PAPER

Detection of atmospheric pressure plasma-induced removal of fingerprints via analysis of histograms obtained by imaging ellipsometry

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Abstract

In this publication we report on the suitability of imaging ellipsometry for the semi-quantitative investigation of a contactless removal of human fingerprints from surfaces by an atmospheric pressure plasma treatment. Special attention is paid to the impact of the applied plasma on the complex mixture of biological substances of a fingerprint. For this purpose, the cleaning effect of an argon plasma at two different electrical powers is investigated. By using imaging ellipsometry as an analysis method it could be shown that the cleaning efficiency increases with increasing electrical power and plasma treatment time. In addition, measurements were made by means of x-ray photoelectron spectroscopy (XPS) in order to characterize the chemical composition of surface-adherent contamination and its plasma-induced modifications. It was found that especially the amount of organic impurities is reduced in the course of plasma treatment.

1. Introduction

In many technical areas, decontaminated surfaces are indispensable, for example in the packaging of food [1]. Even in more exotic fields of application such as space missions, care is taken to prevent biological contamination of the solar system [2]. Clean surfaces must also be guaranteed for optical components, so that in addition to ultrasonic cleaning methods, cleaning methods based on a combination of UV light and ozone [3, 4] or a pulsed excimer laser are used [5]. Some of the contamination of optical components, which occurs either during handling or manufacturing, is due to unwanted carbonaceous contaminants such as fingerprints on the surfaces. Due to the cost-efficiency of atmospheric pressure plasmas and their proven ability to remove carbonaceous compounds from surfaces without any contact [6], the decontaminating effect on silicon surfaces contaminated with human fingerprints is examined in this contribution. The high potential of such plasmas for the precision cleaning of optics by a removal of residual contaminants was already shown in previous work [7–9]. For instance, an increase in laser-induced damage threshold can be realised by the plasma-induced removal of absorbing surface-adherent carbonaceous contaminants from optical media [10]. Such plasma cleaning and further associated effects as for example surface activation also allows an enhanced stability of cemented optics [11].

The human fingerprint is a very complex mixture of different substances. The main components are eccrine and apocrine sweat, sebum secretion, substances from external contamination with the environment, and proteins. Eccrine and apocrine sweat is a mixture of inorganic substances and water-soluble organic substances (NaCl, urea, amino acids). Sebum secretion consists of fat-soluble substances such as glycerides, fatty acids, wax esters, squalenes, sterols and sterol esters [12, 13]. As shown by several authors, certain biomolecules have...
already been successfully removed using low-pressure plasmas [14, 15]. Atmospheric-pressure plasmas were moreover applied to sterilize surfaces in other studies [16–18]. Plasma cleaning is ultimately based on transfer of the impurity into the gas phase. Basically, chemical and physical processes contribute to such removal [5]. Chemical processes include the formation of active species such as ozone or atomic nitrogen in the air plasma, which can oxidize or reduce surface contaminants. The physical cleaning effects include thermal decomposition as well as bombardment with heavy particles (sputtering) [19, 20], and the decomposition of molecular compounds such as hydrocarbons by UV radiation. Depending on the process gas used in the plasma, these cleaning effects are differently pronounced and can overlap [21]. In this work, the suitability of the particularly applied atmospheric pressure plasma for the removal of human fingerprints from surfaces is investigated applying the analytical method of imaging ellipsometry, since it had already been shown in work by An [22] that fingerprints could be detected particularly well with this method, especially on surfaces of semiconductor materials. The advantages of imaging ellipsometry versus optical microscopy and Raman spectroscopy in the analysis of thin films are resolution, flexibility, measurement speed and range [23]. Analysis of polarization results in better contrast than pure intensity-based methods [24]. Compared to x-ray photoelectron spectroscopy (XPS), sample preparation is quite simple and vacuum technique is not required when imaging ellipsometry is used.

The used plasma does not require a vacuum chamber and can therefore be easily integrated into existing manufacturing processes. Furthermore, the use of argon as a process gas enables economical use due to the low ignition voltage. The efficiency of this contactless and simple cleaning method based on atmospheric pressure plasma is additionally screened by x-ray photoelectron spectroscopy for comparison.

2. Methods

2.1. Sample preparation

For the investigation of plasma-induced surface cleaning, a a polished silicon wafer was cleaned with isopropanol and a fiber-free tissue so that no visible impurities remained on the surface. Then, a fingerprint was placed on the wafer following a slightly modified procedure based on the standard procedure described in [25]:

1. The tip of the thumb was cleaned with a tissue (Carl Roth, type Rotizell) soaked in isopropanol and then wiped with a dry tissue.
2. Ten strokes with the cleaned thumb were made carefully and evenly from the bridge of the nose to the cheeks.
3. Ten strokes were then applied carefully and evenly from the center of the chin to the lower edge of the chin.
4. Subsequently, ten strokes were applied carefully and evenly from the center of the forehead to the temples.
5. Afterwards, ten strokes were made with the thumb through the hair.
6. Finally, the thumb and index finger were carefully pressed together and rubbed for a few seconds.
7. The silicon wafer was placed on a laboratory balance, and a fingerprint was applied to the surface of the wafer with a defined force of approx. 1.5 N in order to produce the most reproducible thickness and quantity of contamination.

This method results in a fingerprint with inhomogeneous thickness distribution, which varies between 0.1–1 μm as determined with the aid of a laser scanning microscope (Zeiss, model LSM 700).

2.2. Plasma treatment

The plasma treatment of the samples was performed by a dielectric barrier discharge (DBD) as shown in figure 1. Here, a dielectric made of ceramic was used at the high voltage electrode. The dielectric at the ground electrode was given by a glass plate of approximately 1 mm thickness. The process gas argon was fed through a slit-shaped diffuser into the discharge gap between the electrodes with a constant gas flow of 2 l min$^{-1}$. The entire area of the plasma discharge was encapsulated from the ambient air by a cover made of PMMA, so that a homogeneous atmosphere of the introduced gas was given in the chamber. With the volume of the used chamber of 1.2 l and a given gas flow, rinsing with argon had to be performed about half a minute before the experiment. The samples were treated in consecutive experiments so that the accumulated treatment time $t$ for each sample was 60 s, 120 s, 240 s and 600 s in total. The operating parameters at the two operating points (OP) of the plasma source are listed in table 1. The gas temperature in the discharge gap was determined with the aid of a fiber thermometer (FISO Technologies, model UMI universal Multichannel Instrument) before plasma treatment of the samples. Optical
emission spectroscopy (OES) was used to identify reactive species and rotational temperature in the plasma discharge. Spectra were taken using an Échelle-Spectrometer (LTB Lasertechnik Berlin, model Aryelle-Butterfly 400), calibrated to wavelength and relative intensity, with a wavelength resolution of <80 pm. For both power settings, three spectra with an integration time of 100 ms each were taken in the wavelength range of 300 to 960 nm, dark-corrected and integrated. The resulting spectra show reactive OH (A-X) and ArI species, whereas higher excited species and impurities from the surrounding air as nitrogen or further oxygen species are absent. The Boltzmann-plot method was used to determine the rotational temperature, parameters were taken from Chidsey et al [26]. The results are summarized in table 1. Electron density is assumed to be below 10^{20} m^{-3} as a determination using the Stark broadening technique was not possible due to the strong influence of Doppler broadening [27]. The U-I method was used to calculate the electrical power where the voltage and current were measured with a voltage probe (Tektronix, model P6015A) and a current monitor (Pearson Electronics, model 2877), respectively. An electric pulse and its voltage and current components are shown in the inset of figure 1.

### 2.3. Data acquisition by imaging ellipsometry

To characterize the cleaning effect on the surface, the contactless measuring method of imaging nulling ellipsometry was applied. The setup of the used device (Accurion, model nanofilmepy4) is shown schematically in figure 2. For the qualitative examination, a simple intensity image of the surface was taken for all treatment times; this image allows a visual evaluation of the cleaning condition of the surface. The data from the background corrected intensity measurements are not suitable for statistical analysis. To obtain quantitative information about the cleaning effect, polarized light is used in the next step where ellipsometric parameters $\Delta$ and $\Psi$ are recorded and finally evaluated. The ellipsometric parameters are defined as follows [28]:

$$\Delta = \delta_p - \delta_i$$

Table 1. Electrical parameters and gas temperature during plasma treatment for two operating points (OP).

| Parameters                        | OP 1   | OP 2   |
|-----------------------------------|--------|--------|
| Electrical power [W]              | 4.05   | 6.05   |
| Power surface density [W·cm^{-2}] | 0.32   | 0.48   |
| Amplitude [kV]                    | 3.42   | 4.52   |
| Electrical current [A]            | 0.76   | 1.38   |
| Frequency [kHz]                   | 19.38  | 19.38  |
| Pulse duration [$\mu$s]           | 0.58   | 0.61   |
| Gas temperature [°C]              | 27.4   | 29.0   |
| Rotational temperature [°C]       | 211 ± 181 | 181 ± 157 |

Figure 1. Schematic representation of the setup for plasma treatment of the samples: 1. HV-ceramic electrode, 2. Sample (silicon wafer with fingerprint), 3. Glass plate (second dielectric), 4. Aluminum block (ground), 5. Slit-shaped diffuser, 6. Shielding container made of PMMA, 7. Oscilloscope, 8. Voltage probe, 9. Current monitor; Inset: Single electric pulse of the plasma discharge.
Here, $\Delta$ measures the phase difference $\delta_p - \delta_s$ between p- and s-polarized light after reflection. The p(s)-polarization indicates the component of an electric field of light that is parallel (perpendicular) to the plane of incident. $\Psi$ on the other hand describes the magnitude ratio of the reflection coefficients $r_p$ and $r_s$ between these two wave components. The change of the polarization state during reflection at the investigated sample surfaces allows a quantitative statement, which is described in more detail in section 2.4. All measurements were performed at room temperature at an angle of incidence of $53^\circ$ and a wavelength of 550 nm. Each merged image consists of 20 single images in size of $675 \mu m \times 525 \mu m$ (aspect ratio 4:5), which were taken by a Scheimpflug lens in spatial resolution and then merged with an overlap of 3 $\mu m$ per image to a total area of $2640 \mu m \times 2500 \mu m$. For the comparability of ellipsometric measurements before and after plasma treatment a sample holder with a positioning accuracy of 50 $\mu m$ was used.

2.4. Data processing

During the evaluation of the measurement results, a region of interest (ROI) of $2110 \times 2210$ pixels was placed on each merged $\Delta$ and $\Psi$ map. Each pixel is assigned a specific $\Delta$ and $\Psi$ value so that the distribution of the ellipsometric parameters can be plotted using a histogram analysis. Such histogram analysis was performed for the clean silicon surface, the untreated fingerprint, and for all treatment steps in the temporal range from 60 s – 600 s. The measured values were then analyzed by mean value and variance, so that the influence of plasma cleaning can be expressed by these parameters. In doing so, the cleaning effect can be evaluated semi-quantitatively.

2.5. XPS-analysis

For the XPS measurements, a monochromatized x-ray photoelectron spectrometer (Ulvac-phi, model PHI VersaProbe II) was used. The system is equipped with an Al-K$_x$ source at a photon energy of 1486.6 eV and was operated at a base pressure of $2 \cdot 10^{-6}$ Pa. With this system, the Ag 3d$_{5/2}$ peak was measured with a FWHM of 0.6 eV at a pass energy of 23.5 eV. During all measurements, active charge compensation was applied with a cool cathode electron flood source and low energy argon ions. All measurements were conducted at room temperature. The x-ray power was set to 25 W; the beam size was 100 $\mu m$ in diameter. The electron take-off angle was kept constant at $45^\circ$. A constant analyzer energy mode was applied with a pass energy of 23.5 eV for high-resolution spectra. Data processing and analysis was carried out with appropriate software (Ulvac-phi, software package MultiPak v9.9.). The energy scale was corrected by using the C1s peak at 284.8 eV as a charge reference. Peak fitting analysis was achieved using Voigt profiles and a Shirley background was subtracted from all spectra. For fitting of the samples in the C1s energy level, the FWHM were kept at the same value for all peaks within one measurement, and peak separation between the C–C/C=C–H peak and the subsequent peaks were set to 1.5 eV, 2.8 eV and 4 eV, respectively. No other FWHM or binding energy constraints were applied for peak fitting.

$$\Psi = \arctan \left( \frac{|r_p|}{|r_s|} \right)$$

Figure 2. Schematic illustration of the used ellipsometer. 1. Laser-driven xenon lamp, 2. Optical grid for wavelength selection, 3. Polarizer, 4. Wave plate, 5. Sample holding device with sample, 6. Scheimpflug lens, 7. Analyzer, 8. CCD-Detector, 9. Motor controller, 10. Computer.
3. Results

3.1. Ellipsometric evaluation

Figure 3 shows the maps of the parameters $\Delta$, $\Psi$, and intensity (right) for (A) clean silicon surface, (B) untreated sample and (C)–(F) plasma-treatment at atmospheric pressure in an argon plasma. Treatment times: (C) 60 s, (D) 120 s, (E) 240 s, (F) 600 s. The treatment was performed at an electrical power of 6.05 W.

Figure 3. 2D maps of ellipsometric parameters $\Delta$ (left), $\Psi$ (middle), and intensity (right) for (A) clean silicon surface, (B) untreated sample and (C)–(F) plasma-treatment at atmospheric pressure in an argon plasma. Treatment times: (C) 60 s, (D) 120 s, (E) 240 s, (F) 600 s. The treatment was performed at an electrical power of 6.05 W.
treatment duration. The composition of such residues with respect of the initial fingerprint is discussed in section 3.2.

A purely qualitative observation is only poorly suited for comparing the effectiveness of different treatment parameters. Therefore, figure 4 shows the results of the histogram analysis for the low plasma power (A)–(B) and the high plasma power (C–D) for both ellipsometric parameters Δ and Ψ. It can be seen that in the course of cleaning, the particular histograms of the samples change and ideally, the histogram of the plasma-treated sample should match that of the clean reference surface. Even at the low power level it can be seen that the plasma treatment causes a shift of the histograms for the ellipsometric parameter Δ. The difference of the mean values between the clean reference surface and the untreated fingerprint is 5.43°. After a treatment time of 600 s this difference is reduced to 1.58°. Also the variance could be reduced significantly close to the variance of the clean surface up to the treatment duration of 600 s. When the ellipsometric parameter Ψ is displayed, only a very slight shift of the mean value towards the histogram of the clean silicon surface occurs after 600 s.

For a quantitative comparison, this is also visualized by figure 5 where the particular mean values \( \mu \) and variance \( \sigma^2 \) of the histograms are plotted as a function of the plasma treatment duration for both power levels. It turns out that for Δ, the highest deviation in mean is found between the clean silicon surface and the untreated fingerprint. With increasing plasma treatment duration, the mean value approaches the one of the clean surface. This behavior shows the cleaning effect of the argon plasma. It can further be stated that for the higher applied plasma power, such cleaning occurs earlier or faster. This means that for the considered plasma powers, the higher value features a higher cleaning efficiency. However, for Ψ, the mean value at the beginning of the plasma treatment even deviates from the mean value of the clean reference surface, contrary to expectation, from where the initial value is approximately reached after 600 s. Merely a reduction in variance is clearly observed for both ellipsometric parameters.

The mean values \( \mu \) do not align apparently with the peaks of the histograms shown in figure 4. This deviation results from the fact that small and high Δ (less than 172° and higher than 180°) and Ψ (less than 29.75° and higher than 31°) values with a small relative frequency are not shown in figure 4, but are included in the calculation of the mean values. For example, the maximum relative frequency of these non-displayed values of Δ is 0.0968% for the untreated fingerprint and 0.00509% after a plasma treatment time of 600 s. Additionally it can be seen in the data that the number of non-displayed values of measurement is decreased with plasma treatment time because of the cleaning effect. However, the small non-displayed values are attributed to the surface

![Figure 4. Histogram plots of a fingerprint treated in an argon plasma discharge by (A), (B) 4.05 W and (C), (D) 6.05 W. Due to the larger range displayed for Δ, only every tenth measuring point is shown for reasons of clarity.](image-url)
contamination. For this reason, the deviation between the calculated mean value and the peak position of the histogram is decreased by the plasma treatment. For the clean reference, the mean value and the peak position of the histogram match very closely.

It should be mentioned that the Histograms of $\Psi$ for untreated samples obviously vary in shape, which is due to the fact that these are two different fingerprints with individual characteristics. However, this circumstance does not seem to have much influence on the final result after a treatment time of 600 s, as can be seen when comparing the respective histograms in figures 4(B), (D).

3.2. Chemical composition of fingerprints and thickness

Figure 6 shows the results of the XPS analysis, focusing on the detection of components of human sweat—organic carbon, oxygen compounds, and sodium chloride—and the constituents of the clean (but oxidized) silicon wafer, i.e. silicon and silicon dioxide.

As expected, the highest proportion of organic substances is found in the untreated fingerprint, at about 60%, while the combined proportion of silicon and silicon dioxide is about 37%. The proportion of sodium as detected by the Na1s peak signal is 3% and can be attributed to sodium chloride (NaCl). Only sodium was detectable here, since chlorine often has a much smaller effective cross-section than sodium in XPS analysis according to Beard [29] and thus the signals intensity was too low for our XPS instrument. With increasing duration of the plasma treatment the percentage of organic substances decreases until it reaches a minimum of 20% at an average at a treatment time of 600 s. The proportion of silicon and silicon dioxide increases up to an average portion of 80%, which is due to an adequate cleaning by the applied plasma. Such cleaning preferentially occurs for carbonaceous compounds. In contrast, the relative proportion of NaCl increases with increasing plasma treatment duration, which indicates that the plasma-induced removal of this substance is not possible. The comparison between the sample treated for 600 s and the clean silicon area (A) confirms this observation, since no NaCl could be detected on the clean silicon area. Merely organic compounds were detected on the clean reference surface with a content of about 10%. This contamination is a common and well-known effect and is due to the adsorption of hydrocarbons from the ambient air [30]. Furthermore we determined the peak-to-valley roughness $R_{pv}$ over three friction ridge of the fingerprint before and after plasma treatment using a laser scanning microscope (Zeiss, model LSM 700) and the evaluation software ConfoMap® (Digital Surf) according to DIN ISO 25178. When measured over one papillary ridge of the fingerprint, $R_{pv}$ indicates a good estimate of the maximum film thickness. After a treatment time of 60 s, the maximum layer thickness decreases to about 63% and after 600 s to about 50%. These percentages correlate quite well with the fingerprint components of the XPS measurement, see figure 6.

4. Discussion

According to Belkind et al [31], plasma cleaning of surfaces is due to a combination of chemical and physical cleaning effects, depending on the process gas used. In the inert gas argon, the physical cleaning effect of sputtering predominates, since there are almost no chemically reactive species in the plasma [19]. This non-selective cleaning method means that layer removal is highly dependent on the type of contamination. Although all atoms can be sputtered, the rate of ablation is highly dependent on the type of atoms bombarded. The lattice energy of NaCl for example with a value of 786 kJ mol$^{-1}$ [32] is nearly two times higher than the binding energy of hydrocarbons with 348 kJ mol$^{-1}$ for a C–C bond [33]. Since both the ellipsometric measurements and the
XPS measurements showed that not all substances can be removed completely, it might be useful to apply the chemical cleaning effect by etching, with a thermal component and a radiation component in addition, in order to obtain even better results. This could be achieved, for example, by adding oxygen as a process gas in the plasma and by applying a higher plasma power. The thermal cleaning component is likely to be rather low in the experiments performed, since the temperature in the plasma did not rise above $29^\circ$C even at the high power. In the work of Helmke et al\[5\], the use of an air plasma for cleaning organically contaminated surfaces led to significantly better results. However, considerably higher gas temperatures in the range of $154^\circ$C–$209^\circ$C were achieved there compared to the plasma gas temperature of $27.4^\circ$C–$29.0^\circ$C in the present work (see table 1). Therefore, the thermal cleaning component is significantly higher. On the other hand, the surface contamination treated in the cited work consisted of an oil without further additives, while a fingerprint is a much more diverse mixture. In the maps of the ellipsometric measurements, the degree of cleaning can be clearly observed, where the ellipsometric parameter $\Delta$ is more sensitive here. This deviation with respect to $\Delta$ is due to the basic differences between the signals of $\Delta$ and $\Psi$: $\Delta$ follows from the phase shift of the polarized light components. This value is significantly altered by surface-adherent contaminants with indices of refraction that differ from the one of the substrate material. The change in $\Delta$ is also equivalent to the thickness of the contaminant layer. A change in coverage by the fingerprint thus consequently results in a change in $\Delta$. In contrast, $\Psi$ is given by the inverse tangent of the ratio of the reflectance for perpendicular and parallely polarized light. Thus, should components of the impurities have high absorption, this would be shown particularly in the change of $\Psi$. The fingerprint contamination obviously affects $\Psi$ in the first step, as can be seen in figures 4(B), (D) from the shift of the histograms away from the clean reference. However, histogram analysis shows that as plasma treatment progresses, contrary to $\Delta$, no significant change in the mean of $\Psi$ is induced, although the layer of organic components is shown to be ablated. This fact leads to the assumption that $\Psi$ is mainly determined by those substances which cannot be removed by the plasma treatment, such as NaCl and organic oxygen compounds. It should be noted that the histogram analysis is a very sensitive tool for the detection of contamination on silicon surfaces, since the parameters of the histograms show significant differences, although the maps visually appear to be already cleaned to a large extent. According to XPS analysis, the plasma treatment reduced the amount of carbon-based organic substances by 65% on average. However, the NaCl from human sweat and the organic oxygen compounds were detectable on the surface even after 600 s. In combination with the measurement data of the XPS analysis and the film thickness measurement with the LSM, an estimate of the
film thickness can be made based on $\Delta$ in figure 7 due to the theoretically proportional dependence of $\Delta$ on film thickness. It can be seen that all three measurement methods show a significant trend towards decreasing film thickness. Especially the data from the XPS analysis and the LSM measurement are quite similar.

5. Conclusion

Imaging ellipsometry is adequately suited for a semi-quantitative evaluation of the cleaning degree of plasma-treated surfaces, or other cleaning processes, as it offers the advantage of a contactless measurement. The degree of cleaning can be compared by histogram analysis via mean and variance. It is also possible to approximate the cleaning effect by an estimation of the remaining amount of contamination trough the mean value in $\Delta$, which depends on the film thickness. The histograms of the treated samples should ideally approximate the histogram of the reference surface. Unfortunately, with the parameters selected in this work, plasma treatment can apparently only remove organic carbon compounds of the fingerprint, since even after the maximum treatment time, visible residues such as NaCl are still present. However, the visibility of the fingerprint can be notably reduced by using an argon plasma, with a higher power being advantageous. For a complete cleaning, the largest adjustment factor in future experiments will be the plasma parameters and the process gas. Optionally, a combination of plasma cleaning and conventional cleaning methods such as ultrasonic baths may be useful. In any case, the suggested approach of ellipsometric evaluation represents a useful tool with a number of potential applications in modern surface finishing and inspection.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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