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# HYBRID MICROASSEMBLY IN ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE USING ROBOTIC MANIPULATOR AND ADHESIVES

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## Background

Starting from mid-90s, scanning electron microscope (SEM) has been used as an observation and operation tool for robotic manipulation systems for handling, characterizing and testing of micro- and nanoscale, e.g. manipulation and characterization of nanotubes [1], [2], mechanical characterization of nanowires [3], [4] and electrical measurements of nanoscale objects in-situ [5]. One of the major motivations to use SEM is its high imagining resolution, down to sub-nanometer scale, which is orders of magnitude better than optical microscopes. Besides, SEM can also be used as a tool, for e.g. electron induced deposition (EBiD). However, robotic manipulation in conventional SEM cannot work with liquids, partly due to the requirement of high vacuum inside the specimen chamber which makes imaging any conventional liquid, e.g. water, impossible in a temperature in the range of ambient environment temperature.

The introduction of environmental scanning electron microscope (ESEM) has alleviated the requirement of high vacuum imaging conditions of the conventional SEM. The ESEM allows pressures high enough for liquid water in the specimen chamber by introducing a variable pressure aperture and specialized gaseous secondary electron detector [6]. The ESEM has been used extensively to observe wetting of liquids on different surfaces, e.g. water condensation on lotus leaves [7], study of wetting of water on hierarchically structured surfaces [8], [9] and nanopipes [10], studies on the wettability of water on silicon surfaces modified by electron irradiation [11] and wetting behavior of water on structured surface during evaporation and condensation [12]. Recently, robotic manipulation of biological samples has also been reported, e.g. cell adhesion measurements [13], determination of the elastic properties of cells using indentation [14] and cell viability testing [15]. Compared to an optical microscope the ESEM offers a stable environment with controllable temperature, pressure and ambient gas combined with a nanometer imaging resolution in high pressure mode, both of which are important aspects for experimental tests.

Capillary self-alignment is a key process for many self-assembly process and hybrid microassembly. In capillary self-alignment, microparts are aligned to corresponding receptor sites by the surface tension of a liquid meniscus between the part and the receptor site. Capillary self-alignment is one of the major alignment principle for many self-assembly techniques [16]. Recently, hybrid microassembly technology combining capillary self-alignment and traditional robotic microassembly has been demonstrated to be a promising method to achieve fast yet accurate assembly of microchips [17-19].

However, the capillary self-alignment process is effectively influenced by the micro- and nano scale features on the surfaces and the associated wetting phenomena. Traditionally the self-alignment process is observed under optical microscopes, which can provide limited details due to the resolution, as the theoretical limitation is in the submicron range and the practical resolution is usually much worse.

In this paper, we report our initial tests on droplet self-alignment assisted hybrid microassembly of microchips with adhesive inside an ESEM. To authors' knowledge, such tests involving self-alignment were first time done in an ESEM. The motivation is to observe details of the microassembly process during the experiments to gain better understanding of the process, instead of only the aftermath assembly results. We are also aim at gaining better knowledge on how to work with liquid and a robotic micromanipulator inside ESEM.

The rest of the paper is structured as follows. Section II describes the experimental setup inside the ESEM and the preparation of samples. The section III reports the experimental results and discusses our current understanding on self-alignment inside ESEM. Section IV concludes the paper.

## System setup

The experiments were conducted using a Carl Zeiss EVO HD15 environmental scanning electron microscope (ESEM) with LaB6 electron source. As opposed to conventional scanning electron microscope, the ESEM allows a pressure up to 2000 Pa in the specimen chamber, which makes imaging of liquids and uncoated insulators possible. The microscope has a specified resolution of 3 nm using 30 kV acceleration voltage in a variable pressure environment. The large chamber size of the microscope allows the use of samples with a maximum diameter of 250 mm and a maximum height of 145 mm, respectively. The microscope is equipped with a motorized 5 axis compucentric stage, which has a travel range of 125 mm in x and y directions and 50 mm in z direction. The stage can be tilted from -10 to 90 degrees and has a rotation range of 360 degrees. The large chamber size and the integrated motorized stage make this microscope an ideal platform for hybrid microassembly tests.

The robotic manipulation largely relies on the 5DOF motorized stages of the ESEM. A microgripper (SmarAct model SG-06) was installed inside the specimen chamber of the microscope to allow pick-and-place operations. The gripper has a stick-slip piezoelectric actuator with a nanometer resolution and an exchangeable gripper head. The gripper was mounted to the wall of the specimen chamber with an aluminum mounting structure. The position of the gripper can be adjusted with a 3 axis manual stage (Newport M-MT) with 9.5mm travel range in all three dimensions. The gripper mount attached to the ESEM specimen chamber door is illustrated in Fig. 1(a). The sample holder and part of the motorized stage can also be seen in the figure. The gripper is controlled with a SmarAct HCU-3 hand control unit. Picture of the gripper installed inside the specimen chamber is presented in Fig. 1(b).

The decision to mount the gripper on the chamber wall instead of e.g. the motorized sample stage was based on the fact that the design is simple and fast to build. As the 5 DOF sample stage could be utilized for moving sample relative to the gripper, no additional motorized stages are required. Likewise, by mounting the gripper to the chamber wall it is insulated from the sample stage of the microscope and the built-in touch detection of the ESEM instrument can be utilized in contact detection.

The gripper was selected based on its exchangeable gripping head combined with the nanometer resolution, which allows using the same gripper for manipulating objects with various shape and size, thus making the manipulation system easily adaptable for using e.g. smaller chips in the future.

The hybrid microassembly experiments were conducted on a nanostructured black silicon substrate [20] with silicon dioxide receptor sites. The receptor sites are slightly higher (0.5 μm) than the background, and the edge is used to confine the adhesive to the pad based on Gibbs equation [21].

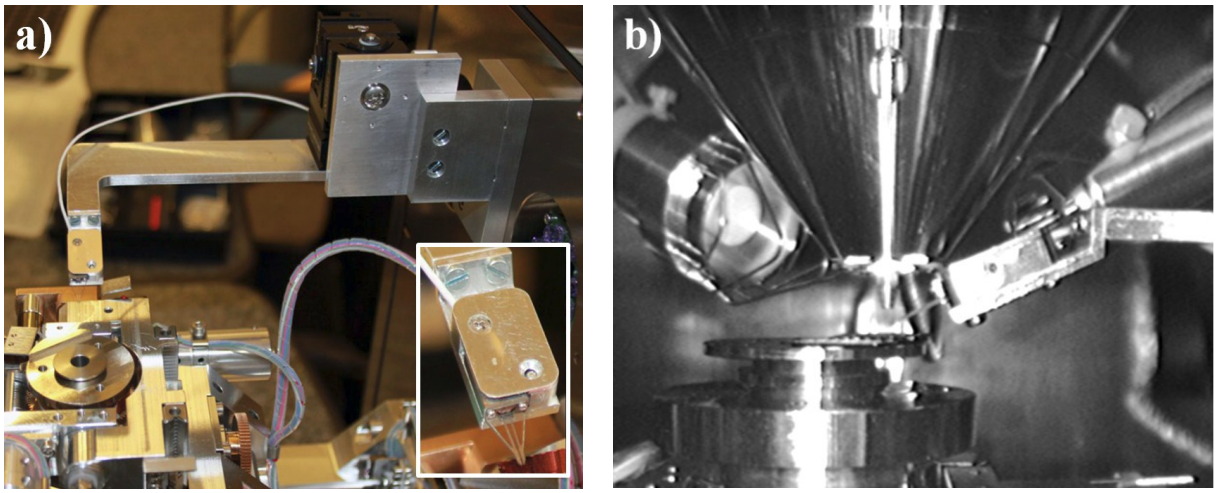


**Figure 1.** *The gripper a) mounted to the chamber door of the ESEM, and b) in position when the chamber door is closed.*

Test chips were fabricated from SU-8 epoxy-based photoresist following the standard fabrication process. The lateral size of both the chips and the receptor sites is 200 μm × 200 μm. The thickness of the chip is 30 μm.

The liquid medium used in the experiments is adhesive. The reason for using adhesive in the self-alignment tests instead of more widely studied water is the smaller volatility, which makes maintaining the liquid phase easier. To keep water liquid inside the ESEM, the atmosphere must be kept close to 100% RH to balance the condensation and evaporation. In practice this is difficult to achieve for prolonged periods required to perform the self-alignment experiments. Furthermore, since the amount of liquid deposited on single receptor site is small, in the range of nanoliters, the water would have to be deposited to the receptor sites in-situ to avoid the evaporation of droplets during sample transfer and ESEM chamber pumping down. In contrast, adhesive can be deposited to the receptor sites before the sample is moved to the ESEM.

Numerous adhesives have been tried. The most promising one is Delo MONOPOX AD VE 111986. The adhesive can be thermally cured and has a viscosity of 900 mPa s and surface tension of 34 mN/m, which is approximately half of the surface tension of water. The adhesive was deposited on the receptor sites with a pneumatically operated dispensing pen equipped with a glass capillary in ambient conditions using a micro-assembly station. After adhesive dispensing, the substrate and the chip tray containing the SU-8 microchips were attached to a 30 mm sample stub using carbon tape and put inside the ESEM for the self-assembly experiments.

Inside the ESEM the experiments were done in a water vapor atmosphere with a pressure of 650 Pa, which was found out to be suitable in terms of image quality. The temperature inside the chamber was not controlled and thus assumed to be the same as the room temperature. With these parameters, the adhesive remains in liquid form.

## Results

Inside the ESEM, the hybrid assembly tests were done by first picking up a SU-8 chip from the chip carrier wafer with the microgripper and then transporting the chip over a receptor site with deposited adhesive droplet. The chip was then lowered to a set height and released from the gripper. If the conditions are right, the chip should self-align with the receptor site due to the capillary force of the meniscus between the chip and the receptor site.

As the SU-8 chips do not display transparency in ESEM as they do with optical microscope the height where the adhesive starts to wet the bottom of the chip cannot be determined as easily as with an optical system. The correct release height was determined by trial and error.

Tens of tests with different adhesives have been tried. Among them we observed promising results with the Delo MONOPOX AD VE 111986. Two successful alignments are shown in Fig. 2.

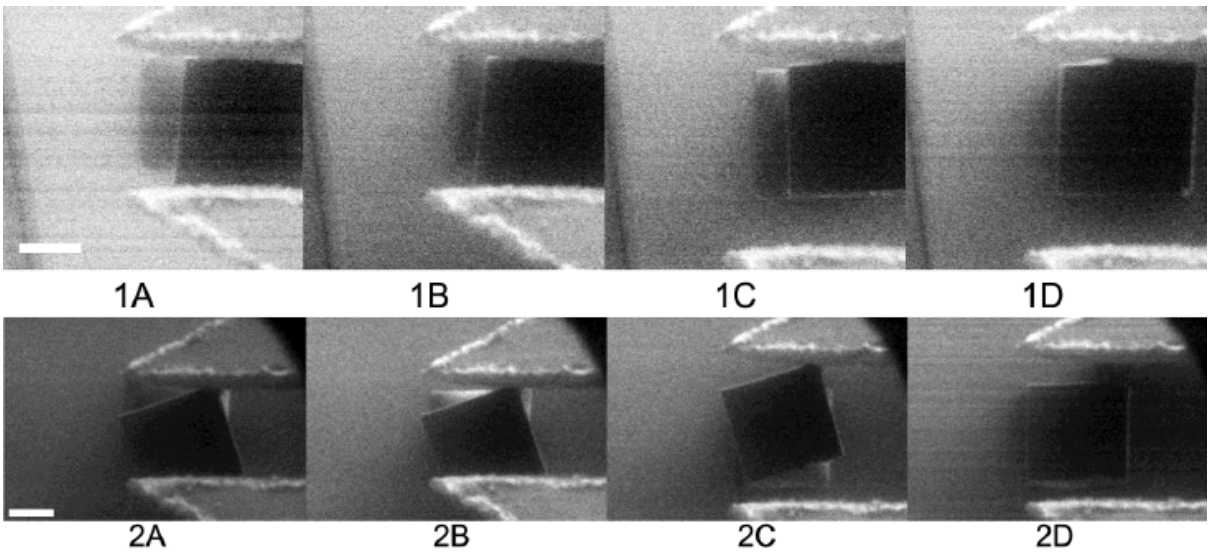


**Figure 2.** *Self-assembly sequences. Scale bars 100μm.*

In 1A, the SU-8 chip has been picked up and is moved on top of the receptor site with deposited adhesive. In 1B the chip is lowered towards the receptor site. The gripper is opened on 1C and self-alignment occurs, resulting in the final alignment shown in 1D.

On sequence 2A-2D, instead of gripping the chip by tweezing, the chip was picked up by adhering it to the other jaw of the gripper. In 2A and 2B, the chip is transported on top of the receptor site and the approach to the receptor site is started. On 2C, the gripper is opened and the chip is lowered in contact with the adhesive. Self-alignment occurs once capillary force between the chip and the adhesive overcomes the adhesion force of the gripper and the chip, resulting in the final alignment shown in 2D.

When the hybrid microassembly was performed, the scan area of the microscope was restricted to small region of interest around the gripper and high scan speed was used to achieve fast refresh rate of the image. To obtain high quality images of the wetting process, the movement can be stopped and a slow scan speed combined with noise reduction techniques used.

Multiple failed hybrid microassembly experiments were witnessed. Different failure cases are illustrated in Fig. 3. The main reason for failed self-alignment was excess amount of adhesive on the receptor sites. As the chip was brought to contact with the adhesive, the adhesive overflow occurs and the self-alignment naturally fails, resulting in final alignments shown in Fig. 3(a). Other reasons for failed self-alignment were incorrect release height and adhesive wetting the gripper, which are illustrated in Fig. 3(b,c). Too high release height will result in the adhesive not wetting the chip at all and too low release height in adhesive overflow due to the forced wetting, respectively. Because of the relatively large size of the gripper jaws relative to the microchip and the receptor site, the adhesive could wet the gripper if the chip was brought too close to the receptor site prior to releasing it, resulting in adhesive overflowing from the receptor site. The problem can be solved by using a gripper jaw of different design.

The initial results show that hybrid microassembly is possible inside an ESEM, which is very promising. Nevertheless, a lot of future work is needed to fully utilize the power of ESEM for studying hybrid microassembly and self-alignment phenomena in-situ. The main challenges reside in chip handling inside the ESEM chamber, namely to improve the robustness of the chip releasing process. As accurate control of the amount of adhesive on the receptor site to guarantee uniform chip drop height is challenging, the correct release height should be selected on-chip basis.

To overcome the problem, few possible solutions are identified. First, the chip release process could be observed from side, which would require the sample to be tilted. Since the current manipulator design does not allow tilting of the sample to the extent required to determine the correct release height of the chip, a refined design of the manipulator is required.

Second, as depth detection from SEM images is a common problem, some reported solutions can be found from the literature. A method to calculate droplet contact angle from topview SEM images has been presented by Stelmashenko et al. [23] , the correct release height could be determined based on the contact angle and size of the adhesive droplet prior to chip release. In addition, focus and defocus based depth detection techniques have been reported [24] that could also be used for determining the correct release height, although the large depth of focus of the microscope and lack of features on the droplet might hinder the accuracy of such technique.

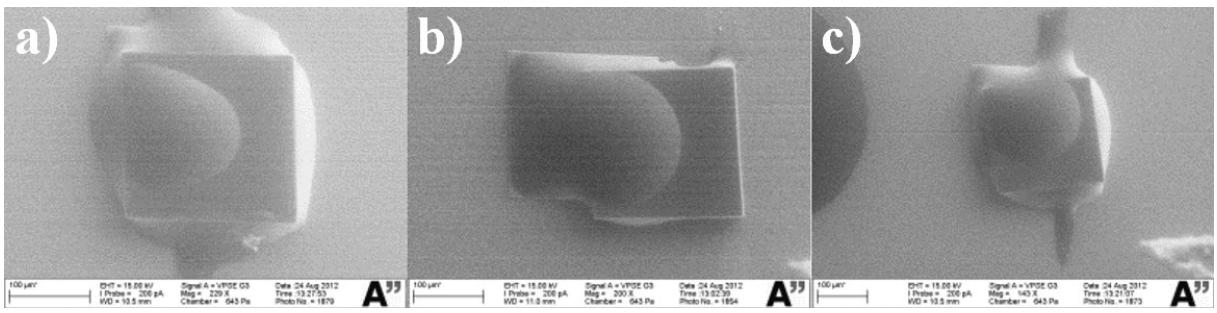


**Figure 3.** *Different failure cases of adhesive self-alignment: Adhesive overflow (a), incorrect release height resulting in adhesive overflow (b) and adhesive wetting the gripper jaws (c).*

## Discussion

In summary, droplet self-alignment assisted hybrid microassembly experiments with adhesive is possible to be carried out inside an environmental scanning electron microscope, utilizing SU-8 microchips and protruded silicon dioxide receptor sites. Contact angle measurements of adhesive droplets on the black silicon surface in ambient air and water vapor atmosphere inside the ESEM show slightly bigger differences that can be expected from instrumental errors, which are suspected to originate from the different interfacial energies in the two respective atmospheres. To the best of authors' knowledge, self-alignment was observed for the first time inside ESEM.

While some challenges remain in handling the chips inside the ESEM chamber, the first successful results and the controlled environment and the high imaging resolution make it a valuable research tool for studying self-alignment and liquid-solid interactions in general. Future work on the subject will include the development of an advanced manipulation system for chip handling and studies with different liquids.

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