voigt model. This behavior is also found in the experimental data of the present study, as displayed in the right diagrams of Figures 6 and 7. As a consequence, the ratio between electrode layer and polymer thickness can be optimized. Considering the data of Figure 6, the actuator structure with 35 nm-thin electrodes and 5 µm-thick PDMS provides the best performance.

The strain resolution shown in Figure 7 can be improved applying an optimized substrate. If the material, here PEEK, is selected and therefore the mechanical properties including the Young's modulus of the substrate given, one can adjust the film thickness. For example, the replacement of a 25 µm-thick PEEK film, used in the present study, by a 6 µm-thick PEEK substrate, which is also commercially available, an increased bending will be observed. This means that it is important to choose the appropriate thicknesses of substrate and electrodes for the pre-selected PDMS layer thickness.

5. CONCLUSION

The present communication reveals that for the bending of an asymmetric dielectric EAP structure the preparation of the electrodes is of prominent importance. The bending of comparable EAP structures, consisting of a 25 µm-thick PEEK substrate and a 5 µm-thick PDMS film sandwiched between 25 nm-thin Au electrodes, with thermally evaporated and magnetron sputtered Au electrodes differ by up to 39 %. For the manufacturing of multi-stack actuators this difference is more than critical, since the stiffening from metal electrodes is regarded as killer criteria. In addition, the preparation of the electrodes determines the optimization of layer thicknesses for the envisioned nanometer-thin multi-layer actuators in

medical implants. The selection of the electrode materials and their preparation procedure affect the conductivity in the multiple operation stages. The conductivity, in turn, dictates the response time, a critical parameter of an artificial sphincter to treat stress incontinence. The quantification both using simulations and experimental approaches, however, is challenging as one observes a nonlinear behavior below thicknesses of 5 to 10 nm [2-4, 13]. The bending bar method [23, 24] is an invaluable experimental tool to optimize EAP micro- and nanostructures.

6. ACKNOWLEDGMENTS

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