NEW PHOTOIMAGEABLE BONDING ADHESIVES: OPTICAL PROPERTIES AND REAL APPLICATION

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IN COOPERATION WITH MICRO CHEM

In this work, optical properties and real application of new type of photoresist devoted to permanent bonding of silicon and glass microstructures are presented. Two versions of such photoimageable bonding adhesives, solvent and alkaline developable, have been tested and compared with a standard SU-8 photoresist. Planar optical waveguides were fabricated using each photoresist and next used to investigate optical transmittance and autofluorescence of materials. One of the bonding adhesives was next used for the first time to fabricate microfluidic chip for capillary gel electrophoresis. The chip was successfully applied for rapid separation and fluorometric detection of genetic material samples.

Photoimageable bonding adhesives

Photoresists are applied in various microengineering processes, mainly in patterning of masking layers for wet/dry etching of silicon and glass or as matrices for molding PDMS structures. Some photoresists (e.g. SU-8) may be also utilized as an intermediate layers for indirect bonding of micromechanical structures, but such application is not efficient in comparison to direct bonding methods. However, application of photoimageable polymers in permanent sealing of microstructures is highly desired, as it would remarkably simplify fabrication of Micro-Opto-Electro-Mechanical Systems (MOEMS) and lab-on-a-chip (LOC) devices.

New types of negative-tone photoresists dedicated to permanent bonding have been developed recently (PermiNex™ bonding adhesives series, MicroChem Corp). These photoimageable bonding adhesives (PBAs) provide superb adhesion to silicon or glass and low temperature sealing (<200°C) using a single coating process. As they may be potentially applied in MOEMS or LOC solutions utilizing optical detection methods, in this work optical transmittance and autofluorescence intensity of two PBAs: solvent developable PermiNex™ 1000 and alkaline developable PermiNex™ 2000 were investigated, and compared with a standard SU-8 resists (MicroChem Corp). PermiNex™ 2000 has been also utilized for the first time in fabrication of hybrid LOC for capillary gel electrophoresis (CGE), which was next applied for rapid and simultaneous analysis of two DNA samples.

Optical properties

Optical parameters were measured using planar optical waveguides of various lengths (2.5 mm to 20 mm) patterned in layers of each examined photoresist: PermiNex™ 1000, PermiNex™ 2000 and SU-8. Test structures comprised of 2" borosilicate glass wafer, 150-200 µm thick film of photoresist with patterned optical fibre ports and input/output optical fibres (OM2 pigtails: 62.5/125 µm) for supply and collection of optical signal (Fig. 1). Terminals of the fibres were prepared using precision cleaver and glued into ports using optical adhesive.

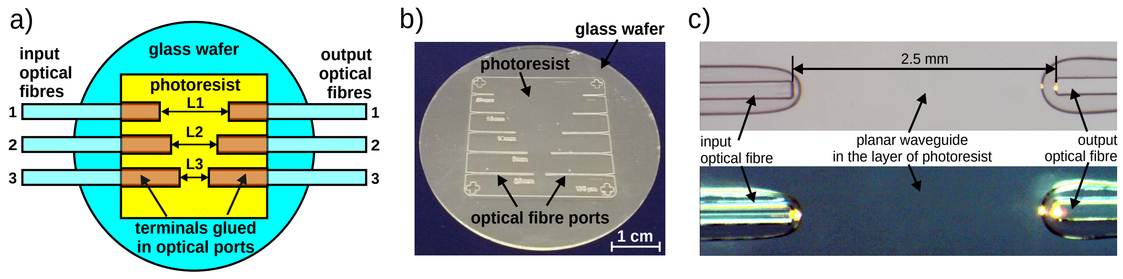


Fig. 1. Test structures with planar optical waveguides made of photoresist: a) top-view scheme (L1-L3 correspond to various length of planar waveguides), b) real-view of the structure fabricated of SU-8, before gluing optical fibres, c) magnified view of 2.5 mm long planar waveguide made of bonding adhesive (PermiNex™ 2000): before (top) and during measurements (bottom)

Optical transmittance

Optical transmittance was measured using setup presented in Figure 2. Input fibres were connected to a source of white light (HL-2000 halogen lamp, Ocean Optics), while the output fibre was connected to miniature VIS-NIR spectrometer (USB 4000, Ocean Optics). Collected data was transferred to personal computer and processed using software for measurement of spectral intensity (Spectra Suite, Ocean Optics). Spectral characteristics of 2.5 mm, 5 mm and 10 mm long planar waveguides were compared for all the photoresist (Fig. 3).

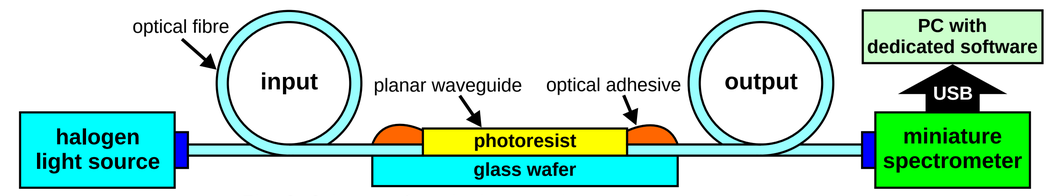


Fig. 2. Scheme of optical transmittance measurement setup

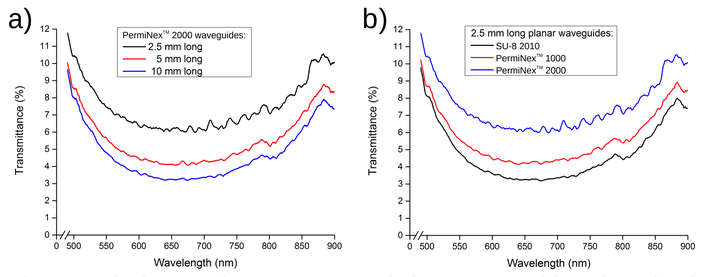


Fig. 3. Optical transmittance spectra of planar waveguides: a) made of PermiNex™ 2000 (various length of optical path), b) made of each tested photoresist (comparison for 2.5 mm long optical path)

Conclusions

- optical transmittance measured for planar waveguides made of both PBA versions is lowest at 650-700 nm and increases towards NIR and UV
- for 2.5 mm long optical path, SU-8 indicates lowest and PermiNex™ 2000 highest values of optical transmittance (3% and 6% at 650 nm, respectively)
- higher transmittance values were obtained for shorter optical paths, as expected

Autofluorescence intensity

Autofluorescence was measured using orthogonal arrangement of excitation light source and output fibre (Fig. 4). Photoresist layer was illuminated from the top by focused laser/LED light beam. Light sources suitable for standard red, green and blue-line fluorophores, commonly applied in fluorometric detection systems, have been used. Scattered light of the source (λ_{EX}) and autofluorescence signal of the material were collected by output fibre and processed in the spectrometer (Fig. 5).

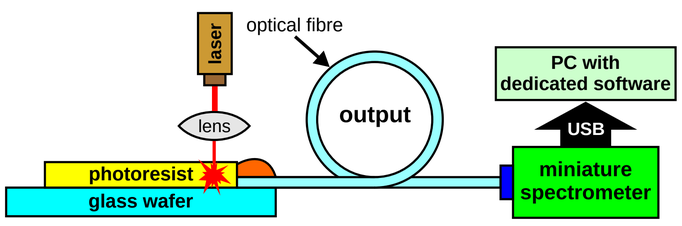


Fig. 4. Scheme of autofluorescence intensity measurement setup

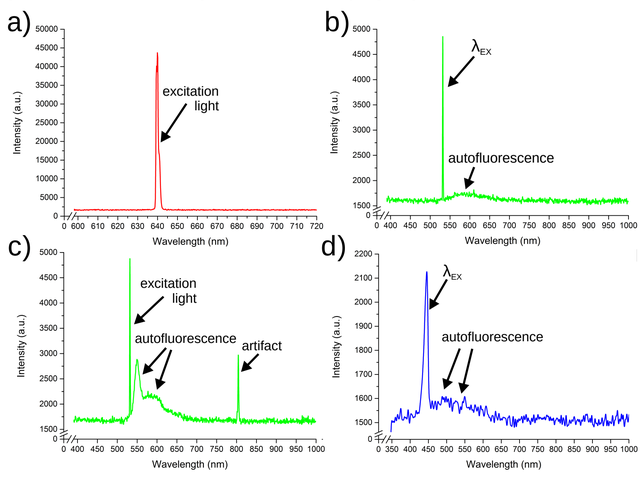


Fig. 5. Autofluorescence intensity spectral characteristics of: a) PermiNex™ 1000 excited with 25 mW red-line laser diode (λ_{EX} = 643 nm), b) SU-8, and c) PermiNex™ 1000 excited with 100 mW green-line laser diode (λ_{EX} = 532 nm), d) PermiNex™ 2000 excited with 50 W blue-line power LED (max. λ_{EX} at 448 nm, 20 nm FWHM; short pass optical filter (<450 nm) was used in this case)

Conclusions

- there was no autofluorescence effect for any photoresist excited with red-line light source (25 mW laser diode, λ_{EX} = 643 nm)
- for green-line light source (100 mW laser diode, λ_{EX} = 532 nm), all the photoresists showed significant autofluorescence. Intensity of this effect was slight for SU-8, but high for both PBAs, with maxima at 550 nm and 600 nm. Proportions of signal intensity at 550/600 nm to λ_{EX} were: 2.4%/4.3% (SU-8), 37.7%/14.9% (PermiNex™ 1000), and 30.1%/13.6% (PermiNex™ 2000)
- excitation with blue-line light source (50 W LED, max. λ_{EX} at 448 nm) had no effect on SU-8, but slight autofluorescence for both PBAs was observed

Example of application

PBAs may be used for permanent and easy capping of silicon/glass structures, including microfluidic devices and MOEMS. In comparison to direct bonding methods, such a solution requires lower temperature or pressure and simpler procedure (Fig. 6, Tab. 1). When compared with indirect bonding using standard photoresist, higher bonding strength are obtained and only single coating is required. These features make PBAs interesting material for quick fabrication of LOCs, analytical microsystems and other microdevices. In this work for the first time, photoimageable bonding adhesive was used to develop hybrid CGE LOC for genetic analysis.

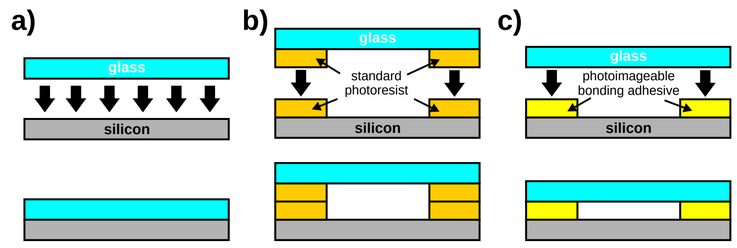


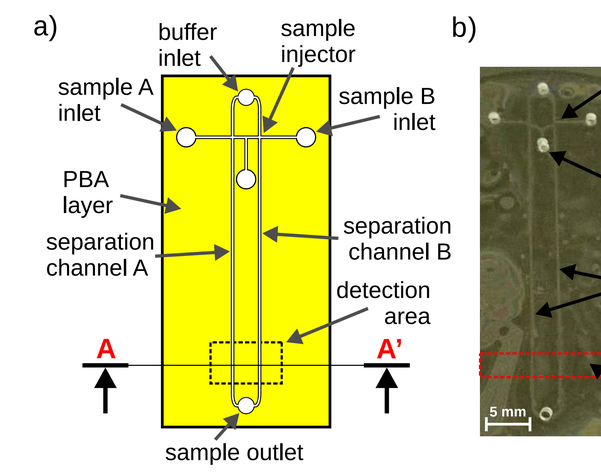
Fig. 6. Various schemes of silicon/glass bonding methods: a) direct bonding, b) indirect bonding using standard photoresist, c) indirect bonding using photoimageable bonding adhesive

Table 1. Characterization of various silicon/glass bonding methods

Process parameter	Direct bonding	Indirect bonding using standard photoresist	Indirect bonding using bonding adhesive
Temperature	High	High	Low
Pressure	High	Low	Low
Photolithography	None	Double	Single
Complexity	High	Medium	Low
Bonding strength	Very high	Low	High

CGE LOC design and fabrication

Developed PBA-glass CGE LOC enables simultaneous analysis of two genetic samples. It contains two symmetrical separation channels (32 mm long, 200 µm wide), two double-T sample injectors, and common buffer inlet, waste and sample outlet (Fig. 7). All microfluidic components were fabricated in a single 14 µm thick layer of PermiNex™ 2000 on a glass substrate, utilizing standard photolithography. Next, PBA-glass structure was bonded with top glass wafer containing vias. As fluorometric detection method was applied (DNA fragments were labelled with fluorophore and detected at the ending of the separation channel), PBA layer also played role of planar waveguide which was used for coupling of excitation light beam into microchannels.



LOC fabrication protocol:

- preparation of bottom glass wafer
- spin coating of PBA (4000 RPM, 60 s)
- softbake (95°C, 10 min)
- UV exposure (i-line, 80 s)
- post-bake (70°C, 2 min)
- development (2.38% TMAH, 3 min)
- rinsing and drying (developer, nitrogen)
- alignment of the top glass wafer
- bonding and hardbake (170°C, 1 h)

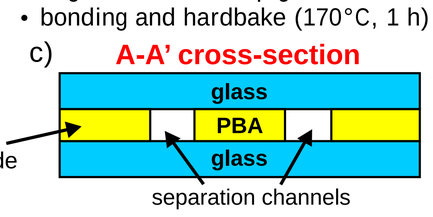


Fig. 8. a) scheme of fluorometric detection setup, b) results of simultaneous electrophoretic separation of DNA ladder samples (mix of 100, 200 and 500 base pairs (bp) fragments, each) in the developed hybrid CGE lab-on-a-chip

Discussion and summary

- Optical detection methods and integrated planar waveguides are often used in MOEMS and LOC devices. In such solutions high optical transmittance of construction material is required. Planar waveguides of various optical path length were fabricated using two types of photoimageable bonding adhesives (PermiNex™ 1000 and PermiNex™ 2000), as well as reference SU-8 resist. The shape of optical transmission spectrum of all three resists was similar, but at a set optical path length, the transmission of the PBA's was up to twice as high as SU-8. It may be concluded, that PBAs are more preferable for fabrication of integrated planar waveguides working in a visible spectrum.
- As many solutions of lab-on-a-chips for biochemical analysis utilize fluorometric detection with laser-induced fluorescence (LIF), it is crucial to utilize construction material with low autofluorescence. It was found, that for applied here standard excitation light sources autofluorescence of PBAs is low at blue-line spectral range, high at green-line range and not observed at red-line range. Therefore, these new materials may be applied in solutions utilizing Cy5, TOPRO-3 and other „red" fluorophores, but for „green" dyes (e.g. HEX, PE-Cy7, Alexa Fluor 532) low signal-to-noise ratio must be taken into account.
- For the first time, CGE chip for genetic analysis was fabricated using photoimageable bonding adhesive (PermiNex™ 2000). All microfluidic components were patterned in the layer of PBA using standard photolithography, which was next used to bond glass wafers in a low temperature process. Total time of LOC's fabrication was less than 24 hours. Two samples of DNA ladder were simultaneously separated and successfully detected in the developed LOC. It may be concluded, that bonding adhesives may be applied for easy and quick fabrication of microfluidic electroseparation devices utilizing fluorometric detection method.

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