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TECHNICAL PAPER

Two photon absorption coefficients and processing parameters for photoresists

Zahidur R. Chowdhury · Robert Fedosejevs

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Abstract The two photon absorption (TPA) process is currently used to write high resolution microstructures for a variety of applications. Key parameters required to predict the final structure formation for this process are experimentally determined and reported in this article for two commercially available resists, Ormocore and SU-8. The measured TPA coefficients measured at 800 nm for Ormocore and SU-8 are 27 ± 6 and 28 ± 6 cm TW^{-1}, respectively. For Ormocore and SU-8 the dose required to write 35 and 50 μm high structures, respectively, were 54 ± 8 and 3.5 ± 0.5 J cm^{-3}, respectively, and the measured contrasts were 15 ± 2 μm per decade J^{-1} cm^3 and 55 ± 8 μm per decade J^{-1} cm^3, respectively.

1 Introduction

Many micromechanical or MEMS devices and photonic crystal structures are three dimensional (3D) microstructures with sub-micrometer resolution. It has recently been demonstrated that such high resolution microstructures can be produced by direct writing using the two photon absorption (TPA) process (Kawata and Sun 2003; Serbin et al. 2003; Seet et al. 2005; Teh et al. 2005; Lee et al. 2004). In this process the absorption in the medium is quadratically dependent on the intensity of the laser beam. Absorbed energy changes the physical or chemical properties of a the photoresists. Thus exposed and unexposed portions of the resist will react differently during further processing depending on the absorbed energy per unit volume. If the absorption and etch rate properties are determined for a given resist it should then be possible to predict the resultant structures for any given irradiation pattern and allow the design of optimum exposure patterns to obtain a given desired structure. The goal of the present investigation is to determine these controlling parameters.

Different research groups have characterized the resist behavior for UV and X-ray lithography utilizing conventional single photon absorption (SPA) processes. Griffiths (2004) examined the effect of photoelectron doses on the dimensions of structures produced in PMMA by deep X-ray lithography. He calculated different limiting factors like minimum producible size, maximum aspect ratio using both analytical and numerical solutions. Yang and Want (2004) simulated fabrication of refractive lens using UV lithography process and SU-8 resist. The behaviour at the interaction of two cylindrical light beams was analyzed to produce such microlens. Reznikova et al. (2005) studied contribution of exposure, baking and resist thickness on final structure formation for SU-8 5 resists. Crack formation due to shrinkage was reported by them. Moreover, Tian et al. (2005) proposed modification of the Fresnel diffraction model for UV proximity lithography for thick resist and for off-axis regions. They reported simulated intensity profiles inside the resist for different resist thickness, gap between mask and the resist and the resist thickness. The success of this model to predict structure formation was demonstrated in that paper using SU-8. The minimum width of a line/wall produced in SU-8 is around 100 nm produced using 100 KeV protons as reported by Stumbo and Wolfe (1993). The highest aspect ratio reported using UV-lithography is 190:1 (1,150 μm:6 μm) by

Z. R. Chowdhury · R. Fedosejevs (✉)
Department of Electrical and Computer Engineering,
University of Alberta, Edmonton, Canada T6G 2V4
e-mail: rfed@ece.ualberta.ca

Yang and Wang (2005). Glycerol was used between the mask and the solid SU-8 resist to compensate effects of air-gap between these layers.

The essential parameters required to characterize the structure writing due to the TPA process are reported here. Two commercially available photoresists, Ormocore and SU-8, were studied. The TPA coefficients for these resists were measured from the variations of transmittances at different intensities following the techniques of Dragomir et al. (2002) and Chróinín et al. (2001). The method was modified for a double layer system since the energy of the light transmitted through the combined resist layer and the substrates was measured in these experiments. The effective TPA cross-section of Ormocore, defined as the product of the ordinary two-photon absorption cross section, δ, and the efficiency of the initiation process, η (< 1), was reported as 3×10^{-55} cm^4 s $photon^{-1}$ by Serbin et al. (2003). Variation of voxel (volume element) height and width with exposure time and power were utilized to determine the parameter.

2 Theoretical background

The linear dependency of transmittance with intensity for modest beam intensities can be used to determine the TPA coeffient of a material. At the same time, height or depth variations of the developed resists for different SPA exposure energies can be utilized to establish the etch rate dependence on absorbed energy.

2.1 TPA coefficient of a single layer material

Variations of transmittance as a function of intensity has been used to find TPA coefficients of bulk materials like commercial fused silica and crystaline quartz (Dragomir et al. 2002; Chróinín et al. 2001). The TPA coefficient of 0211 glass substrate, used in the present experiment, was determined using the same procedure.

The intensity distribution of a beam with spatial and temporal Gaussian profile with a beam radius ω at a particular location along its propagation path can be described as

$$I_i(r,t) = I_{max} \exp\left[-2\left(\frac{r}{\omega}\right)^2\right] \exp\left[-\left(\frac{2t}{\tau_p}\right)^2\right] \tag{1}$$

where I_{max} is the maximum on-axis intensity, τ_p is the pulse width at e^{-1} level. The FWHM pulse width τ is equal to $\tau_p\sqrt{ln2}$. The incident pulse energy, which is obtained by integrating the intensity over space and time can be expressed as,

$$E_i = \frac{\pi\sqrt{\pi}}{4} I_{max}\tau_p\omega^2 \tag{2}$$

Now let's consider the pulse is incident on a medium of thickness Δd and having a linear absorption coefficient, α, TPA coefficient, β, and refractive index, n. At low intensities and/or for a low value of β the transmittance, the ratio between transmitted and incident energy, can be found to be,

$$T \equiv \frac{E_t}{E_i} = T_0\left[1 - \frac{\beta I_{max}(1-R)\{1-\exp(-\alpha\Delta d)\}}{2\sqrt{2}\alpha}\right] \tag{3}$$

where R is the reflectance of the surface of the sample at normal incident and T_0 is the transmittance at zero-intensity defined as:

$$T_0 = (1-R)^2 \exp(-\alpha\Delta d) \tag{4}$$

Here R is given by the usual Fresnel reflections at the entrance and exit surfaces; namely

$$R = \frac{(n-n_0)^2}{(n+n_0)^2} \tag{5}$$

where n and n_0 are the refractive indices of the material and the surroundings.

To measure the TPA coefficient the transmittance through the sample is measured for different intensities as described in references (Dragomir et al. 2002; Chróinín et al. 2001). The TPA coefficient, β, can be easily calculated from the rate of change of transmittance with intensity, dT/dI_0, since,

$$\beta = \frac{2\sqrt{2}\alpha(dT/dI_{max})}{(1-R)[1-\exp(-\alpha\Delta d)]T_0} \tag{6}$$

Moreover, for cases where $\alpha\ \Delta d << 1$ Eq. 6 can be simplified as

$$\beta = \frac{2\sqrt{2}(dT/dI_{max})}{\Delta d(1-R)T_0} \tag{7}$$

The slope should be calculated from the linear region of the transmittance change with peak intensity.

2.2 TPA coefficient of a double layer material

The photoresists used for TPA writing are coated on a Corning 0211 glass substrate. So, the above calculation should be modified for a double layer system. Let us consider a laser beam passing through two layers of different materials A and B as shown in Fig. 1. Pulses with

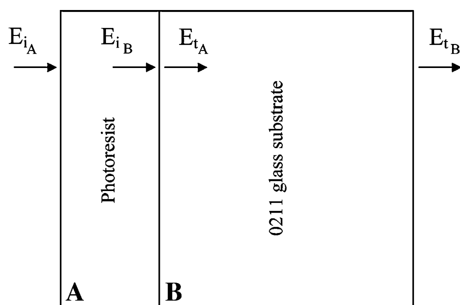

Fig. 1 Geometric layout for TPA coefficient determination in a two-layer system

incident pulse energy, E_{i_A}, leave the system with transmitted energy, E_{t_B}. The ratio between E_{t_B} and E_{i_A} can be represented by Eq. 3 as transmittance through B where one side contacts material A and the other side is in air. The equation can be re-written to represent this case as,

$$T_{B_{A-B-0}} \equiv \frac{E_{t_B}}{E_{i_B}} = T_{0B_{A-B-0}} T_\beta \tag{8}$$

where $R_{A\text{-}B}$ is the reflectance at the surface between A and B and $T_{0B_{A-B-0}}$ and T_β are the zero intensity transmittance and intensity dependent transmittance for the arrangement, respectively. These terms can be defined as,

$$T_{0B_{A-B-0}} = (1 - R_{A-B})(1 - R_{B-0}) \exp(-\alpha_B \Delta d_B) \tag{9}$$

$$T_\beta = 1 - \frac{\beta_B I_{B_{max}} (1 - R_{A-B}) \{1 - \exp(-\alpha_B \Delta d_B)\}}{2\sqrt{2}\alpha_B} \tag{10}$$

where, $R_{B\text{-}0}$ is again the reflectance at the surface between air and B.

The incident intensity on B depends on incident energy E_{i_B}. Replacing $I_{B_{max}}$ by $4E_{i_B}/\left(\pi\sqrt{\pi}\tau_p\omega^2\right)$ in Eq. 8, E_{i_B} can be expressed in terms of a quadratic equation of the form $aE_{i_B}^2 + bE_{i_B} + c = 0$ where,

$$\begin{aligned} c &= E_{t_B} \\ b &= -T_{0_B} \\ a &= \frac{4T_{0_B}\beta_B(1 - R_{A-B})[1 - \exp(-\alpha_B \Delta d_B)]}{2\pi\sqrt{2\pi}\tau_p \alpha_B w^2} \end{aligned} \tag{11}$$

Given that the properties of the medium B (0211 glass substrate) are known then the solution of the quadratic equation can be used to find transmitted energy from A entering the medium B as $E_{t_A} = E_{i_B}(1 - R_{A-B})$. Once incident and transmitted energy of A is known the TPA coefficient can be found using a similar method to that described earlier for a single layer material. We have to keep in mind that in this case, the surrounding material is not only air.

2.3 Etch rate for different absorbed energy

Absorbed energy in a sample due to SPA process can be easily calculated from the SPA coefficient, the intensity of the light source and the exposure time. The absorbed energy can be calculated as a linear function of exposure time because there is no significant change of SPA coefficient of the material over the total exposure time. Thus, the exposure time can be used to control the absorbed energy. Arrays of straight lines with different widths were exposed for different time during the SPA based fabrication process. Final dimensions of the transferred pattern for different exposures were measured using a surface profilometer. The final dimensions are related to the etch rates of the photoresists which should vary with the absorbed energy. Finally, a correlation between absorbed energy and etch rate of the photoresists in the developer is established.

For a light source with a spectral intensity distribution $S(\lambda)$ (per unit wavelength), the incident spectral intensity energy on the resist, $I_0(\lambda)$, is simply

$$I_0(\lambda) = T_{Surface}(\lambda) \times S(\lambda) \tag{12}$$

here, $T_{Surface}$ is the total transmittance through the different surfaces of the exposure system into the photoresist. A material of thickness, Δd, absorbs $E(\lambda)$ amount of energy per unit area per unit wavelength for an incident intensity $I_0(\lambda)$ where

$$E(\lambda) = I_0(\lambda) t_{\exp}[1 - \exp(-\alpha(\lambda)\Delta d)] \tag{13}$$

where $t_{\exp}$ is the exposure time. From the Eq. 13 it is evident that absorbed energy in the resist can be varied linearly by varying the exposure time. $E(\lambda)$ can be also calculated from wavelength dependent absorption in dB/m, $\alpha_{dB}(\lambda)$, using

$$E(\lambda) = I_0(\lambda) t_{\exp}\left(1 - 10^{\frac{\alpha_{dB}(\lambda)\cdot\Delta d}{10}}\right) \tag{14}$$

3 Experimental details

Eight hundred nanometer laser sources are often used for TPA writing in Ormocore and SU-8 due to the fact that the resists have significant absorption around 400 nm but are almost transparent for 800 nm light. An 800 nm femtosecond (fs) laser source was used for the TPA experiment reported have to produce the high peak intensities required for TPA writing. In addition, a Mercury and Xenon lamp source having emmision peaks at 365 and 400 nm was utilized for the SPA experiments.

3.1 Light sources

A 110 fs, 800 nm Ti:Sapphire laser amplifier (Spectra Physics Hurricane) was used in a single shot basis to determine the TPA coefficients of the photoresists. The maximum average output power of the laser system was around 500 mW with a pulse repetition rate of 1 kHz. The experiment was performed on a single shot basis with pulse energies from 100 to 400 μJ.

A UV resist exposure source (manufactured by AB-M Inc.) was used for the SPA experiments. The source is designed for light emission at 365 and 405 nm wavelengths. Mercury and Xenon lamps inside the system produce radiation in several discrete wavelength regions in the UV region. A compact spectrometer (Ocean Optics USB 2000) with 1.34 nm resolution was used to measure the spectrum of the source. Figure 2 shows the spectrum with large peaks at 365, 405 and 437 nm and minor peaks at 550 and 582 nm as reported by other researchers (Reznikova et al. 2005; Yang and Wang 2005). The spectrum was corrected for efficiency of the spectrometer at different wavelengths. The intensity distribution is uniform within 6% over the four square inch exposure area available.

3.2 Sample preparation

Five hundred micrometer thick 0211 glass substrates cleaned with Piranha etch (H_2SO_4:H_2O_2 3:1) were used for both SU-8 and Ormocore sample. The substrate was also dried and baked at 200°C for a minute to drive away the moisture. A 50 µm layer of SU-8 2050 from MicroChem Corporation was coated on the substrate by spinning the photoresist at 3,000 rpm. The substrate was then prebaked at 65°C for 3 min and at 95°C for 6 min according to the manufacturers recommendation. It was then exposed for different time periods using the UV mask aligner as mentioned in Sect. 3.1. Post exposure baking was 1 min at 65°C and 5 min at 95°C. Then it was developed for 6 min in SU-8 developer, Methoxy 2-propanol acetate, rinsed with isopropyl alcohol (IPA) and dried out by N_2 gas flow. SU-8 had poor adhesion to glass, which was solved by adding an extra thin layer of XP Omnicoat between the SU-8 and glass. This additional thin layer was coated at 3,000 rpm. The adhesive layer was hard baked for 1 min at 200°C. The adhesive layer was around 1 µm thick. The adhesion layer remained on the substrate after all the processing.

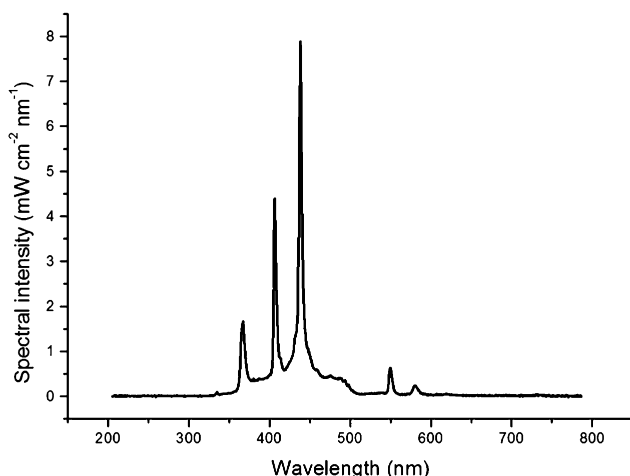

Fig. 2 Incident spectrum of the UV source during SPA for Ormocore

A 35 µm Ormocore layer was coated on the substrate for both SPA and TPA experiments. The resist is for proximity printing as it remains in a form of viscous liquid before the exposure. A 100 µm thick spacer (Mylar) was placed around the boundary leaving the inside region for the photoresist free before placing the mask on top of the sample for SPA experiments.

3.3 Experiments

A 110 fs, 800 nm Ti:Sapphire laser beam was focused using a 75 cm (at 589 nm wavelength) lens to find the TPA coefficient of the resists. Samples were placed around 5 cm away from the true focal plane which was approximately 80 cm for 800 nm light. Detailed beam profiling at different locations was performed before the lens and around the focal region of the lens to determine experimental beam parameters. Knife edge experiments were used to measure the beam radius before the lens where the beam spot was larger while a CCD camera (Cohu C 6612D) along with a beam analysis software (Spirikon) was used to find the beam radius around the focal region of the lens. The measured average beam radius at the focal position of the lens, true focal distance and rate of beam radial convergence after the lens were 54 µm, 80 cm and 4.7 µm/m, respectively. The beam produced an elliptical spot at the surface of the resists. At the position where measurements were made, the beam major and minor radii at the resist surface were (240 ± 10) µm and (205 ± 10) µm on SU-8 resist surface, respectively. The major and minor beam radii at the Ormocore resist surface were (174 ± 10) µm and (120 ± 10) µm, respectively. The difference is due to the use of different measurment positions along the beam path.

The incident intensities on the samples were varied using a half-wave plate and polarizer assembly. Moreover, incident and transmitted energies were calculated using photodiodes calibrated by a Spectra Physics power meter (Model 407 A). Transmittances were calculated from the incident and transmitted energy at different intensities. Other material properties like SPA coefficients and

refractive indices were collected from different sources (Ling et al. 2000; Houbertz et al. 2003; Yang and Want 2004) and were used to find the TPA coefficients as described in Sects. 2.1 and 2.2.

The main objective of the SPA experiment was to expose the photoresist with different exposure times while maintaining other processing parameters constant. For each exposure a series of lines as shown in Fig. 3a with widths varing from 10 to 500 μm and the with lengths of 2,000 μm were exposed simultanously. The width variation ensured several observations for a particular setting. The transmission of the mask for the operating wavelength was measured through a transparent block at the corner of the mask using a separate Monochromator (High Speed Monochromator System, HS-190). Each group of lines was exposed with a different illumination time which was controlled by an overlayed aperture mask.

The surface profile was measured using an Alpha Step 200 contact profilometer. The device can measure profiles with a ±0.25 μm accuracy in height. The resolution of the profilometer along the surface was limited by the dimension of the tip, used to scan the surface. The stylus which can be modeled as a 60° cone rounded to a spherical tip with a 5 μm radius gives a lower limit for the vertical resolution.

For the SU-8 resist a rapid transtion between no structure and structures with full heights on the substrate was observed as the exposure was increased. Similar results were reported for SU-8 5 by Reznikova et al. (2005) and the authors suggested that the shrinkage at the low exposure causing reduced adhesion to the substrate as a possible reason for such a result. A solution to this problem is to fabricate the low energy exposure zone surrounded by fully exposed resists to ensure better adhesion. A selected area of the resist was exposed for a particular exposure time and then an opaque patterned mask covering a partial area at the center of the exposed zone was placed on the resist to overexpose a surrounding anchor region (see Fig. 3b). Then the whole substrate was exposed for a long time (15 s). In this way the regions covered by the opaque objects gave the information about the etch rate for different exposure and remained on the substrate.

Transmission through different layers and absorption in the resists were calculated from material properties at different wavelengths convolved with the irradiation spectrum. The spectrum of the UV light source was calibrated by a power meter for 365 nm. The spectral response for the power meter was convolved with the source spectrum for these calibrations.

4 Results

For the TPA measurements the input and transmitted pulse energy were recorded to calculated the incident peak intensity as well as transmission through the sample. New spots were chosen for every shot. For 0211 glass substrate alone the calculation was relatively easy and the TPA coefficient was calculated from the variation of transmission with intensity. This coefficient of beta was used for the samples where the resists were coated on the glass substrate. Figure 4a, b, c show the variations of transmission for 0211 glass, Ormocore and SU-8 layers, respectively. These figures contain data for one sample. The TPA measurement was repeated three times on each sample and measured on three different samples for each material. The TPA coefficients determined from these measurements are tabulated in Table 1.

There are errors involved with measurements of different experimental parameters like energy, beam spot sizes, pulse widths as shown in Table 2. Considering possible errors for TPA experiments the overall error of β is ±20%. The resultant TPA coefficients for Ormocore and SU-8 are 27 ± 6 and 28 ± 6 cm TW^{-1}, respectively. The peak intensities on the SU-8 resist during the TPA experiments were of the same order of magnitude as reported by

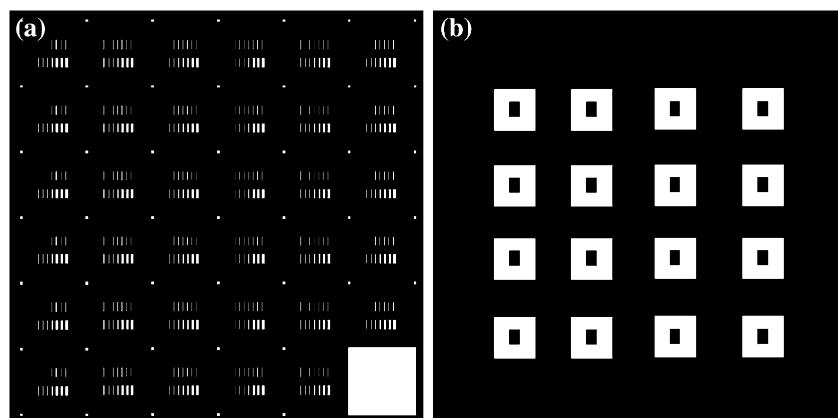

Fig. 3 **a** Mask for SPA experiments for Ormocore showing multiple repeated groups of the *line pattern* used and **b** exposure mask with opaque objects at the *center* of each *square* for SPA experiments of SU-8. The *white areas* in the second figure represents the region which is overexposed to anchore the area under the objects

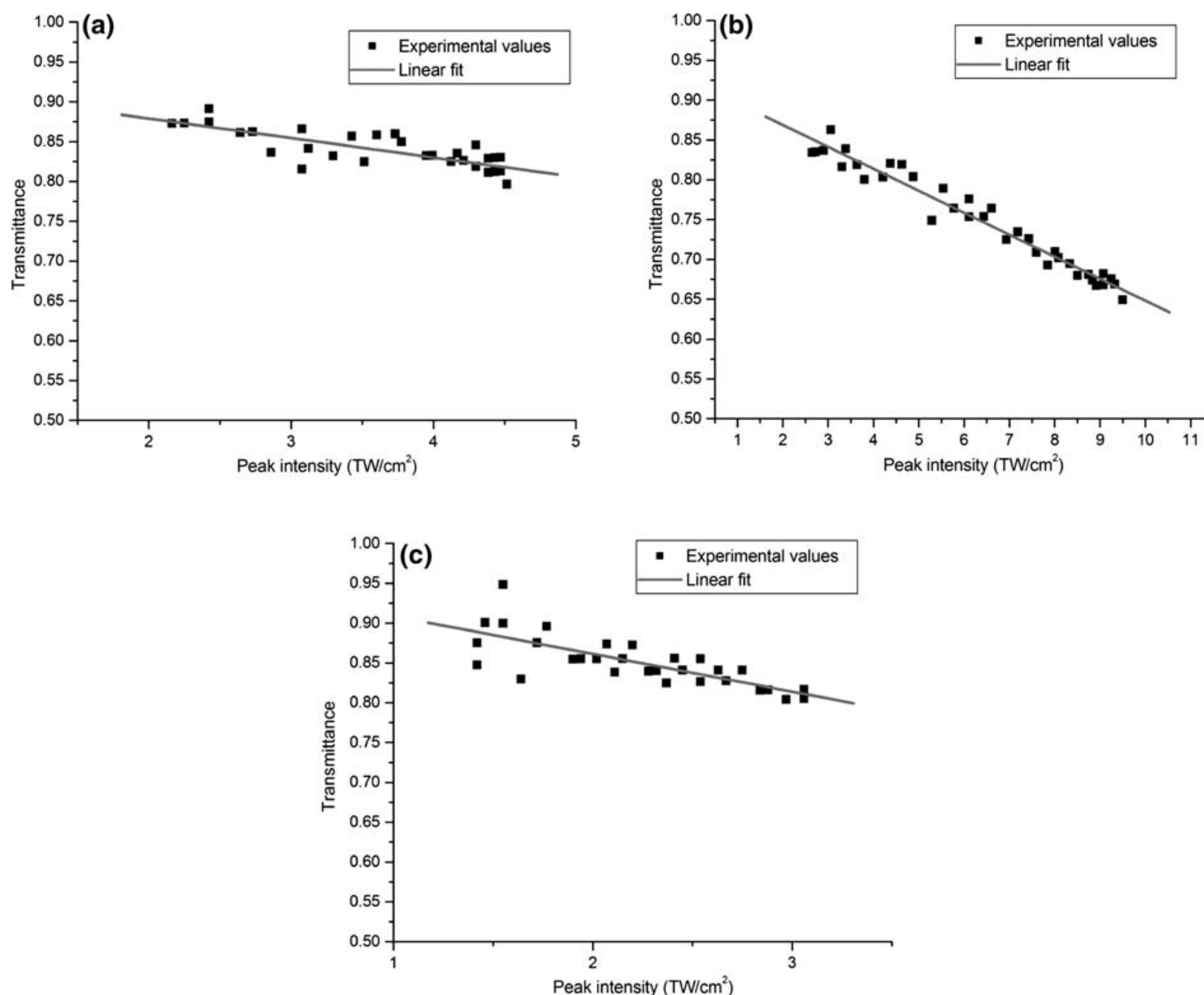

Fig. 4 Variation of overall transmission with peak intensity for **a** 0211 glass substrate, **b** Ormocore coated on the substrate and **c** SU-8 coated on the substrate. The TPA coefficient is determined using the slope along with other material parameters

Witzgall et al. (1998) during the single shot TPA polymerization of SU-8 performed by the researchers. Thus the required intensities for onset of significant TPA observed here are consistent with those reported in the reference (Witzgall et al. 1998).

Table 1 TPA coefficients for different samples of 0211 glass substrate and Ormocore and SU-8 resists

Material	Sample number	β (cm TW^{-1})				
		Observations			Sample	Overall
0211	1	1.5	1.4	1.5	1.5	1.4 ± 0.2
	2	1.3	1.3	1.4	1.4	
	3	1.3	1.5	1.6	1.5	
Ormocore	1	27	27	26	27	27 ± 6
	2	26	27	29	27	
	3	27	26	28	27	
SU-8	1	27	27	27	27	28 ± 6
	2	30	32	25	29	
	3	22	30	31	28	

The absorption in the material was calculated based on the SPA coefficients (Ling et al. 2000; Houbertz et al. 2003) as described in Sect. 2.3. Figure 5a, b show absorbed intensities versus wavelength for the Ormocore and SU-8 resists, respectively. Total absorbed intensities for Ormocore and SU-8 were 2.7 and 3.15 mW cm^{-2}, respectively. The absorbed intensity or the absorbed energy per unit volume per unit time can be easily found by dividing the the total intensity with the thickness of the resist. For a 35 μm Ormocore layer the absorbed energy per unit volume per second is 0.77 J cm^{-3} s^{-1} while that for SU-8 is 0.63 J cm^{-3} s^{-1} for a 50 μm thick layer. Energy per unit volume can be obtained by multiplying by the exposure time. Figure 6 shows the height or depth variation for the resists and corresponding etch rate for the corresponding developing time.

The heights of the structures from the substrate surfaces were measured using the surface profilometer for

Table 2 Error estimates in measuring different parameters for TPA and SPA experiments

Parameter	Unit	% Error	Typical error	Typical values
Refractive index, n	–	–	0.005	1–2
Measured energies, E	μJ	–	10	300
Pulse width, τ_p	fs	–	5	110
Beam radius, ω,	µm	–	10	200
Thickness(resist), Δd	µm	10%	–	50
SPA coefficient, α	m^{-1}	10%	–	10–1,000
Spectral intensity, $S(\lambda)$	mW cm^{-2} nm^{-1}	10%	–	0–10
SPA power meter calibration	–	10%	–	2–3
Exposure time, t_{exp}	s	–	0.0025	0–10
Developing time, t_{dev}	min	–	0.2	6
Etched resist height/ depth	µm	–	0.25	0–50

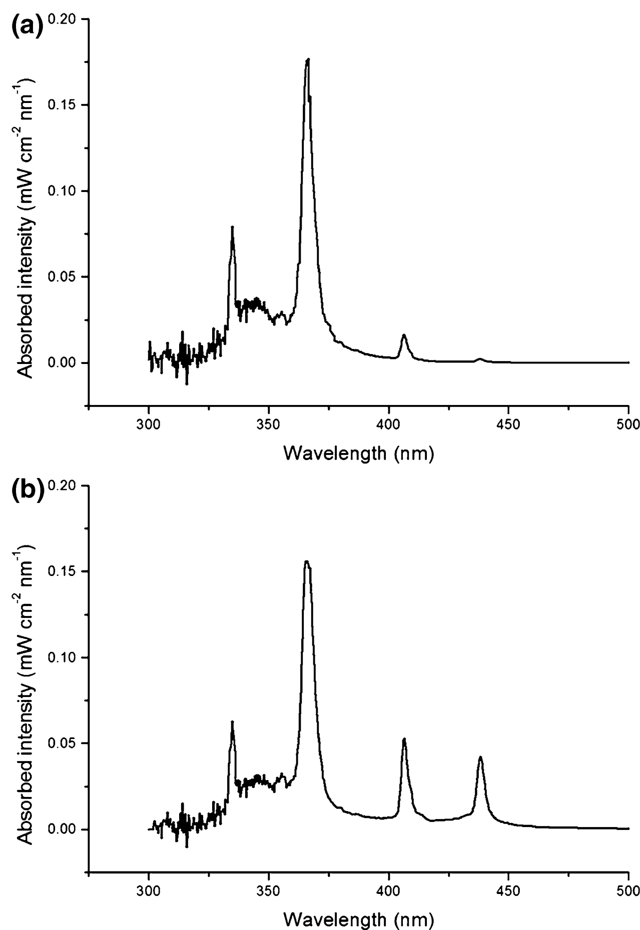

Fig. 5 Absorbed spectral intensity versus wavelength for **a** Ormocore and **b** SU-8

Ormocore. For SU-8 the depth of the exposed resists from the surrounding fully exposed resists were measured. The surrounding structures were to prevent the wash away of the low energy exposure zone due to poor adhesion because of the shrinkage. There were ripples visible for threshold energy exposures for which the etching process just started.

Higher energy exposure produced cracks. The cracking effect diappeared gradually for much higher energy exposure. No such cracks were observed for structures produced in Ormocore.

Sensitivity of a negative photoresist is defined as the minimum aborbed energy required to resist the etching during the developing process. Moreover, the contrast is the slope measured in the transition region of the resist behavior. Ormocore produced a linear transition in bar height versus the logarithm of the absorbed energy as shown in Fig. 6c. For SU-8 the data was more scattered and could be fit with a linear slope either as a function of energy or log of energy. The sensitivity for Ormocore was determined by extrapolating the straight line of Fig. 6c to find the energy to produce a 30 µm high structure. The sensitivity of SU-8 is the minimum energy to produce no etching of the resist. There is a 15% error involved with measured sensitivity and contrast, respectively for some errors mentioned in Table 2. Therefore, the sensitivity and contrast for Ormocore are 54 ± 8 J cm^{-3} and 15 ± 2 µm per decade J^{-1} cm^{3}, respectively. On the other hand, the sensitivity and contrast for SU-8 2050 are 3.5 ± 0.5 J cm^{-3} and 55 ± 8 µm per decade J^{-1} cm^{3}, respectively. Figure 6e, f also presents the calculated etch rate during the developing process for Ormocore and SU-8, respectively. The transition region of structure formation for SU-8 was from 2 to 10 J cm^{-3} as reported by Reznikova et al. (2005) which is comparable with the result presented in this paper. The lower threshold and higher sensitivity may be due to the use of SU-8 2050 which is designed to meet these criterias.

5 Conclusion

Important material properties for the resists used for TPA writing were measured in order to be able to model the process in the future. The TPA coefficients were measured using a 110 fs Ti:Sapphire laser source. The coefficients were determined by measuring the transmittances as a function of incident intensity and other known material properties. A modification of the conventional technique to calculate the coefficients was employed for a double layer system. The TPA coefficients measured by this technique were 27 ± 6 and 28 ± 6 cm TW^{-1} for Ormocore and SU-8, respectively.

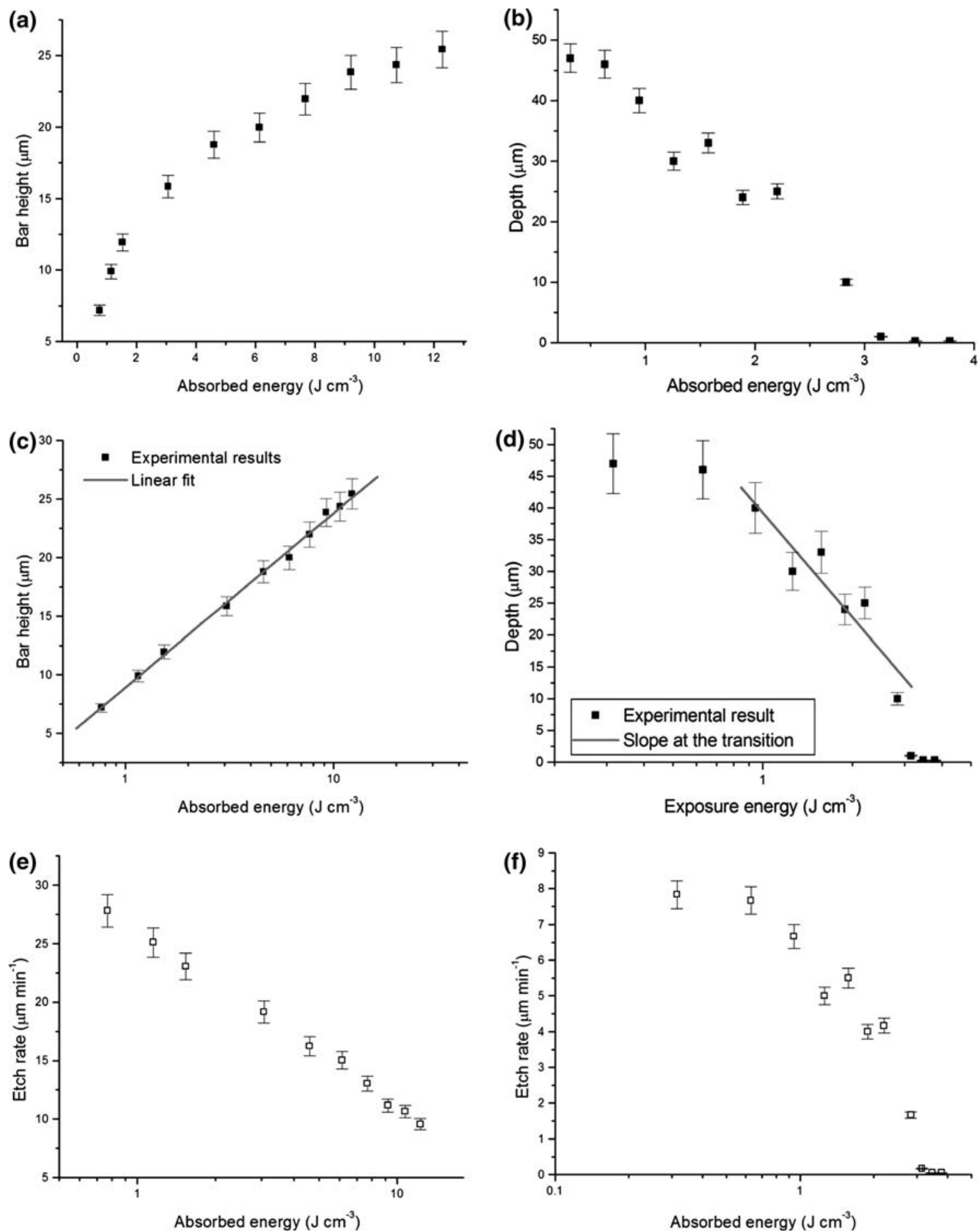

Fig. 6 **a**, **c** Height variation of Ormocore for different absorbed energies, **b**, **d** depth variation for exposed SU-8 for different absorbed energies. Variation of etch rate for **e** Ormocore and **f** SU-8 for these energies

The TPA coefficient measured for 0211 glass was 1.4 ± 0.2 cm TW^{-1}.

The resist etch rate versus absorbed energy for single photon absorption was also measured. For SU-8 shrinkage of the resist for low exposure doses led to a problem which was solved by connecting the exposed area to solid over exposed regions of the resist. The heights or depths were measured and etch rates were measured for the two resists.

It is expected that these parameters can now be used to simulate the behavior of the resists for different two photon

exposures and predict resultant structures for a given exposure geometry.

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