C₄F₈ plasma treatment for the modification of the focal length of liquid-based plano-convex lenses on different substrates

Christoph Gerhard¹, Gerrit Mielke¹, Daniel Tasche¹,²

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Abstract
In this contribution, we present a plasma-based approach for modifying the focal length of plano-convex lenses realised by the deposition of droplets made of optical cement. For this purpose, PMMA and soda-lime glass substrates were treated with a direct dielectric barrier discharge plasma at low pressure. To obtain plasma-induced changes in surface energy of the substrates by the formation of functional groups, octafluorocyclobutane (C₄F₈) was used as process gas. Such treatment lead to a notable decrease in surface energy and especially its polar fraction, which is attributed to the generation of fluoro-carbon compounds such as polytetrafluoroethylene (PTFE) as also confirmed by FTIR spectroscopic measurements. It is shown that this effect has a considerable impact on the contact angle and radius of curvature, respectively, of the deposited cement droplets where differences by a factor of about two were obtained. After curing of the cement, front focal lengths in the range from approx. 5 to 13 mm were realised depending on the duration of the plasma pre-treatment. The cement lenses finally feature dimensions in the range of some millimetres.

Keywords Liquid-based lenses · Optical cement · Plasma treatment · Lens printing · Surface modification

1 Introduction
Micro lenses or smallest macro optical components with dimensions of some millimetres have become important elements of modern optical systems. As an example, lens arrays are used for the homogenisation of laser beams [1] or for the detection of defective vision via wave front analysis in ophthalmology [2]. For the production of such optical devices, different approaches and techniques were developed in the last few decades to realise the necessary patterns on substrates [3]. The methods of thermal reflow [4] and micro jet printing [5] can be applied as the essential preparation step prior to etching in micro lithography or for the direct generation of optically functional devices on plane or even curved substrates. For the latter application, Okamoto and co-workers have shown the suitability of simple pouring processes. Here, arrays consisting of lenses made of the UV-curable organic polymer thioether methacrylate (TEMA) with diameters in the range from 0.2 to 2 mm were generated by pouring and subsequent UV-curing and tempering [6]. Another approach was reported by Chang et al. where micro lens arrays were produced by roller stamping of a polydimethylsiloxane (PDMS) pre-polymer [7]. Moreover, Bardinal et al. have presented the fabrication of micro lens arrays with focal lengths in the range from some tens to hundreds of microns by the deposition of polymer droplets on coated glass substrates using a micro-cantilever spotter system [8]. Here, the volume and dimensions of the generated lenses were controlled by the contact time of the spotter system on the substrate. Such printing of precise micro lenses made of a two-component epoxy resin via micro plotting systems was also reported by Zang et al. [9].

An interesting point for the production of liquid-based lenses is the possibility of varying the contact angle between the applied optical liquid drop and the solid substrate surface selectively. This can be achieved by changing either the surface tension of the liquid or the surface energy of the substrate according to the Young–Laplace equation. As a consequence, the radius of curvature and finally the focal
length of the formed plano-convex lens can be influenced. Such a variation of the contact angle can quite easily be realised by varying the temperature and viscosity, respectively, of the used liquid [10, 11] where a larger viscosity results in a smaller radius of curvature and a shorter focal length. This approach was applied by Kim and co-workers for the application of tailored micro lenses on fiber end faces [12]. However, heating an optical liquid is limited to a certain temperature range, restricting the range of realisable contact angles. Another approach for shaping liquid-based lenses was presented by Lee et al. After micro jet printing, the generated PDMS-lenses were stored overhead to benefit from the gravity-induced mechanism of self-organisation of hanging droplets. After the actual curing process of the first PDMS-droplet several further layers were added sequentially, finally resulting in aspherically-shaped lens surfaces [13].

As already mentioned above, shaping of liquid lenses can also be obtained by changing the surface energy of the solid substrate. This approach was applied by Hartmann et al. for the production of micro lens arrays where the substrate was coated with a hydrophobic material. After applying an UV-curable monomer solution, spherically-shaped drops were formed by self-assembly due to the proper surface energy of the hydrophobic material [14]. The opposite effect, i.e. the formation of hydrophilic surface properties, was presented by Chen et al. First, a plane a mask made of an epoxy-based negative photo resist was realised on a substrate, resulting in a pattern of hydrophilic circular areas. The surface shape of subsequently applied drops of photo resist drops was then influenced due to hydrophilic confinement [15]. A completely different method was reported by Vespi et al. Here, the shape of liquid droplets was controlled by applying an electric field to the substrate. The driving force of this method is the electro-hydrodynamic effect that induces high surface charge at the liquid surface [16].

In this paper, the suitability of direct plasma treatment of substrates for shaping subsequently applied droplets made of optical liquids is presented. Plasmas allow a number of advantageous surface effects such as coating, cleaning, and activation and an accompanying change in surface energy and contact angle of liquids, respectively. For instance, a change in contact angle is achieved by simple plasma cleaning and the removal of carbonic contaminations from surfaces [17, 18]. For this purpose, a mixture of argon as carrier gas with smallest admixtures of oxygen (0.03 to 2%) has turned out to be an appropriate process gas for plasma operation where surface cleaning is rapidly obtained (in the range from some tens of milliseconds to one second) [19–21]. Apart from cleaning, plasma treatment also allows an activation, i.e. a chemical modification of surfaces, for example by attaching molecular chains or functional groups such as polymerised hydroxyl or carboxyl groups (–OH or –COOH) via plasma polymerisation. This effect is used for a modification of surface wettability [22] where both hydrophilisation and hydrophobisation can be realised by the choice of the used process gas and type of plasma as shown by Yamamoto et al. [23]. Fang and co-workers achieved even a long-term stable increase in hydrophobicity of glass surfaces by applying a plasma at ambient air and a poly-dimethyl siloxane fluid to the substrate. In this way, a hydrophobic layer was generated by plasma activation and the formation of chemically bonded CH₃-containing groups [24]. Plasma treatment is thus a powerful tool for the modification of the surface energy and the wetting characteristics of any surface. Against this background, the approach of plasma-induced surface modification and activation was applied in the present paper. To our best knowledge, this is the first work reporting the use of plasmas for tailoring the radius of curvature and focal length, respectively, of liquid-based plano-convex lenses.

2 Materials and methods

2.1 Generation of liquid-based lenses

For the generation of liquid-based lenses, a two-component optical cement (type M-62 from Lens Bond/Summers Optical) was used as optical liquid. Such cements feature high transparency in the visible and near infrared wavelength range and are usually applied for fine cementing of lens or prism groups in optical manufacturing [25]. According to the supplier’s datasheet, the used cement features a viscosity of 275–320 mPa·s and a surface tension of 44 mN/m; its refractive index amounts to 1.53 before and 1.55 after full curing at 25 °C [26]. Full curing of the cement is achieved by storing at ambient temperature for 48–96 h, depending on the catalyst ratio. In the present case, the cement was mixed at a catalyst ratio of 3:100, resulting in a curing time of 72 h.

After mixing, cement droplets were applied on two different types of substrate. First, poly(methyl methacrylate) (PMMA) plates with a thickness of 6 mm were used. This well-established optical plastic features a refractive index of $n_e = 1.4945$ [27]. Second, cement lenses were generated on microscope glass slides made of soda-lime glass with a thickness of 1 mm and a refractive index of $n_e = 1.5253$ [28]. For the deposition and the measurement of the diameter and contact angle of cement droplets, a commercial contact angle measurement system (Contact Angle Measurement System G10, Krüss GmbH) with an integrated dosing unit was employed. The cement volume used for each lens was 8 µl. The measurement system was also used for the determination of the contact angle between the cement droplet and the substrate surface and the drop.
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These parameters were measured directly after the application of the droplets and after final curing which was performed at room temperature for 72 h. In addition, the plasma-induced changes in surface energy of the substrates were detected using this measurement device.

2.2 Plasma pre-treatment of substrates

To vary the surface energy characteristics of the used substrates and the contact angle of the cement, respectively, a direct dielectric barrier discharge (D-DBD) plasma was applied. In such plasma configuration, the substrate is placed between the high voltage electrode and the ground electrode as shown in Fig. 1. The substrate itself thus acts as dielectric barrier, resulting in a high efficiency of plasma-induced mechanisms at the substrate surface such as the formation of dipoles or the initiation of chemical reactions. The plasma source was a rotational-symmetric cone-shaped plasma source as described in more detail in [29]. It consists of an internal high-voltage (HV) electrode and an external ground (GND) electrode. This device was mounted in a vacuum chamber and experiments were performed at low vacuum at a pressure of 50 Pa. Consequently, the plasma was ignited over the entire sample surface, forming a closed surface discharge.

Plasma treatment was carried out for 1, 3, 5, 7, and 10 s where octafluorocyclobutane (C₄F₈) was used as process gas for plasma operation to modify the surfaces of the substrates chemically by attaching functional groups. As a consequence of Paschen’s law, different breakdown voltages had to be applied in the course of plasma treatment due to the different thicknesses of the substrates (and the resulting difference in thickness and electrical resistance of the effective discharge gap). The particular values of the applied voltage were 7.7 kV for PMMA and 6.8 kV in the case of soda-lime glass. The frequency, i.e. the repetition rate of the high-voltage pulses was 26.2 kHz for both materials.

To detect plasma-induced changes in the chemical composition of the used substrates, infrared absorption spectra were measured (using a Frontier MIR/FIR Spectrometer from Perkin Elmer) before and after plasma treatment within a wavenumber range from 4000 to 400 cm⁻¹, corresponding to wavelength range from to 2.5 to 25 µm. For high sensitivity of the measurement the method of attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy was applied.

2.3 Determination of the focal length

The focal lengths of the deposited cement drops were determined in two ways. First, the focal length was calculated based on the measured contact angle and the diameter of the cement drops applying the following theoretical approach: once a drop is deposited on a plane substrate surface, a spherical segment is formed as long as surface tension of the cement can withstand deformation due to gravitation. The spherical segment can then be characterised by its contact angle with respect to the substrate surface and its dimension or diameter as shown schematically in Fig. 2.

The radius of curvature $R$ of the cement drop, which is hereafter regarded as a plano-convex lens made of cement, can be derived from its diameter $D$ and the contact angle $\theta$ according to

$$R = \frac{\mu}{2 \sin \theta}$$  \hspace{1cm} (1)

Assuming perpendicular incidence of light on the plane surface, the effective front focal length $f_{\text{front}}$ of the cement lens is then given by

\[
\text{(a)} \quad \text{(b)}
\]

Fig. 1 Schematic of the setup used for plasma pre-treatment of substrates

Fig. 2 Photograph of a cement drop (a) and overview on geometric parameters of a spherically-shaped cement drop on a plane substrate surface including the contact angle $\theta$, the drop diameter $D$ and the radius of curvature $R$ (b)
with \( n_c \) being the refractive index of the used cement.

Second, the front focal length was measured via a simple opto-mechanical setup consisting of a laser source, a reflective diaphragm, a beam dump and a camera as shown in Fig. 3.

Here, a HeNe-laser with an emission wavelength of 543 nm was used. The generated cement lenses were brought into the laser beam and the front focal length was determined by shifting the lenses with respect to the diaphragm and measuring the laser intensity on the camera chip. For the configuration where the focused laser beam passed the diaphragm entirely and arrived on the absorbing beam dump the lowest laser intensity was measured on the camera. The distance between this position of the lens vertex and the diaphragm was defined as the front focal length, see Fig. 3.

\[
 f_{\text{front}} = \frac{R}{n_c - 1} 
\]  

(2)

3 Results and discussion

3.1 Surface energy and contact angle

The applied plasma treatment has a notable impact on the polar and the dispersive part of the surface energy and the resulting total surface energy of both investigated substrate materials, PMMA and soda-lime glass, as shown in Fig. 4.

It can be seen that all fractions of the surface energy are significantly decreased regardless the substrate material. After merely one second, the total surface energy and its dispersive fraction approach a common constant value of approx. 13.5 to 14 mN/m. Moreover, the polar fraction of surface energy nearly disappears. This behaviour can be expressed by the polarity \( P \) given by

\[
P = \frac{\gamma^P}{\gamma_{\text{tot}}}
\]  

(3)

where \( \gamma^P \) is the polar fraction of the total surface energy \( \gamma_{\text{tot}} \). For PMMA, polarity decreases from an initial value of the untreated surface of 0.17–0.03 after ten seconds of plasma treatment. This corresponds to a ratio of 5.7. In case of soda-lime glass, a decrease in polarity from 0.65 to 0.12 was measured, resulting in a ratio of 5.5. It can thus be stated that in terms of polarity, the plasma treatment has the same effect on both substrates as expressed by the similar ratios. This effect is most likely a direct reaction of fluorine provided by the plasma with the sample surface or with surface-adherent carbonaceous contaminants. In case of PMMA (C\(_5\)H\(_8\)O\(_2\)), carbon is an essential component of the material. In contrast, soda-lime glass bulk material does not contain any carbon. However, glass surfaces are usually contaminated with carbon and hydrocarbons originating from cleaning agents.
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For plasma treated PMMA, the mean total surface energy that was calculated from the particular values for the treatment duration range from 1 to 10 s amounts to 14.01 ± 0.93 mN/m. A quite similar value, 13.62 ± 0.44 mN/m, is found for glass. Since the start values of the total energy were significantly different (i.e. 43.16 ± 1.17 mN/m for PMMA and 63.79 ± 1.00 mN/m for glass), this comparison clearly shows that the surfaces feature quite the same conditions after short-term plasma treatment, indicating the formation of surface-adherent fluorocarbon compounds. For PMMA, the formation of such compounds due to plasma-induced surface modification was verified by ATR-FTIR spectroscopic measurements shown in Fig. 5.

In this spectrum, characteristic footprints of PMMA can be identified at a wavenumber of 1140 cm⁻¹ (C–O) and 1300 cm⁻¹ is superimposed by the C–O peak from the PMMA substrate an obvious decrease in IR-transmission at a wavenumber of 1213 cm⁻¹ can be observed (highlighted in yellow in Fig. 5). Here, transmission is reduced from 73.70% before plasma treatment to 58.36% after plasma treatment, corresponding to an increase in absorption by 15.34%. This indicates the formation of CF₂ groups (asymmetrical CF₂ stretching) [34]. Due to the superposition by the C–O peak at 1140 cm⁻¹, the second characteristic line of CF₂ groups at 1155 cm⁻¹ (symmetrical CF₂ stretching) [34] cannot be detected and evaluated. However, for another characteristic line of these groups (CF₂ wagging), i.e. 639 cm⁻¹ [35], a decrease in transmission from 90.75 to 87.90% and an increase in absorption by 2.85%, respectively, is found. These detected plasma-induced compounds are known to be highly hydrophobic.

For soda-lime glass, no significant changes in the measured FTIR spectra before and after plasma treatment were observed. This can be explained by the marginal thickness of the formed fluorocarbon layer. The layer of surface-adherent carbonaceous contaminants such as hydrocarbons normally features a thickness of several angstrom [36]. Such thin layers with thicknesses much lower than the measuring wavelength cannot be detected by FTIR spectroscopy. However, C–F groups were most likely formed in the course of the plasma treatment as indicated by the results obtained for the surface energy (see Fig. 4) and the contact angle of cement as presented hereafter (see Fig. 6). It can finally be assumed that the plasma-modified layer thickness is much higher in case of carbon-based PMMA since the bulk material of this medium contains a considerable fraction of carbon.

The effect of C₄F₈ plasma-induced formation of fluorocarbon compounds on surfaces is well known and applied for the deposition of Teflon-like or fluorocarbon films [37–39]. For instance, it is used for the generation of superhydrophobic surfaces [40]. A comparable behaviour was also observed in the present case: Depending on treatment time, the applied plasma treatment allows a selective change in surface energy and adhesion, i.e. the contact angle θ of liquids applied on the surface, which in the present case is optical cement. This effect is shown in Fig. 6.

It turns out that the most significant change occurs for PMMA. Here, the contact angle of the applied cement was 35.30° before and 68.43° after plasma treatment for ten seconds. It was thus nearly doubled (factor 1.94). Due to the curing process at room temperature for 72 h, the contact angles were generally reduced with respect to the liquid state values. After hardening, the contact angles varied from 25.30° before to 57.47° after plasma treatment. Here, the increase in contact angle thus can be expressed by the factor 2.27. In case of soda-lime glass, the plasma-induced change is noteworthy lower; the contact angles feature a ratio of merely 1.41 (44.50° → 62.63°) before and 1.54 (33.67° → 51.90°) after curing. These differences between the contact angles as measured directly after cement deposition and after curing can be explained by a spreading of the cement drop in the course of storing due to its fluidity. Moreover, the used cement features a shrinkage of approx. 4% according to the supplier’s datasheet [26]. Such shrinkage may also contribute to the systematic delay of the curves shown in Fig. 6.
3.2 Properties of liquid-based lenses

According to Eq. (1), the contact angle is directly related to the radius of curvature of the cement-air interface, which is formed due to surface tension. Consequently, the focal length of the cement drop follows from the contact angle. Against this background, the contact angle and the diameter of drops was measured after curing and complete hardening of the cement. Based on these values the front focal length was calculated according to Eqs. (1) and (2). Here a refractive index of 1.55 as specified for the cured cement by the supplier’s data sheet [26] was taken. The front focal length was additionally measured as described in detail in Sect. 2.3.

As shown in Fig. 7, the calculated and the measured values are generally in quite good accordance.

It turns out that for PMMA, the front focal length can be varied in the range from 13.38 mm to 5.06 mm (measured values), so the maximum change in front focal length is 8.32 mm (factor 2.64) In case of soda-lime glass, this maximum change is 5.40 mm (10.66 mm → 5.25 mm), corresponding to a factor of 2.03. It can thus be stated that within these ranges, the radius of curvature and the focal length, respectively, can be adjusted by controlling the plasma treatment duration where the most significant changes take place within the first second of plasma treatment. For longer treatment durations a nearly constant focal length of about 6 mm is obtained. This behaviour can be explained as follows: Once a closed layer of C–F groups has formed on the substrate surface, the surface energy and contact angle, respectively, are completely altered with respect to the untreated substrate. For realising focal lengths between the initial state of an untreated surface and the final state after plasma treatment for more than one second, shorter treatment durations should thus be chosen. In that vein, surfaces
that are not entirely covered by the plasma-induced layer could be generated.

To visualise the size of the generated lenses some selected geometric parameters are finally shown in Fig. 8. These parameters are the cement drop or lens diameter $D$, the radius of curvature $R$, and the sagitta $S$ that was calculated according to

$$ S = R - \sqrt{R^2 - \left(\frac{D}{2}\right)^2} $$

(4)

It can be seen that the lens sizes are well in the millimetre-range. This order of magnitude could be referred to as an intermediate between micro optics and classical macro optical components. The size of liquid-based lenses produced by the presented approach could further be decreased by reducing the volume of applied cement and by choosing another type of cement with a lower viscosity. For this purpose highly fluid UV-curing optical cements are in hand.

The use of such cements or the change in cement viscosity, for example via heating, may further broaden the range of realisable focal lengths. Apart from classical fine cements used in optics, non-conventional polymers could be used for this purpose [41]. Moreover, the type of plasma treatment plays an essential role. Depending on the operating parameters quite differing surface effects can be obtained. For instance, comparative experiments using a direct dielectric barrier discharge plasma at atmospheric pressure where the process gas was argon have shown that also an increase in focal length instead of a decrease as presented in this paper can be realised. This effect is due to a decrease in contact angle of the applied cement as a result of a plasma-induced surface cleaning and a potential activation. Since the choice of the plasma process gas composition allows the formation and attachment of either hydrophobic or hydrophilic groups on the substrate surface, the variation of both the plasma process parameters and the used cement optionally offers a wide range of focal length variations.

4 Conclusion

The plasma-based approach presented in this paper has turned out to be a suitable method for tailoring the radius of curvature and focal length, respectively, of liquid-based plano-convex lenses made of optical cement in a certain limited range. Due to the induced chemical surface modification, merely a decrease in focal length with respect to the initial value on pure, untreated substrates was achieved. However, it was observed in comparative experiments that the use of other plasma process gases and pressure ranges principally allows for an increase in focal length. In any case, the major changes take place within the first second of plasma treatment. For longer treatment times a quite constant focal length is found. To realise smaller changes in focal length, plasma durations of some tens to hundreds of milliseconds should thus be applied. Due to the circuit and reaction times of the used plasma source and the high-voltage generator, the repeatable realisation of such short treatment times is quite challenging, but possible. Further investigations and improvements of this approach will thus be part of future work. Moreover, the use of other cements with different viscosity is of great interest to evaluate the range of realisable focal length variations and to identify suitable materials for liquid-based lenses.

The presented technique could be used for the fabrication of lenses with tailored focal lengths where further miniaturisation of the generated lenses by reducing the volume and viscosity of the used cement could even allow for the generation of micro lens arrays. A plasma treatment could also be carried out on fibre end faces to generate lenses with defined focal length for fibre coupling. For all applications, the presented differences in lens size and geometry due to spreading of the droplets and shrinkage of the cement should be taken into account initially. Further improvements of the lenses could finally be achieved by the curing process, e.g. by the use of cements that are cured and hardened by UV
irradiation within some seconds to avoid deformations which can occur during comparatively long curing processes at ambient conditions for several days. In contrast, the shape of the droplets could also be positively influenced by a selective utilisation of deformations during long-term curing via overhead storing. As suggested by some authors, this allows the generation of aspherically shaped lenses.

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