



Design of interfaces with lithographically patterned adhesive pads for gluing at the microscale



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ABSTRACT

The creation of small adhesive pads by traditional dispensing methods is technically limited. However, the miniaturisation of micromechanical components requires the parallel development of adhesive pads with sizes in the sub-50 μm range combining good geometrical confinement and mechanical strength. Therefore, a new design of interfaces with adhesive pads of 32–8 μm are presented through local deposition of a liquid adhesive by means of “top-down” or “bottom-up” patterning. Using lithography and photochemical process, the shape of the adhesive pads is first stabilized by partial cross-linking and effective adhesive bonding with a counterface subsequently takes place during full cross-linking. The parameters for photochemical cross-linking of the adhesive pads are optimised and the mechanical performance of the patterned adhesive interfaces is evaluated. For “top-down” patterned adhesive interfaces, the geometrical stabilisation of the adhesive pads requires relatively long cross-linking times consequently resulting in low mechanical strength. For “bottom-up” patterned adhesive interfaces, the formation of adhesive pads is controlled by self-organisation of the adhesive over chemically structured substrates and requires short cross-linking times for geometrical stabilization, leading to higher mechanical strength during adhesive bonding. The fabrication of adhesive pads by a “bottom-up” approach is further discussed in relation to the influences of processing parameters on dewetting of the adhesive.

1. Introduction

Gluing is a straightforward and flexible tool for assembling complex systems, integrating various micro-electronic, optical and/or fluidic components onto different substrates. However, the ongoing miniaturization of microsystem components requires a parallel development of appropriate assembly techniques. The latter presents new challenges to adhesive technologies with respect to dimensions of local adhesive pads: a tiny amount of adhesive should be applied on a targeted position with good precision and high strength. At present, high-precision bonding at the microscale is limited because of poor control over the deposition process, geometrical stability and/or viscosity of the cross-linked adhesives.

Techniques for deposition of liquid adhesive are based on dropwise application onto the surface by means of dispensing methods. Conventional dispensers allow to handle volumes of 0.01 ml, while high-end microdispensers operate with volumes of 1 nl and some recent developments employ techniques that can deliver single drops with volumes as low as 5 pl. The ink-jet printing is a traditional technology to control the precise deposition of liquid droplets. For unfilled or low-

viscosity fluids such as inks, the fluid partially dries during ink-jetting and forms relatively confined drops on the substrate. For printing filled and/or high-viscosity materials such as adhesives, however, the droplets are applied under wet conditions and yield proportionally larger dimensions after deposition due to capillary forces and spreading on the substrate: e.g., adhesive drops in the range of 150 μm easily spread up to 1 mm [1], or the deposition of 80 μm adhesive drops by ink-jet printing of thermoset epoxies results in drop diameters of 150 to 200 μm . The reliability of dispensing tiny amounts of adhesive has been improved under industrial conditions by integrating sensors and design of specific dispensing heads [2]. Other units comprise a positive-displacement dispensing system with integrated adhesive tempering, process gas supply and process optics [3]. Other techniques such as screen printing enable to form drops with a diameter of about 100 μm and line-widths of 80–150 μm . Micro-stencils with 100 μm pitch and 50 μm apertures were recently developed for printing solder pastes [4]. Whereas most printing technologies are in general optimised for one specific substrate such as paper, adhesive technologies should be more flexible. The confinement of adhesives into small geometries requires sophisticated designs with glue guiding channels [5], or stamping and

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selective transfer [6]. A novel micro-bonding technique using a focused laser beam with a spot size of 30 μm for local cross-linking [7]. However, those techniques often require the initial formation of a full adhesive layer, while adhesive residues outside the joint are removed afterwards. New approaches in designing adhesive interfaces includes micro- to nanoscale patterned substrates [8], or capillary-driven self-assembly of pads in the 100–300 μm range [9]. The need of small adhesive pads has been inspired by nature, observed as arrays of fine adhesive spots on the foot pads of many insects.

Among different available adhesives, photochemically cross-linkable materials are most promising for microscale glueing, because they have advantage of low shrinkage and interfacial stresses, while the cross-linking is an ecologically friendly process at room temperature. Dissimilar materials are preferentially bonded as such, by using e.g. polyester [10], benzocyclobutene [11], polyacrylate [12], or epoxy [13] glues. The adhesives are available as monomer mixtures with photo-initiators (e.g., thiol [14], benzophenone [15] or copolymerizable initiators [16]) and are cross-linked into densely compacted networks [17] or (semi-)interpenetrating networks [18]. During cross-linking, an elastic fluid converts into a visco-elastic gel by radical polymerisation. The rheological behaviour of such adhesive networks has been studied as a function of the cross-link density [19]. Using molecular dynamic simulations, the role of network connectivity and polymerisation kinetics for different cross-linker functionalities and interfacial bond densities can be followed [20]. The resulting mechanical properties depend on the type of components and entire processing: the failure stress decreases and failure strain increases at lower percentages of cross-linking, as a volume fraction of non-polymerised or 'free' molecules into a cross-linked network significantly alters the adhesive properties [21]. On the other hand, a small number of polymer chains with free end segments increases the adhesion if they can penetrate into the opposing surface [22]. The photo-definable adhesive films with light curing resin may allow for photo patterning, although it is often difficult to reacting high adhesiveness after patterning and it was previously used in combination with a thermal curing resin [23]. Therefore, a close control of the processing parameters will thus plays an important role in setting of the adhesive bond, its dimensional stability and final adhesive strength.

Literature search reveals that flexible techniques for deposition of adhesives pads with sizes in the sub-50 μm range are scarce. In this paper, we present an alternative strategy to form small adhesive pads where the dimensions are confined through direct lithographic patterning of the adhesive layer (here defined as "top-down") or the template-dependent self-organization of the adhesive layer onto a lithographically patterned substrate (here defined as "bottom-up"). In particular, the "bottom-up" approach first requires a lithography process to define a hydrophobic coating on the surface which then acts as a template to guide self-assembly of the adhesive layer deposited by dip coating. We present here a two-step cross-linking process to create small adhesive pads by lithographic patterning and photochemical cross-linking: (i) partial cross-linking of the adhesive will be used to generate and stabilize an adhesive pattern ('pre-curing'), and (ii) full cross-linking of the residual photo-active groups in contact with the counterface will allow for effective bonding ('full-curing'). The performance of the resulting adhesive interfaces will be analysed by spectroscopy and shear testing.

2. Materials and methods

2.1. Glue type

A commercially available UV cross-linkable adhesive NOA 74 (Norland Optical Adhesive) was used: this is a solvent-free one component glue containing 15 to 35 wt% isodecyl acrylate and 4–15 wt.% trimethylpropane polyoxypropylene triacrylate ester as monomers, together with 43–65 wt.% thiol-ester as photo-initiator. The viscosity at

room temperature is 0.09 Pa.s, with a density of 1.1 g/cm³ and surface tension of 0.04 N/m. In order to optimise the viscosity and wetting properties of the adhesive, it was mixed in different concentrations between 100 and 30 vol% with ethylacetate, ethanol or acetone (Sigma Aldrich, Germany). The photochemical cross-linking was done in a UV Stratalinker at 365 nm wavelength with a power of 65 W for different times.

2.2. Substrate types and surface preparation

In a first design for "top-down" patterning of the adhesive interface, standard microscope slides of polymethyl-methacrylate (PMMA) were used as substrates that were cleaned with ethanol and D.I. water prior to use.

In a second design for "bottom-up" patterning of the adhesive interface, the microscopic slides of polymethylmethacrylate (PMMA) were patterned with alternating hydrophilic and hydrophobic surface areas. Therefore, the substrates were dip-coated in a 1 mg/ml solution of a 1% benzophenone-fluoropolymer, poly [(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-hepta-decafluoro-decylacrylate)-co-methacryloyloxy-benzophenone] or MABP-FP, resulting in a film with thickness of 8 to 10 nm when deposited under controlled withdrawal speed. This hydrophobic film was lithographically patterned during cross-linking in a UV Stratalinker at 250 nm wavelengths for 30 min under the application of a mask with square patterns of 32 \times 32 μm^2 , 20 \times 20 μm^2 , 16 \times 16 μm^2 or 8 \times 8 μm^2 and interdistances of 32, 20, 16 or 8 μm respectively. Under UV radiation, the benzophenone groups act as photo-initiators and allow for photochemical cross-linking of the fluoropolymer film together with covalent attachment to the substrate. The non-crosslinked areas covered by the mask were subsequently removed by extraction with 1,1,2-trichlorofluoroethane for 2 h in a Soxhlet apparatus. The substrates prepared by this procedure have a hydrophobic surface with hydrophilic patterns defined by the mask layout.

2.3. Characterisation

2.3.1. Adhesive characterisation

The adhesive composition was evaluated at different steps of cross-linking by spectroscopy. For FTIR spectroscopy, the adhesive was coated on a silica surface and cross-linked for different times using a UV Stratalinker at 365 nm wavelength with a power of 65 W. The FTIR measurements were done on an Excalibur FTS 3000 instrument (Bio-rad Laboratories, Cambridge, MA) at wavenumbers between 4000 and 400 cm⁻¹ with a resolution of 4 cm⁻¹. For UV/VIS spectroscopy, the adhesive was coated onto a glass slide and cross-linked under same conditions as mentioned before. The UV/VIS measurements were done on a Cary 50 Bio (Varian Inc., Palo Alto, CA) between 420 and 300 nm wavelengths.

The mechanical strength of patterned adhesive interfaces was evaluated by a lap-shear test on joint samples with dimensions 25 mm \times 75 mm and an overlap area of 25 mm \times 20 mm following international standards ASTM D1002. The "top-down" and "bottom-up" patterned adhesive interfaces were joint with a counterface during a second cross-linking step, so-called "full-curing" (minimum pre-curing times were chosen for stabilization of the adhesive pads, i.e. 60 min pre-curing of "top-down" and 15 min pre-curing of "bottom-up" patterns). A non-patterned PMMA substrate cleaned with ethanol and D.I. water, was placed on top of the patterned adhesive interface under given load (10 N) and the joint was cross-linked for different times. The maximum tested curing time was taken as a practical compromise after a parametric time study, where the joint reached 80% of its maximum strength after 20 min cross-linking and 100% of its maximum strength after further ageing for 6 days. As a reference adhesive joint, a continuous adhesive layer was applied in between two non-patterned PMMA substrates by dip-coating and cured under comparable

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