Surface and mechanical analysis of metallized poly(dimethylsiloxane) gel for varifocal micromirrors

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Varifocal micromirrors are alternative and miniaturized mirror systems that are used for imaging procedures in biomedical diagnostics, mirror-based tunable lenses, or the correction of spherical aberration. In this study, we demonstrate a new concept for focal length variation by electrostatic mirror deformation of a compact and integrated electroactive polymer actuator based on metallized poly(dimethylsiloxane) (PDMS) gel thin films. The varifocal micromirrors have a lateral size of $70 \times 70 \, \mu\text{m}^2$ and are capable of deforming in either convex or concave direction, depending on a defined surface treatment by O$_2$-plasma or UV/ozone of the PDMS prior to metallization by physical vapor deposition (PVD). Surface and interface analysis by wetting experiments, sputter depth-profiling X-ray photoelectron spectroscopy (XPS) combined with scanning electron microscopy (SEM) on cross-sections processed with focused ion beam (FIB) as well as polymer and metal surfaces are used to understand and to improve the metal film growth and quality with respect to high reflectivity and conductivity. In addition to the high quality of the metallic mirror layer, the mirror displacement is important and inevitably depends on the gel stiffness of the actuator. Therefore, we investigate the gel mechanics and the performance of the actuator with rheology, confocal microscopy, and image formation on the electrically deformed mirrors. Our concept of varifocal micromirrors offers a wide range of applications for tunable mirror-based optics due to their simple and compact design.

KEYWORDS
- electroactive polymer actuator
- PDMS gel
- surface activation
- surface metallization
- varifocal micromirror
- XPS sputter depth-profiling

1 | INTRODUCTION

Varifocal mirrors with micrometer dimensions and adaptive short focal lengths are key elements for miniaturizing and expanding the applications of tunable optical mirror systems. Depending on a convex or concave mirror curvature, varifocal micromirrors are used for imaging processes in biomedical diagnostics, for laser focusing, for mirror-based tunable lenses or for correction of spherical aberration. Conventional electromotive drives of varifocal mirror systems are not able to meet these requirements in terms of space and energy consumption. Miniaturized concepts are based on microelectromechanical systems (MEMS) with electrostatic, piezoelectric, or even electrothermal and optothermal displacement mechanisms but require an air gap and therefore complex manufacturing processes such as resist-based photolithography and etching. An alternative approach to adaptive optics is based on elastomer technology with electroactive polymer (EAP) actuators, which have a compact, comparatively simple structure and have been applied as viscoelastic control layer for spatial...
light modulators. Here, the electrostatic deformation of a metalized poly(dimethylsiloxane) (PDMS) gel layer is limited to a sinusoidal waveform and must be adapted for other miniaturized applications.

In this study, we demonstrate a new concept of varying the focal length of micromirrors due to electrostatic mirror deformation by miniaturized, compact and integrated thin film EAP actuators based on PDMS gel that is capped by narrow metal electrodes. In our previous work, we have already shown the strength of these metallized PDMS gel layers for other miniaturized optical application such as electrically tunable optical microcavities.

To realize a metallized PDMS layer with high reflectivity and high conductivity, the surface activation of hydrophobic PDMS before surface metallization is a state-of-the-art processing. Especially noble metals with high cohesive energy such as gold and silver tend to diffuse deep into the pure polymer and further agglomerate into metal nanoparticles. This process is enhanced by the highly mobile macromolecular chains of the PDMS gel and increased temperatures due to the deposition process. To avoid the undesired diffusion, many different activation processes like O₂-plasma, photochemically produced ozone (UV/ozone), corona discharges or the plasma polymerization of tetraethyl orthosilicate and hexamethyldisiloxane monomers are already known. These methods increase the surface energy and functionality of the PDMS surface and form a hard organoceramic layer as interface between the polymer and the metal layers.

Nevertheless, with the surface and interface analysis of the O₂-plasma and UV/ozone-activated PDMS gel layers, the metal layer formation and the corresponding correlations performed in this thesis, we make a contribution that cannot yet be deduced from the literature. The X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and wetting experiments performed here contribute to improve the growth and quality of the metal layer with respect to high reflectivity and conductivity. Furthermore, the study is completed by rheological and confocal microscopy measurements to investigate the gel mechanisms and electrode deformation of the activated EAP thin film actuators.

2 | EXPERIMENTAL

2.1 | Preparative work

Samples for investigating the surface activation effects are processed by 1:1-mixing of components A and B of PDMS Gel-8100 (NuSil Technology), spin coating of 14 ± 1 μm thin films on previously cleaned (acetone, isopropanol, and water) glass slides (24 × 24 mm²) and subsequent heat curing for 3 hours at 100 °C. The oxidizing surface activation of PDMS thin films by O₂-plasma (PlasmaCleaner Zepto, Diener Electronic GmbH und coKG, 40 kHz) is carried out at 40 Pa with an oxygen flow of 80 L/h and a power of 100 W. The UV/ozone treatment (UVN-16, Hönlé Group, uv-technik Speziallampen, 16 W) is performed under normal atmosphere. The activation time is varied for both methods.

PDMS gel samples are initially manufactured as described above and further processed by Electron-Beam-physical vapor deposition (EB-PVD) metallization of a 2 nm Ti (bottom), 46 nm Ag, and 2 nm Au (top) layer stack to investigate the metal surface and interface. The electron beam evaporator EV M-5 II of Ferrotec GmbH is part of the cluster system MS 150x4-AE-B of FHR Anlagenbau GmbH. The layer thickness is controlled by a quartz microbalance. The deposition rates are 0.5 nm/s for Au and Ag as well as 0.2 nm/s for Ti.

Samples for rheological experiments are prepared by heat curing the 1:1-mixtures of PDMS Gel-8100 as 10 mm thick layer in an aluminum mold.

The varifocal micromirrors are mounted on glass substrates starting with an EB-PVD metallized layer stack of 50 nm Au on 5 nm Ti as lower electrode, having a line width of 70 μm. The patterning is realized by PVD metallization using shadow masks, which are cut with a CO₂-laser (200 W, λ = 10.6 μm) at the laser device microCUTms 10.6 from 3D-Micromac AG.

The material for this shadow mask is a 250 μm thick Al₂O₃-ceramic Rubalit 708S from CeramTec. The PDMS gel layer and the flexible top metallization are processed as described above (2 nm Ti, 46 nm Ag, and 2 nm Au). The top electrode and mirror are also structured up to a line width of 70 μm. In the final structure, the bottom and top electrodes overlap perpendicular and are separated by the PDMS gel. This results in a matrix layout with active areas as pixels forming the EAP capacitor structure.

2.2 | Analysis

The XPS analyses are performed with the spectrometer PHI Model 5700 ESCA system (Physical Electronics, ULVAC-PHI) using a nonmonochromatized Al-Kα (1.486 keV) X-ray source with 250 W. Spectra are recorded with a hemispherical electron energy analyzer at a pass energy of 29.35 eV for high-resolution scans and at a 45° angle to the sample surface. The diameter of the measuring range is about 800 μm. The typical base pressure of the XPS analysis chamber is 3 × 10⁻⁶ Pa (before sputtering). For sputter depth-profiling, Ar is injected with 12 Pa and ionized by an ion gun (4 kV, 0.8 μA, 2 × 2 mm² scan area) to Ar⁺-ions for sputtering. Each sputtering/measuring cycle lasts 3 minutes. The sputtering rate is 1.6 nm/min with Ta₂O₅ as reference material. The spectra are evaluated by MultiPak Version 9.3 and corrected to the C 1s signal at 284.8 eV. For the quantitative elemental analysis, a Shirley background subtraction is performed on the peak areas of the core levels C 1s, O 1s, Si 2p, Au 4f, Ag 3d, and Ti 2p.

The wetting studies are carried out with the device OCA 20 from DataPhysics Instruments GmbH. The contact angles of water, ethylene glycol, and diiodomethane are measured as sessile drops on different surface-activated PDMS gel thin films using the tangent method. The surface energies are calculated with the methods of Wu and Owen, Wendt, Rabel, Kaelble (OWRK). SEM observations of the metal surfaces are conducted with the cold field emission SEM Hitachi S-4700 using the upper or in-lens
secondary electron (SE) detector at an acceleration voltage of 10 kV with a set magnification of 100 k.

The FIB investigations of the metal cross-sections are performed on a Helios NanoLab 660 of Thermo Scientific-FEI. The samples are first coated with strips of carbon and platinum, using electron and ion beam induced deposition and serving as protective layer during ion milling. The FIB preparation is continued by ion bombardment with Ga⁺ at 30 keV at a 90° angle parallel and orthogonal to the metal surface, resulting in a stair-like stripping. The sample preparation is completed by FIB-based fine polishing of the structures at 30, 5 and 2 keV. The SEM measurements are done at an angle of 52° to the cross-section with a set magnification of 120 k to 650 k at an acceleration voltage of 2 kV and an absorption current of 0.1 nA.

Rheological experiments of thick-film PDMS gel samples are performed with the rheometer ARES-G2 from TA Instruments in compression mode with the 8 mm plate/testing specimen at 25 °C. A normal force of 0.05 N and an angular frequency of 0.1 to 100 rad/s are applied.

3 | RESULTS AND DISCUSSION

3.1 | PDMS gel surface activation

PDMS gel is a highly hydrophobic polymer that requires a prior surface activation before metallization with noble metals such as silver. Recently, we have shown the effects of the oxidation methods O₂-plasma and UV/ozone on PDMS gel surfaces by XPS survey measurements paired with quantitative elemental analysis and infrared spectroscopy.¹¹ Our investigations confirmed higher oxygen (rise from 27 at.% to 37-39 at.%) and lower carbon (drop from 49 to 40 at.%) contents due to the mentioned surface activation methods that were linear depending on the selected activation time. The decreasing carbon content is consistent with the Si–OH-formation as Si–C-groups break and carbon is released as CO₂. At the same time, the Si–OH-groups tend to condensate and form Si–O–Si-groups with further PDMS gel cross-linking.²⁰ Within these chemical processes, the silicon content is almost constant. However, both methods differ. Activation by UV/ozone treatment takes much longer than with O₂-plasma, because the ozone molecules have a lower reactivity than the ionized oxygen species.

The higher functionality with oxygen containing groups will also increase the surface energy σₛ of hydrophobic, low-energetic PDMS.²⁰ Higher energy values are reported to improve metal deposition and stability on PDMS surfaces, but no accurate values have been published.¹⁵ Instead, for other polymers, such as poly(acrylonitrile butadiene styrene), the surface energy is increased after Ar⁺-sputtering treatment from σₛ = 40 to 75 mN/m, which doubled the adhesion strength of a sputtered Cu-layer.²⁹ With σₛ = 40 mN/m, the adhesive strength of 8 MPa is already quite high for nonactivated polymer surfaces and could be a surface energy value for sufficient PDMS metallization. To prove the effects of O₂-plasma and UV/ozone on the PDMS gel, wetting experiments are conducted by contact angle measurements to calculate the surface energy with the methods of Wu²⁵ and OWRK.²⁶-²⁸

The combination of both methods is used, because the method of Wu allows a better description of hydrophobic polymer surfaces, and the OWRK method is specified for polarity changes after surface activation.²⁵,²⁷ The O₂-plasma activation enables an increase of the surface energy from σₛ, Wu = 16 mN/m to σₛ, OWRK = 61 mN/m after 30 seconds of activation (Figure 1). Instead, a σₛ, OWRK = 31 mN/m is achieved after 120 minutes by means of UV/ozone treatment, which shows its lower energetic effect.

The impact of activation processes on the PDMS gel mechanics is investigated by rheology on thick film samples. The results are shown in Figure 2A using the frequency depending storage modulus E’. Here, the enormous increase of E’ = 1.8 to 3.7 kPa at 60 minutes UV/ozone treatment or even 5.2 kPa at 120 minutes confirmed a material stiffening due to surface activation related cross-linking and formation of Si–O–Si-groups inside the PDMS gel. It is interesting to note that a short activation of 10 or 20 minutes results in a slight decrease of E’, which is probably due to changes in the adhesion behavior of the gel. The O₂-plasma, on the other hand, does not significantly change the mechanics and prevents the initially high viscoelasticity and low stiffness of the PDMS gel. As this rheological experiment is a bulk measurement, defined mechanical changes on the top side or even a distribution in depth are not measured.

To get a feeling for these questions, XPS sputter depth-profiles are created using various surface-activated PDMS samples. A high oxygen content in the depth of the PDMS layer suggested an increased polymer cross-linking and proved the influence on the gel mechanics. Figure 2B nicely shows that the O₂-plasma treatment influences the upper gel surface to depths of about 10 to 20 nm (considering a sputtering rate of 1.6 nm/min of Ta₂O₅) with a certain amount of oxygen of more than 25 at.% in pure PDMS gel, which is released by further Ar⁺-sputtering. Instead, the UV/ozone activation increases the oxygen content even at larger depths, resulting in higher Si–O–Si-cross-linking and proves the mechanical gel stiffening. However, the real depth-effect of UV/ozone cannot be detected because outgassing limits the sputtering time. The schemes illustrate the different depth-effects of both oxidation methods.

The investigations on surface activation of the PDMS gel by UV/ozone and O₂-plasma show higher surface functionality and surface energy due to a higher number of oxygen-containing groups, which allow a sufficient surface metallization according to the suggestions from the literature. However, both methods show different depth-effects, whereby presumably stiffer metal layers and thus EAP actuators can be realized by using UV/ozone-treated samples.

3.2 | PDMS gel surface metallization

The EB-PVD metallization (2 nm Ti, 46 nm Ag, and 2 nm Au) of the PDMS gel is carried out on various pretreated surfaces and examined by photography and SEM micrography of the metal surface and FIB cross-sections showing also the interface to the PDMS (Figure 3). The metallization of pure PDMS gel leads to brown, nonreflecting, and nonconductive surfaces, because metal nanoparticles are formed at a depth of several hundred nanometers of the PDMS gel due to metal
diffusion and high cohesive energy of the metals (see Figure 3A). In contrast, the oxidizing surface activation of the PDMS during subsequent PVD metallization allows the formation of metal crystallites by Volmer-Weber island growth. The UV/ozone activation (30 minutes) of PDMS results in a noncompact metal layer that is formed due to an insufficient surface energy of $\sigma_{\text{OWRK}} = 18 \text{ mN/m}$. Cracks and pinholes disturb the conductivity and reflectivity of the metal layer (see Figure 3B). An obviously sufficient surface activation with UV/ozone by 60 minutes ($\sigma_{\text{OWRK}} = 25 \text{ mN/m}$) or with O2-plasma by 5 seconds at 100 W ($\sigma_{\text{OWRK}} = 40 \text{ mN/m}$) enables the formation of compact metal layers on the PDMS gel (compare Figure 1A,B with Figure 3C,D). Here, the metal deposition leads in a single layer of overlapping grains with a size of 20 to 50 nm and thus guarantees high reflectivity and conductivity.

A further view into the metal layer stack on the PDMS gel is given by XPS sputter depth profiling, shown in Figure 4, which is a very challenging investigation due to the combination of soft and hard matter with a distinct boundary layer. As different sputtering rates of different elements or morphology cannot be considered, we apply the commonly used sputtering rate of 1.6 nm/min ($\text{Ta}_2\text{O}_5$) to make statements about the sputtering depth. The nonactivated PDMS layer shows a large distribution of metal atoms from a depth of about 40 nm. The simultaneously occurring C-, O- and Si-signals prove the large PDMS presence in the nanoparticle range (see Figure 4A). After 30 minutes of UV/ozone activation, the Au- and Ag-atoms (55 at.% Ag) are accumulated during the metallization in an upper region of approximately 5 to 25 nm depth. The high PDMS content of more than 50 at.% (assumption from the Si-content = 11-23 at.%) in this region confirms that the pinholes and cracks observed through SEM cross-sections are filled with PDMS gel (see Figure 4B). The low Ti-content compared with Au can be caused by the high reactivity and the wide distribution in the depth of the PDMS gel or different sputtering rates. A sufficient activation by 60 minutes UV/ozone or O2-plasma allows a concentration of Ag-atoms nearby the surface covered by Au (see Figure 4C,D). However, the signals of C, O, and Si in the top layer indicate the presence of PDMS gel that could penetrate through the metal layer defects to the surface and form a slight covering as it shows a pronounced contact creep behavior. By using a sputter rate of 1.6 nm/min, the metal layer thickness can be calculated to be 30 to 35 nm, which is in contradiction to the metal thickness measured by SEM (see Figure 4C,D) and obviously assumes higher sputter rates for the applied metals. The deviations in the distribution of the Ag-content on different activated PDMS surfaces are summarized in Figure 4F.
The experimental optimization of the metallization process of the PDMS gel surface in relation to previous surface activation by UV/ozone and O$_2$-plasma could be demonstrated by XPS, SEM surface, and interface analysis. By providing highly conductive and reflective layers, micromirror structures with integrated EAP thin film actuators can be developed.

The construction of the adaptive micromirrors on a glass substrate is shown in Figure 5 and consists of the overlapping (pixel area 70 $\times$ 70 $\mu$m$^2$) bottom and top electrodes separated by the PDMS gel and forming a capacitor. To realize this, the electrode layers are structured by shadow masks during EB-PVD metallization. The gel layer is previously activated by UV/ozone or O$_2$-plasma, which creates a highly reflective upper mirror. This mirror is flexible and deforms by applying a voltage to the electrodes. The viscoelastic PDMS gel allows a reversible return of the initial layer thickness after discharge.

The deformation behavior of the mirrors after electrical activation is examined with confocal microscopy and is shown by confocal 3D images in Figure 6. The deformation of the differently manufactured mirrors differs significantly. The metallic mirror layer on the PDMS gel, which was previously treated with O$_2$-plasma, shows a centric or

| Photograph | SEM-micrograph from metal surface | SEM-micrograph from FIB cross-section |
|------------|----------------------------------|--------------------------------------|
| (A) Pure PDMS | ![Image] (A) | ![Image] (A) |
| (B) UV/ozone 30min | ![Image] (B) | ![Image] (B) |
| (C) UV/ozone 60min | ![Image] (C) | ![Image] (C) |
| (D) O$_2$-plasma 5s | ![Image] (D) | ![Image] (D) |

FIGURE 3 Poly(dimethylsiloxane) (PDMS) gel metallized by physical vapor deposition (PVD) with different surface activation. The metal layer quality is shown by photos and scanning electron microscopy (SEM) micrographs of the metal surface and focused ion beam (FIB) cross-sections. (A) No activation leads to brown, nonreflecting, and nonconductive surfaces because metal nanoparticles are distributed in the PDMS. (B) Oxidizing surface activation allows the formation of overlapping metal crystallites on the gel surface due to Volmer-Weber island growth. Because of lower dose, time or intensity of surface activation, no compact metal layer is formed. Cracks and pinholes cause poor conductivity and reflectivity. (C, D) Sufficient surface activation allows the formation of compact nanocrystalline metal layers on PDMS gels containing grains with a size of 20-50 nm.
concave deformation. In contrast, a UV/ozone-treated sample allows a convex or edge-like deformation of the mirror layer. This difference is due to surface activation. The UV/ozone treatment causes a stiffening of the PDMS gel into deep layers, which is proved by rheology and the XPS sputter depth-profiling measurements. This high stiffness is also shown by a much higher activation voltage of 500 V to realize a comparatively large mirror deformation as with the low stiff O2-plasma-treated samples. The mirror cross-sections of both deformation types are still used to show the image formation of a possible varifocal micro mirror application.

![Activation](Activation_U positiv.png)

![Relaxation](Activation_U negativ.png)

**FIGURE 4** X-ray photoelectron spectroscopy (XPS) sputter depth profiles of the top metallization (2 nm Au, 46 nm Ag, and 2 nm Ti) on the poly(dimethylsiloxane) (PDMS) gel with different surface activation using (A) pure PDMS gel, UV/ozone (B) 30 min and (C) 60 min, (D) 5 s O2-plasma and a (E) reference on glass. (F) Summary of the silver content.

**FIGURE 5** Scheme of the micromirror construction forming a capacitor in the overlap area (pixel). The top electrode is flexible and can be deformed by electrical activation with simultaneous reduction of the gel thickness z.

**FIGURE 6** Micromirrors with concave or convex mirror deformation after electrical activation depending on the surface activation of the poly(dimethylsiloxane) (PDMS) gel and the mechanical properties. The shapes of the micromirror surfaces are assumed and simplified to a spherical type in order to realize a theoretical image formation (S = mirror center, F = focal point, M = double focal point). This allows a calculation of the focal length for the application of varifocal micromirrors.
In this regard, it is possible to calculate the focal length changes of both mirror types at different voltages by fitting a circular function to the shape of the mirror cross-section (see Figure 6). To realize this fitting, a simplified spherical mirror surface is assumed. However, the real shapes of the concave and convex mirrors are more complex and correspond more to nonspherical mirror types. The values of the focal lengths are shown in Table 1 and give a small overview how the mirror deformation can be electrically tuned and influences the focal length.

### 4 | CONCLUSION AND OUTLOOK

In summary, we have demonstrated a new concept of varifocal micromirrors with integrated thin film actuator based on metallized PDMS gel. The metallization process of PDMS and its previous surface activation by O$_2$-plasma and UV/ozone was comprehensively analyzed with respect to surfaces, interfaces, and gel mechanics by XPS, SEM, wetting experiments, and rheology. The UV/ozone and O$_2$-plasma treatments were optimized to increase the surface functionality and energy due to higher oxygen contents. Because the UV/ozone treatment had a pronounced depth-activating effect, the mechanics of the PDMS gel showed a significant stiffening. Insufficient surface activation led to the formation of separated nanoparticles or at least isolated metallic aggregates instead of a compact metal layer. On the other hand, a sufficient metallization produces highly reflective and conductive metal layers on the PDMS gel, which were used as electrodes in EAP actuator thin films. They were able to exhibit an electrode and mirror deformation due to activation by electrical voltage. The mirror deformation was analyzed by confocal microscopy and discussed theoretically with respect to image formation and focal length adjustment. For future practical application of varifocal micromirrors, further investigation of imaging by suitable optical methods is absolutely necessary. Furthermore, the electrode design and the deformation of the mirrors could be optimized to realize spherical mirror shapes. Applications for nonspherical micromirrors are also conceivable.

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