European Polymer Journal 59 (2014) 1–7

Contents lists available at ScienceDirect

European Polymer Journal

journal homepage: www.elsevier.com/locate/europolj

Evaluation of chemical kinetics in positive photoresists using laser desorption ionization

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ARTICLE INFO

Article history:
Received 8 May 2014
Received in revised form 26 June 2014
Accepted 1 July 2014
Available online 17 July 2014

Keywords:
Photoresist
Mass spectrometry
Time-of-flight
Dill C parameter

ABSTRACT

Positive photoresists are photosensitive materials widely used in lithographic processes in microelectronics and optics for component relief manufacturing. When exposed to ultraviolet radiation, chemical reactions are induced that modify their physical–chemical properties. This work describes a novel technique to determine the kinetic rates of molecular structure changes of the positive AZ series photoresists after exposure to mercury arc lamp radiation. These positive photoresists consist of a photoactive compound (PAC), known as diazonaphthoquinone (DNQ), and a matrix material, which is a thick base resin. This positive AZ series photoresist was chosen because its technical information is well known while presenting potential for many applications. In the present work, we investigate these processes using laser desorption ionization (LDI) by a pulsed ultraviolet laser coupled to a high resolution time-of-flight mass spectrometer (ToF-MS). The LDI-ToF mass spectra present different relative intensities for some of the characteristic negative molecular ions of the positive photoresist after different exposure energies to the mercury arc lamp radiation. For measurement of the chemical kinetic mechanism, LDI-ToF mass spectrometry was used for the first time to obtain the fractional decay rate of the DNQ per unit of exposure radiation intensity. These results provide a novel use of LDI-ToF-MS to study the chemical kinetics of photosensitive materials.

1. Introduction

A positive photoresist is a photosensitive material which, upon exposure to ultraviolet radiation, undergoes chemical reactions that increase its solubility in alkaline solutions. The radiation absorption properties of the positive photoresist will influence resolution and its process capabilities. The knowledge of the rate (chemical kinetics) at which these chemical reactions occur permits one to determine the possible technological applications of a specific positive photoresist. After exposure to ultraviolet radiation, a positive photoresist is converted into a relief pattern through a development process, the exposed regions being removed much faster than the unexposed regions. These materials have been widely used in photolithographic processes to manufacture integrated circuits in microelectronics [1–3]. Positive photoresists have also been applied to processes for information storage [4], recording of Bragg gratings in optical fibers, holography [5–9] and holographic memories [10]. The optical absorption mechanism at the microscopic level of the positive photoresist is

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http://dx.doi.org/10.1016/j.eurpolymj.2014.07.005
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described in terms of three parameters known the Dill ABC parameters. The A parameter is an optical absorption dependent term, the B parameter is an optical absorption independent term and the C parameter provides the optical sensitivity, or the chemical kinetics, of the positive photoresist. These parameters have been traditionally and conveniently determined employing the technique of actinic transmission measurement. However, the actinic transmission technique is limited by positive photoresists that absorb radiation over 300 nm wavelength [12].

In previous work, we have employed the Photon Stimulated Ion Desorption coupled to Time-of-Flight Mass Spectrometry (PSID-ToF-MS) technique to identify molecular structure changes in unexposed AZ-1518 positive photoresist, and to those previously exposed to mercury arc lamp radiation [17,18].

In this work, we employed a high resolution Laser Desorption Ionization Time-of-Flight Mass Spectrometry (LDI-ToF-MS) technique to study, not only the molecular structural changes due to exposure to mercury arc lamp radiation, but also the AZ-1518 photoresist chemical kinetics, defined by its Dill C parameter. It is well known that AZ-1518 photoresist has high absorption in the wavelength range of 310 nm to 440 nm. In the mentioned range of wavelengths there are three major emissions of the mercury arc lamp at 365 nm, 405 nm and 436 nm [12]. In the present experiments, no attempt was made to filter the radiation source so as to replicate what is currently observed in practice. The results were obtained with an accuracy that will allow studies of optical properties and chemical kinetics by using the LDI-ToF technique. This novel technique should be able to be applied to other photoresists, especially those chemically amplified which are difficult to measure employing the actinic transmission methodology, for example those that absorb at 248 or 193 nm [13]. Singly charged negative ion mass spectra for different exposure energies to a typical mercury arc lamp, in the reflectron mode, were obtained for the AZ-1518 photoresist. Changes in the relative quantities of molecular negative ions were monitored as a function of exposure energy. From these data it was possible to obtain the AZ-1518 photoresist Dill C parameter.

2. Theoretical model

The positive photoresist has three main components: a base resin, a photoactive compound (PAC) and a solvent. In the positive photoresist, the PAC acts as an inhibitor before the exposure, decreasing the dissolution rate of the resin in alkaline solutions. The PAC typically represents about 30% of a dried photoresist film [11]. After exposure, photochemical reactions modify the inhibitor facilitating dissolution of the exposed photoresist in alkaline solutions. After exposure of the photoresist to ultraviolet radiation, its absorption coefficient α decreases as a result of decreasing the concentration of the photoactive compound. This can be described in terms of the relative concentration $M(z,t)$ of the remaining photoactive compound, at any depth z and exposure time t, by comparing the photoactive compound concentration before and after exposure. If little incident radiation is scattered in the photoresist, the absorption constant is

$$\alpha = A\,M(z,t) + B \tag{1}$$

where, for a certain wavelength, the A parameter describes the absorption by the photoactive compound and B parameter describes the absorption by the other photoresist components [11]. The rate of relative concentration change of the photoactive compound is dependent on the local optical intensity $I(z,t)$, the relative concentration $M(z,t)$ and a measurable optical sensitivity term C, as given by

$$\frac{\partial M}{\partial t} = -I(z,t)M(z,t)C \tag{2}$$

In Eqs. (1) and (2), A, B and C depend upon the photoresist type and exposure wavelength [11]. These constants are referred to as the Dill ABC parameters. The methodology described herein permits determination of the Dill C rate constant by an alternative method with a good degree of correlation and precision using LDI coupled with ToF-MS. In the case where the photoresist is a thin homogeneous film, the intensity of the ultraviolet radiation is not dependent on the exposure time, the solution of Eq. (2) is

$$M(t) = e^{-CIt} \tag{3}$$

or

$$M(E) = e^{-CE} \tag{4}$$

where $It = E$ is the exposure energy E per detector area unit. Therefore, the changes in the relative concentration of the photoactive compound should decrease exponentially as a function of the exposure energy. On a microscopic level, photons are absorbed by an atom or molecule causing an outer electron to be promoted to a higher energy level. This phenomenon is especially important for the photoactive compound since it is the absorption of ultraviolet radiation which leads to the chemical conversion of M to P [12].

$$M(t) \longrightarrow P(t) \tag{5}$$

where the relative concentration of the exposure products P is generated by the reaction of M with ultraviolet radiation. The total amount of components in the photoresist should be conserved and therefore

$$\frac{dM}{dt} = -\frac{dP}{dt} \tag{6}$$

or

$$P(E) = 1 - e^{-CE} \tag{7}$$

Thus, the changes in exposure products $P(E)$ should increase as a function of the exposure energy E. Eqs. (4) and (7) provide an important description of the chemical kinetic mechanisms of photoresists.

In some of the most popular positive photoresists, the photoactive compounds are diazonaphthoquinones (DNQ), as is the case of the AZ-1518 photoresist, whose molecular structure is shown in Fig. 1(a). The matrix material of these photoresists is a thick resin called Novolac. Novolac is a methacrylate-based polymer with an epoxy group, and is shown in Fig. 1(b). For the deposition of

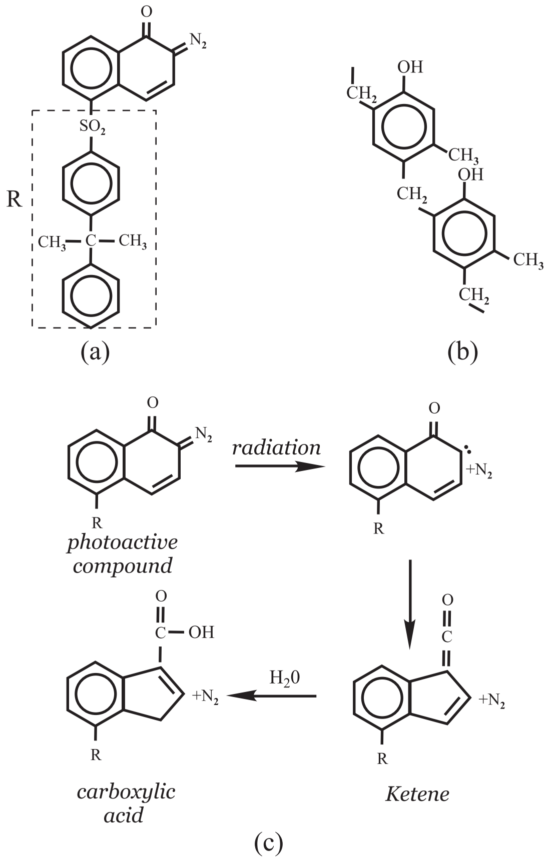


Fig. 1. Molecular structure: (a) 1-naphthoquinone 2-diazo 5-sulfonate (diazonaphthoquinone) (PAC), (b) molecular structure of Novolac and (c) subsequent photolysis of the DNQ due to exposure to a mercury arc lamp radiation.

photoresist on substrates, solvents are added to the polymer to adjust viscosity. The solvents used in positive photoresists are typically aromatic compounds, such as xylene and various acetate esters.

After exposure to ultraviolet radiation, the volatile diazo group present in the PAC is extruded as molecular nitrogen, which results in the formation of an unstable carbene intermediate. The carbene undergoes a Wolff rearrangement which results in the formation of a stable ketene intermediate [14] and is shown in Fig. 1(c). In the absence of water, the reaction stops at this point. In the presence of water, hydrolysis occurs in which the ketene is converted to a carboxylic acid. Due to the acidic nature of the final carboxylic acid, the developed resin matrix dissolves in basic solutions. From the process described above, one can conclude that for the photoresists that have the DNQ as the photoactive compound, the ketene and/or the carboxylic acid are products from exposure to ultraviolet radiation and water. The advantage of the DNQ photoresist is that the unexposed areas remain essentially unchanged in the presence of the developer [15] and, as the Novolac is a long chain polymer, it is very resistant to alkaline attack. Thus, the unexposed regions of the photoresist may be used as a mask towards chemical remove [16].

3. Experimental

The samples were prepared with AZ-1518 photoresists diluted 1 : 25 in AZ thinner (2-methoxy-1-methylethylacetate) solvent (Hoechst Celanese Corporation). Approximately 0.5 μL of the solution was then spotted into several sample wells of a previously cleaned stainless steel sample plate and baked at 90 °C for 20 min. None of the samples were spin coated. After complete drying, the samples were exposed to a mercury arc lamp (Newport Oriel Product Line, Model 6285) with 7.5 mW/cm^2 exposure intensity, in 15.0 mJ/cm^2 up to 120.0 mJ/cm^2 exposure energy per area range. Exposure intensities were measured using a calibrated detector with linear spectral response in the *UV* broad band (model Newport 818-UV) and an optical power meter tuned at $\lambda = 365$ nm (model 1830-C Newport). On the same metallic sample plate, spotted in nearby wells, calibrant samples consisting of a solution of $AgNO_3$ and fullerene (C_{60}), both 1 : 50 diluted in HPLC grade acetonitrile were used to calibrate the mass spectrometry system. After complete drying of the calibrant samples, the metallic plate was placed in the mass spectrometer.

Laser desorption ionization experiments were performed using a pulsed nitrogen ultraviolet laser ($\lambda = 337$ nm) of a commercial AXIMA Performance Matrix Assisted Laser Desorption Ionization Time of Flight (MALDI-ToF) from Shimadzu Biotech. Ion desorption in positive photoresists may occur by two different processes: direct or indirect. As the molecular compounds, DNQ, Novolac, ketene and carboxylic acid contain aromatic rings, they can be desorbed via the LDI technique directly with pulsed nitrogen laser photons. On the other hand, an indirect process of ion desorption may also occur [19]. In this case, the desorption of the molecular compounds of the positive photoresist is assisted by any molecule that absorbs ultraviolet radiation from the nitrogen laser. These interactions may also occur in the hot expanding plume. DNQ is a low molecular weight compound. The use of standard MALDI is precluded in this case because most matrices produce abundant low mass molecular ions [20], which in turn would hamper the identification of the AZ-1518 low mass molecular ion that correlate with ultraviolet radiation exposure. The DNQ molecule, as will be shown, has a high photoabsorption coefficient at the laser wavelength of 337 nm, and therefore, we carried out our studies in a straightforward laser desorption scheme without any matrix.

The experimental setup includes an automatic sample manipulator and a time-of-flight mass spectrometer (ToF-MS), which is operated in a high vacuum chamber with a base pressure of about 3.0×10^{-7} mbar. The time-of-flight mass spectrometer consists basically of an electrostatic ion extraction system, a collimating electrostatic lens, a drift tube, an electrostatic mirror and a pair of microchannel plate (MCP) detectors, disposed in a chevron configuration (reflectron detector). MCP chevron configuration consists of two microchannel plates with angled channels rotated 90° from each other producing a (v-like) shape [21]. After extraction, molecular ions travel through the flight tube until reaching the MCP. We performed several experiments trying to obtain positive ion mass spectra from our samples, with no success. Probably this is due to the high electronic affinity of the molecules of this kind of sample and the relative high yield of the photoelectrons, which would rearrange with fragments of these molecules in the hot expanding plume [22]. The photoelectrons may originate from the surface of the metal plate, or from the ionization of desorbed molecules [23]. As such, the instrument was operated in a negative ion reflectron mode. Normally, deprotonated negative molecular ions are the most abundant, but under laser desorption conditions, ion–molecule reactions can sometimes lead to addition of molecular anions to the parent molecules of the sample [24]. In the negative ion reflectron mode, the high voltage electrical field reflectron (slightly angled) decelerates the ions so that they are reflected back down the flight tube towards the reflectron detector. In this mode, the mass resolution is $R = \frac{m}{\Delta m} \geqslant 2000$ for $m \leqslant 500$ Da. Laser energy was typically 196 μJ/pulse. Each spot was analyzed using a random raster of 160 profiles and each profile contained data from 20 laser pulses. In order to evaluate the mechanisms of interaction by the mercury arc lamp (365, 405 and 436 nm) and by the pulsed nitrogen ultraviolet laser ($\lambda = 337$ nm), AZ-1518 positive photoresist optical absorption spectrum was obtained using a Shimadzu spectrophotometer (Model UV-1800). For this measurement a thin photoresist film, about 1 μm thick, was spin coated on a quartz substrate and baked at 90 °C for 20 min. The UV–vis spectrum was then acquired scanning from 330 to 450 nm.

4. Results and discussion

The proposal of using laser desorption in order to measure photodecomposition steps, in principle, has one major difficulty: distinguishing the effects of the previous mercury arc lamp radiation exposure from the effects caused by the laser desorption itself. Fig. 2 shows the optical absorption spectrum of the AZ-1518 photoresist in the visible and ultraviolet regions. This spectrum shows that light absorption by the photoresist at the wavelength $\lambda = 337$ nm, corresponding to the pulsed nitrogen laser radiation, is actually higher than the absorption at wavelengths $\lambda = 365, 405$ and 436 nm, corresponding to the mercury lamp radiation spectrum. One could expect photochemical decomposition as a function of absorbed laser energy exposure, and we therefore conducted repeated tests to verify whether this would be the case. All results in this regard were negative, which we interpreted as follows. The pulsed nitrogen laser radiation intensity ($\sim 10^6$ W/cm^2) is much higher than the mercury arc lamp radiation intensity ($\sim 10^{-2}$ W/cm^2). However, the laser pulses are of extremely short duration (~ 0.2 ns). This, combined with the optical absorption difference observed in Fig. 2, we conclude that the superficial area which is exposed to each laser shot is completely removed from the sample into the hot expanding plume, leaving the material underneath intact. The laser, therefore, does not contribute to the photochemical decomposition of the material that remains. Another observation which supports this contention is that several mass peaks are only observed if the sample was pre-exposed to the mercury arc lamp radiation, which leads us to conclude that the transitions responsible for the optical absorption at $\lambda = 337$ nm do not contribute appreciably to the formation of those peaks. The weak absorption at $\lambda = 365, 405$ and 436 nm and the low intensity of mercury arc lamp radiation only induce the photochemical reaction in the DNQ molecule. On the other hand, the absorption of $\lambda = 337$ nm is strong and the laser radiation intensity is high, which only produces ionic desorption of the photoresist.

Fig. 3 shows LDI-ToF mass spectra, in the 50.0 to 550.0 m/z range, of AZ-1518 photoresist samples, unexposed (a) and previously exposed to the mercury arc lamp radiation energy per area of 105 mJ/cm^2 (b). The calibration was performed using silver nitrate ($AgNO_3$) and fullerene (C_{60}) mass spectra. The calibration peaks were Ag^- (m/z 106.91), Ag_3^- (m/z 320.72) and C_{60}^- (m/z 720.00). The AZ-1518 photoresist consists mainly of the Novolac polymer, with the DNQ being a small fraction of the photoresist

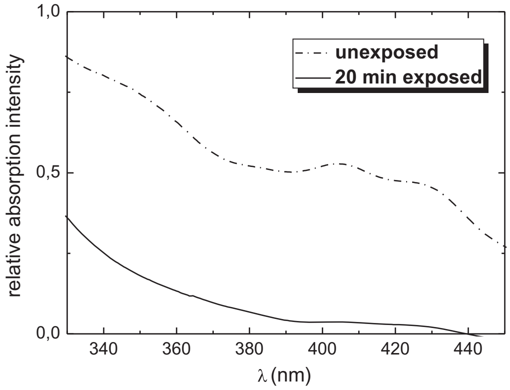
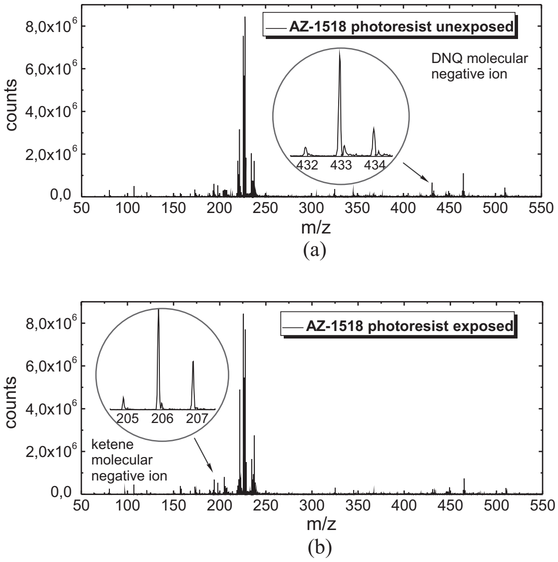


Fig. 2. Optical absorption spectra of AZ-1518 photoresist in the visible and ultraviolet regions.



(a)

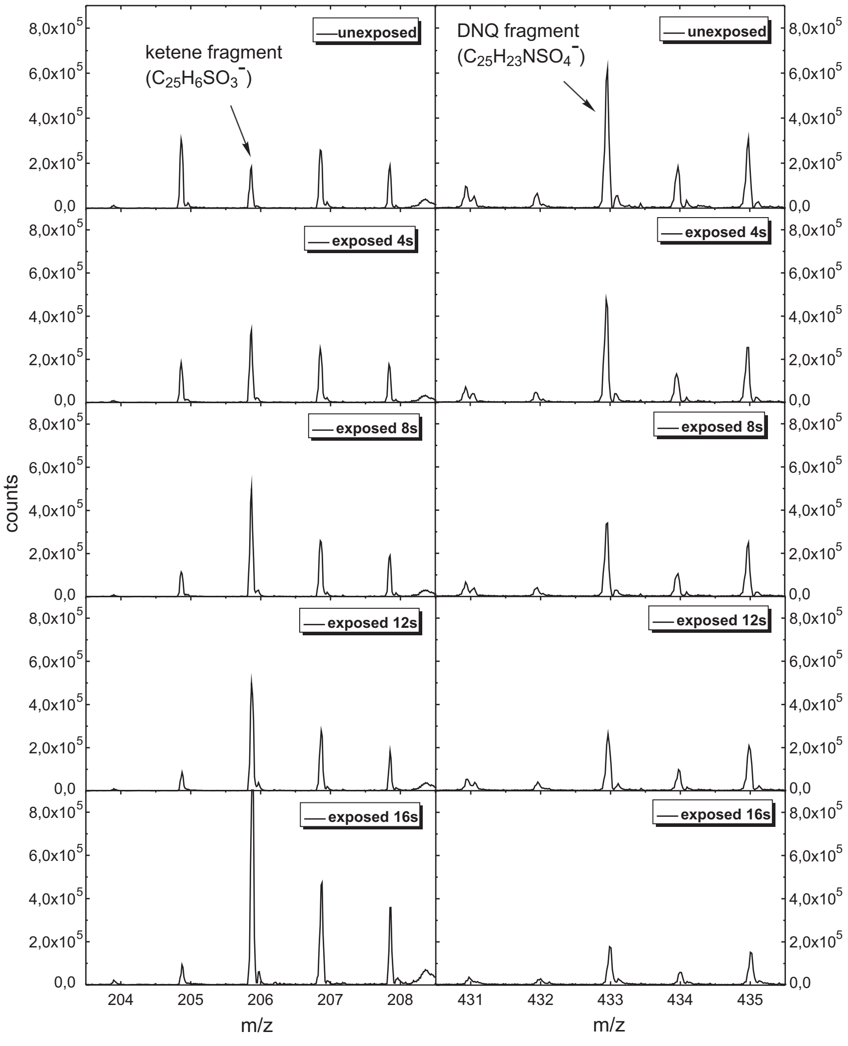
(b)

Fig. 3. LDI-ToF mass spectra, from 50 to 550 m/z range, of AZ-1518 photoresist samples, (a) unexposed and (b) previously exposed to the mercury arc lamp radiation energy per area of the 105 mJ/cm^2.

composition. Thus, the major part of the most intense peaks, in the spectra of Fig. 3, are assigned to the Novolac negative molecular ions. Furthermore, the Novolac is not changed by exposure to the mercury arc lamp radiation, and so peaks corresponding to its negative ions in the mass spectra are not used in the present study of the AZ-1518 photoresist chemical kinetics. As the majority of peaks associated with the DNQ and ketene molecular negative ions are less intense than those peaks associated with the Novolac molecular negative ions, they do not appear clearly in the spectra of Fig. 3. For this reason, peaks corresponding to DNQ and to the ketene molecular negative ion used in the chemical kinetics study of the AZ-1518 photoresist are shown highlighted in this figure.

Fig. 4(a) shows LDI-ToF mass spectra, from 203.5 to 208.5 m/z range, of AZ-1518 photoresist samples, unexposed and exposed to radiation energy per area of 30 mJ/cm^2, 60 mJ/cm^2, 90 mJ/cm^2 and 120 mJ/cm^2 from the mercury arc lamp. Fig. 4(b) shows LDI-ToF mass spectra, from 430.5 to 435.5 m/z range, of AZ-1518 photoresist samples, unexposed and exposed to the mercury arc lamp radiation for the same energy per area. The mass peak m/z 206.00 increases, while the mass peak m/z 433.13 decreases with exposure to the mercury arc lamp radiation. Therefore, the mass peak m/z 206.00 can be assigned to a ketene molecular negative ion($C_{10}H_6SO_3^-$), while the mass peak m/z 433.13 can be assigned to a DNQ molecular negative ion ($C_{25}H_{23}NSO_4^-$). The molecular negative ion of ketene is formed from the ketene molecule with the removal of an ionic positive fragment of the radical R and breakage of a double bond ($C = O$) with the addition of one hydrogen atom. The molecular negative ion of DNQ is formed from the DNQ molecule with replacement of a nitrogen atom by an OH^- ion and two hydrogen atoms.

Although ketene is not the final step of the Wolff rearrangement, the search for its molecular ions in the mass



(a) (b)

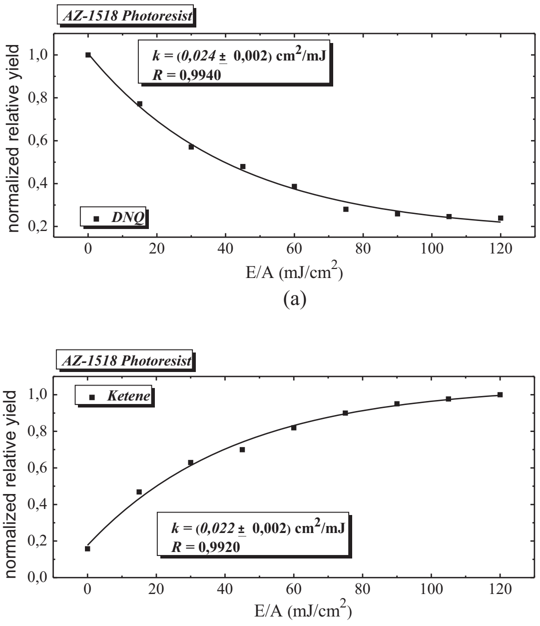
Fig. 4. (a) LDI-ToF mass spectra, from 203.5 to 208.5 m/z range, of AZ-1518 photoresist samples, unexposed and exposed to radiation energy per area of 30 mJ/cm^2, 60 mJ/cm^2, 90 mJ/cm^2 and 120 mJ/cm^2 from the mercury arc lamp. (b) LDI-ToF mass spectra, from 430.5 to 435.5 m/z range, of AZ-1518 photoresist samples, unexposed and exposed to the mercury arc lamp radiation for the same energy per area.

spectra is justified, because the photoresist is analyzed as a solid sample with low amount of water (relative humidity less than 40%), and therefore all ketene molecules are not completely converted to carboxylic acid molecules.

From the mass spectra of several exposed AZ-1518 photoresists the relative yields of m/z 206 and 433 mass peaks were calculated by dividing the area of each peak by the total area of the corresponding spectrum.

The results of our measurements for the AZ-1518 photoresist chemical kinetics are shown in Fig. 5. Fig. 5(a) shows the normalized relative yields as a function of exposure energy per area for DNQ molecular negative ion (m/z 433) and Fig. 5(b) shows the normalized relative yields as a function of exposure energy per area for ketene (m/z 206) molecular negative ion. In the case of the DNQ, the normalized relative yields were calculated dividing the relative yields from the exposed AZ-1518 by the relative yield from the unexposed AZ-1518. In the case of the ketene, the normalized relative yields were calculated dividing the relative yields from the unexposed and exposed AZ-1518 by the relative yields from the most exposed AZ-1518. The fitting of the experimental points with the theoretical model (Eq. (4) for Fig. 5(a) and Eq. (6) for Fig. 5(b)) gives a value for the Dill C parameter, $C = (0.024 \pm 0.002)\ \text{cm}^2/\text{mJ}$ for Fig. 5(a) and $C = (0.022 \pm 0.002)\ \text{cm}^2/\text{mJ}$ for Fig. 5(b).

The ranges of values for the Dill C parameter obtained in this work by molecular negative ions of DNQ (($C = 0.024 \pm 0.002\ \text{cm}^2/\text{mJ}$)) and ketene (($C = 0.022 \pm 0.002\ \text{cm}^2/\text{mJ}$)) are in excellent agreement with the value cited in the literature [12] for a typical positive photoresist thin film ($C = 0.024\ \text{cm}^2/\text{mJ}$ for $\lambda = 436$ nm). This result reinforces the capability of the use of LDI-ToF-MS technique to obtain the Dill C parameter and to study chemical kinetic mechanisms of photosensitive materials.



(a)

(b)

Fig. 5. (a) Normalized relative yields as a function of exposure energy per area for DNQ molecular negative ion (m/z 433) and (b) normalized relative yields as a function of exposure energy per area for ketene molecular negative ion (m/z 206).

5. Conclusions

The LDI-ToF-MS technique was employed in the study of molecular structural changes in AZ-1518 photoresist samples, unexposed and exposed to different radiation energies from a mercury arc lamp. By comparison between the mass spectra of differently exposed photoresists to the mercury arc lamp, it was observed that the mass peak m/z 206.00 increases, while the mass peak m/z 433.13 decreases. Therefore, the mass peak m/z 206.00 was assigned to a ketene molecular negative ion ($C_{10}H_6SO_3^-$), while the mass peak m/z 433.13 was assigned to a DNQ molecular negative ion ($C_{25}H_{23}NSO_4^-$). This is strong evidence for the photochemically induced modification of the positive photoresist. Some other peaks showed practically no changes in their intensities for unexposed as well as for the AZ-1518 photoresist exposed to mercury arc lamp which could be assigned to the Novolac polymer molecular negative ions. In this work the major compounds of the AZ-1518 photoresist and the chemical kinetics of the reaction after radiation exposure were identified and calculated. For the measurement of chemical kinetics, LDI-ToF mass spectrometry was used for the first time to obtain the AZ-1518 photoresist Dill C parameter. The new experimental approach is based on changes in the relative intensities of the mass spectra peaks related to the DNQ and ketene intermediate. The range of values for the Dill C parameter, obtained in this work for the AZ-1518 positive photoresist, is consistent with the range of values found in the literature for positive photoresists of the same series. The relative yields were obtained with an accuracy that would allow studies of the optical properties and chemical kinetics using the LDI-ToF technique. This novel technique should be able to be applied to other photoresists, especially those which are hard to measure employing actinic transmission methodology, *i.e.* those which absorb at 248 or 193 nm. Studies are currently underway to explore this possibility.

Acknowledgments

This work was partially supported by Fundação de Amparo á Pesquisa do Estado de Minas Gerais (FAPEMIG), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES). B.G.A.L. Borges and J.S. Martins would like to thank CAPES for graduate scholarships.

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