## Selective Pick-and-Place of Thin Film by Robotic Micromanipulation

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## Abstract

Micro-engineering is increasingly interested in the use of thin films, with a thickness of less than 20 nm. Before integrating these promising materials in complex Nano Electro Mechanical Systems (NEMS), their properties must be characterized. To do that, they must be transferred on specific substrates for analysis. Current manipulation techniques are not suitable to transfer these thin films since they do not allow to select the parts of the object that must be manipulated and the quality of the sample is altered by traces of chemical residues. To perform the transfer of a selected thin film without modifying its properties, this paper presents a novel approach based on local gluing. This method has been validated by experiments performed on graphite films. Successful transfers of thin films of  $4.2 \times 4.7 \ \mu m^2$  to  $70 \times 12 \ \mu m^2$  with an estimated thickness between 10 to 40 layers are demonstrated. Limits of this technique are discussed.

## I. Introduction

With the development of micromechanics and microelectronics, the interest in thin films is increasingly growing, especially for their numerous physical properties (Lee, Wei, Kysar, & Hone, 2008; Booth et al., 2008). These promising materials will be used to make new devices like MEMS or resonators (Bunch et al., 2007). Fabrication processes of thin films are now well known (Geim, 2009; Shukla, Kumar, Mazher, & Balan, 2009). However, to integrate them in complex devices it is necessary to characterize their physical properties. In particular, structural characterization is a primordial and unavoidable step. Structural characterization, at different scales and levels, can be obtained by various microscopy techniques such as optical microscopy, Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Transmission Electron Microscopy (TEM). Some of these techniques require the use of substrates with specific properties (for example, SEM requires conducting substrates, while for TEM suspended samples are needed). Moreover, many possible applications of thin films also encourage the use of particular substrates (e.g., doped silicon is widely used for electronic applications). Thus, it is necessary to place the area of the thin film that must be analyzed on a specific substrate, and thus to be able to manipulate selected parts of thin films.

An important issue is to reduce chemical contact during this transfert. Indeed chemical residues can change properties of thin films (Boukhvalov & Katsnelson, 2009). Understanding of physical properties modifications, by adding chemical materials (e.g. doping), is the subject of many researches ((Boukhvalov, 2011; Yazyev & Pasquarello, 2010; Boukhvalov, Moehlecke, Silva, & Kopelevich, 2011; Lahiri & Batzill, 2010)). Avoid chemical residues on thin films can ensure a constant quality of analysis. Therefore, analysis of thin films properties, without chemical residues, allows an additionnal comprehension of physical properties.

Manipulation of these thin films is challenging since difficulties are due to both microworld properties and 2D geometry of the object. In the microworld, surface forces (electrostatic, van der Waals, and capillary forces) are dominating compared to volumic force (gravity). Since thin films can be assimilated to planar structure with two dimensions significantly greater than the third one, they present a great ratio surface/thickness hence a great interaction between the surface of the thin film and the substrate. Moreover, because of their two dimensional geometry, they do not offer volume for microhandling, and grippers cannot be used. The problem of surface forces must thus be addressed.

Graphene, a monoatomic thick layer of graphite, is the prototype of 2D crystals (Geim, 2009). Because of its many potential applications into different fields, like nanoelectronics (Hwang, Acosta, Vela, Haliyo, & Regnier, 2009; Eda & Chhowalla, 2009), or nano-mecanics (Lee et al., 2008; Bunch et al., 2008), it is a promising material.

To manipulate selected thin films, this paper proposes a strategy based on local gluing. A drop of glue is created on a small isolated part, to preserve from chemical contact the rest of thin film. This method allows to transfer specific area of thin films, selected for their physical properties. This strategy is validated by experiments performed on graphite thin films. Thin films of  $10 \times 10 \ \mu m^2$  with an estimated thickness of 10 to 40 layers (4 to 16 nm) are transferred.

This paper is organized as follows. Section II presents thin films used and their particularities. A review of classical solutions for micromanipulation using microrobotic is made in Section III. Section IV describe the setup and the performed experiments. Results are discussed in section V.

## II. Sample preparation

The graphitic thin films used in this study, are obtained by an anodic-bonding technique adapted to the preparation of graphene (Shukla et al., 2009).

This consists in sticking bulk graphite onto a pyrex glass substrate, by the simultaneously heating the system (to reach temperatures around 200°C) and applying a high voltage (of the order of the kV). After mechanical cleavage of the graphite on pyrex, the samples present graphitic areas of variable thickness, from few hundreds layers down to the monolayer limit. We focus here on the mechanical transposition of graphitic thin films formed by approximately 10 to 40 layers.



Fig. 1. Multi-graphitic thin films. The difference of the contrast corresponds to the different tickness of thin films. More constrast is dark, more the film is thick. For example, A,B and C corrrespond to three thin films more and more thick.

# III. state of art of conventional micromanipulation strategies

A. Conventional manipulations of thin films

Many strategies have been applied to manipulate and transfer graphene. Most of them use a reversible intermediate substrate, designed to maximize contact forces with the thin film (Table I). Since these forces are higher than the ones between the thin film and the initial substrate, the graphene is stuck on the new substrate. The intermediate substrate, for example, can be made of PMMA (Polymethyl Methacrylate), which is dissolved after transfer (Li et al., 2009; Yamaguchi, Eda, Mattevi, Kim, & Chhowalla, 2010) or a thermal release tape which sticking and unsticking properties are controlled by pressure monitoring and heating (Caldwell et al., 2010; Bae et al., 2010). Another solution uses surface tension to transfer graphene to a TEM grid (Regan et al., 2010). With these methods large surfaces of graphene are transferred  $(4.5 \times 4.5 \text{ cm}^2)$  (Li et al., 2009),  $16 \times 16 \text{ mm}^2$ (Caldwell et al., 2010)). However, all these techniques are closely related to chemical transfers, and do not ensure a high quality of thin films since physical properties of thin films might be affected by the transfer. Moreover, these methods do not allow to transfer selected areas of a thin film.

 TABLE I

 CURRENT MANIPULATION OF THIN FILM

	Transfert substrat	Release method
(Unarunotai et	Palladium	Pd: chemical wet
al., 2010)	(Pd)/Polyimide(PI)	etching PI : reac-
		tive ion etching
(Caldwell et	Thermal release	Heating tape
al., 2010; Bae	tape	
et al., 2010)		
(Li et al., 2009;	PMMA	Acetone
Yamaguchi et		
al., 2010)		
(Regan et al.,	isopropanol	surface tension
2010)		

#### B. Microrobotics micromanipulation strategies

Microrobotics can bring a promising solution for micromanipulation since it enables high resolution movements, the possibility to select the manipulated objects and the possibility to perform automated tasks using prehensiles and actuators. Many micromanipulation strategies exist, based on different physical phenomena. A quick description of the main strategies is presented here. More details can be found in (Régnier & Chaillet, 2008).

In particular two categories of manipulation can be identified: contact-free micromanipulation (see sec.III-B.1) and contact-based micromanipulation (see sec.III-B.2).

1) Contact-free micromanipulation: The main advantage of contact-free manipulation is that it enables an easy release operation, since it frees oneself from surface forces like capillary, van der Waals or electrostatic forces. Table II (Régnier & Chaillet, 2008) presents advantages and disadvantages of contact-free manipulation.

Examples of contact-free micromanipulation:

- Optical tweezers: a laser beam manipulates transparent micro-objects using radiation pressure. The particles are attracted to the region of highest light intensity.
- Electrostatic forces: micro-objects are manipulated without contact using an electric field.

Contact free micromanipulation can be also useful in the fabrication of thin films. For example, electrostatic forces have been used to deposit graphene on  $SiO_2$  (Sidorov et al., 2007).

However, contact-free manipulation cannot solve the problem of the manipulation of graphite thin films. Indeed these processes are often limited to a restricted class of materials, in terms of shape and physical properties (Régnier & Chaillet, 2008). Moreover, these strategies provide only low forces (in the order of piconewton (Régnier & Chaillet, 2008)). This is not enough to counteract sur-

#### TABLE II

#### ADVANTAGES AND DISADVANTAGES OF CONTACT-FREE MANIPULATION

	Advantages	Disadvantages
Optical tweez-	Parallel displace-	Restrictive
ers	ment of several	conditions on
	volumic objects	the refractive
		index of
		object(Ashkin,
		1970) -
		Manipulation
		forces in
		the order of
		0.1 - 10  pN
Electrostatic	Mainly biologi-	Better control
forces	cal applications -	with conductive
	particle	materials

face forces to release thin films (in the order of  $300 \text{ nN}/\mu\text{m}^2$  (Zhang, Small, Pontius, & Kim, 2005)). In addition, it is difficult to avoid disturbances, during the manipulation with contact-free micromanipulation, to assure and keep the original shape at the end of the transfer. So micromanipulation of thin film needs a contact between the tool and the film to succeed.

Contact-free manipulation cannot solve the problem of selective manipulation of thin films.

2) Contact-based micromanipulation: Contrary to contact-free micromanipulation, contact-based micromanipulation is not surfaces forces. Therefore free from release strategies are a crucial step in the transfer operation. These strategies allow to miniaturize the prehension principles and moreover they are also capable of producing for manipulation, considerable forces in the order of  $1 \text{ N/mm}^2$ . In table III (Régnier & Chaillet, 2008), advantages and disadvantages of contact-based strategies.

Examples of contact-based micromanipulation:

- Grippers with jaws: devices similar to a hand, with two or several jaws. Different grippers exist depending on their conception
- Phase changes: this method uses the solidification of a liquid (like water) between the prehensor and the micro-

object to pick it up. It is released by re-liquifying the liquid.

- Prehension by depression: prehension based on the aspiration of objects.
- Manipulation by adhesion: manipulation using basic adhesive contact with a prehensor.
- Surface tension: prehensor using modulated surface tension due to the capillary force by modifying the radius of curvature of the prehensor (Biganzoli, Fassi, & Pagano, 2005; Pagano, Zanoni, Fassi, & F.Jovane, 15-17 November 2006).

It is obvious that grippers cannot be used for thin film manipulation because of the 2D structure of the thin films. Methods based on surface tension have already been proposed to transfer graphene. One of this method uses Isopropanol to improve surface tension (Regan et al., 2010): during its evaporation, graphene sticks to the new substrate because of surface tensions. However, a chemical residue could remain on the material which is a strong limiting factor for many applications. Manipulation based on phase changes is difficult to apply for graphite because most devices use water as a liquid and the surface of graphite is hydrophobic. Moreover, in the case of objects of sizes smaller than  $500 \ \mu m$  there difficulties arise for the release operation (Régnier & Chaillet, 2008). Finally, strategies based on depression face a scaling problem due to the difficulty to create a nanometric contact between the tool and the thin film.

Until now, conventional manipulations of thin films are not compatible with our reducing chemical contact objectives and strategies developed by microrobotics do not bring satisfactory solutions to solve the problem of thin film manipulation. A new approach is proposed in this paper, using a micro-drop of glue to create a precise contact between the tool and the graphite thin film to enable the detachment of the thin film. Compared with strategies described above, the approach by local gluing is situated on the boundaries between different methods like phase changes and manipulation by adhesion.

#### TABLE III

ADVANTAGES AND DISADVANTAGES OF CONTACT-BASED MICROMANIPULATION

	Advantages	Disadvantages
Grippers with	Intuitive manipu-	Only for volumic
jaws	lation	object
Phase changes	Independent of the type of materials and their shape - Complete mechanical connection - Manipulation forces in the order of	Lack of compli- ance - used with liquid like water - size of manip- ulated object > 500 µm
	$1 \text{ N/mm}^2$	
Prehension by depression	Mass application	Shape scale: 0.6 mm (Gastel, Nikeschina, & Petit, 2004)
Surface tension	Used to transfer graphene (Regan et al., 2010)	Hydrophilic, oleophilic (Westkämper, Schraft, Bark, Vögele, & Weisener, 1996)

### IV. Materials and methods

#### A. Materials

The setup is presented in Fig.2. A hollow pipette<sup>1</sup>, where UV glue<sup>2</sup> is introduced, is used for local gluing and moving. This type of glue polymerises under ultraviolet action and its viscosity ( $2.5 \text{ Pa} \cdot \text{s}$ ) allows to move it inside the pipette by monitoring the pressure.

The pipette is fixed with an angle of  $10^{\circ}$  with respect to the vertical direction. It is controlled with a Sutter micromanipulator<sup>3</sup>, which is a three-axis manipulator with stepper-motors. The fine course is  $0.04 \ \mu m/step$ . A programmable syringe pump<sup>4</sup> is connected to the pipette with a plastic tube. It controls the speed of the glue by monitoring the pressure inside the tube. So-

lidification of the glue is realized with a UV lamp<sup>5</sup>. Its power is 75 W and the wavelength of the light is in the range of 280 - 450 nm. Wavelength is dispersed in visible light (blue, purple colors) between 400 - 450 nm, UVA between 320 - 395 nm and UVB between 280 - 320 nm. The intensity of the light is greater than  $9000 \text{ mW/cm}^2$ .



Fig. 2. On the left, CAD representation of setup. Pipette is fixed on a three axis micromanipulator (two axis are in horizontal plane and the third is vertical), which is controled manually. Solidification of glue is succeeded with UV lamp, arranged in working zone. Imaging is made from the bottom with inverted microscope. On the right, detail of working zone.

#### B. Methods

1) Gluing and detachment: The area of the graphite thin film that must be manipulated is selected from an image obtained with the inverted microscope. The pipette is moved down to place it in contact with the thin film (Fig.IV-A). This operation is monitored using vision feedback from the microscope. A drop of glue is then created between the tip of the pipette and the thin film, using the syringe pump, set to 7 ml/min, to control the drop. When the drop of glue has reached the desired size, it is solidified with the UV lamp, by lightening it for 30 s. A link is created between the pipette and the thin film. The thin film can then be detached by moving the pipette with the micromanipulator. The first step is to slowly move up the pipette to unstick the area under

<sup>&</sup>lt;sup>1</sup>Pipettes are made with micropipette Puller P1000 from Sutter Instrument with filament FB255B and pre-installed program number 5. It is in thin wall borosilicate tubing and its reference is B150-110-10. Its final dimensions are: tip  $2-3 \mu m$ , taper 3-4 mm

<sup>&</sup>lt;sup>2</sup>Reference 429 from Dymax corporation

<sup>&</sup>lt;sup>3</sup>Micromanipulator P285 from Sutter Instrument

<sup>&</sup>lt;sup>4</sup>Aladdin pump made by WPI

<sup>&</sup>lt;sup>5</sup>Bluewave75 made by Dymax corporation



Fig. 3. Detachment of a graphite thin film. Substrate is light grey and graphite is dark grey. Image I, pipette is in contact with the thin film through a drop of glue. Images II to VI, pipette is moved to the left to peel off the thin film. Image VII, focus is on the substrate and image VIII focus is on the detached graphite thin film. Difference of focus between image VII and VIII shows that graphite thin film has been detached. The size of the thin film is  $70 \times 12 \ \mu m^2$ . The task time is around 60 s.



Fig. 4. Deposit and mechanical removal of the sticking areas on pyrex. The transferred thin film is the part which as been detached on Fig.IV-A. Substrate is light grey and graphite is dark grey. Image I, pipette is slowly moved down to the substrate and puts down in the lengthways of the thin film, from the free edge towards the glue. Images II, pipette is moved perpendicular to the direction of the deposit, to mechanically remove the glue. Image III, the pipette is moved up to finish the deposit. Image IV, transfer is finished, the thin film is on the substrate and the drop of glue is on the pipette. The size of the thin film is  $70 \times 12 \ \mu m^2$ . The task time is around 40 s

the glue from the substrate. Visual control is important and enables to move the pipette in the direction of the tearing of the thin film to increase the size of the manipulated thin film. To detach the extremity of the thin film from the substrate, special care must be taken to avoid folding it over, and pipette must be moved slowly.

2) Release: After detachment is succeeded, the pipette (with the graphite thin film stuck on the tip) can be moved with the micromanipulator to the substrate that will be used for the analysis. The pipette is then slowly moved down to the substrate. Deposit goes in the lengthways, from the free edge towards the glue (Fig.3). The thin film sticks to the substrate due to adhesion forces. The last step is to separate the thin film and the pipette. Releasing is performed by mechanically removing the sticking areas located under the glue. The pipette is moved perpendicular to the direction of the deposit, to keep only the thin film on the substrate without glue, which is attached to the pipette.

This allows to obtain a thin film on a given substrate with limited chemical residue.

## V. Results

The proposed method allows to detach a thin film and to release it in a different location. Both tasks (take off and release) take about 90 s. This strategy has been validated by different experiments succeed on graphite thin films. Two types of transfer has been succeeded on different final substrats. The first transfers have been done from pyrex glass substrat to pyrex glass substrat(Fig.3) with different size of thin films. Secondly, transfer has been achieved from pyrex glass substrat to TEM grid (see Fig.7). The area of the manipulated films range from  $4.2 \times 4.7 \ \mu m^2$ (Fig.5) to  $70 \times 12 \ \mu m^2$  (Fig.3). We estimate by optical contrast that their formed approximately by 10 to 40 layers (corresponding to thickness ranging from 4 nm to 16 nm). Thickness of transfered thin film Fig.3 has been precised with widthwise measured with AFM (see Fig.6 Image II). It is approximatly

15 nm (see Fig.6 Image I) corresponding to a number of layers ranging from 40 layers. Transfer of thin film released onto TEM grid has been controled with SEM<sup>6</sup>. Its area is  $4.8 \times 11 \ \mu m^2$ .



Fig. 5. Manipulation of a very small area of a thin film. Its dimensions are  $4.2 \times 4.7 \ \mu m^2$ . Substrate is light grey and graphite thin film is dark grey. Image I: local gluing is succeed. Image II: thin film is stuck to the pipette. Image III: the pipette is moved to transfer the film. Image IV: the thin film is released. Total time is around 90 s and take off time is around 15 s.

This method is however currently limited by several factors. According to the results, the control of the formation of the drop of glue (speed, shape) is the most important point to succeed this manipulation. It seems that an oblate drop, a thin flat round drop, allows a successful detachment. It is necessary to get an appropriated velocity of

<sup>6</sup>SEM Hitachi S4500



Fig. 6. Measure of transfered thin film (Fig.3) with AFM. Image 6(a), result of AFM measure. Image 6(b), measure has been obtained along one axis (see AFM measure arrow). Thicknesss of this thin film is approximatly 15 nm, corresponding at a number of layers ranging from 40 layers.



Fig. 7. Thin film transfered on TEM grid. Image 7(a): TEM grid is under inverted optical microscope. Image 7(b), 7(c), 7(d): Image from SEM. Image 7(c) et 7(d): TEM grid is tilted at  $45^{\circ}$ . Image 7(d): Thin film has some defects. It can be dust, which could come from transport of TEM grid into SEM.

glue deposition to create this oblate drop. If the speed is too low, the drop tends to go up because of capillarity. Although physical comprehension of gluing phenomena is not completely achieved, it seems that different physical phenomena help the detachment of the thin film. Two main effects are described here.

First, we think that during polymerisation, the drop of glue retracts itself, breaking the first layers of the thin film and improving the grip. Second, even after polymerisation, the drop remains flexible. This improves detachment. This flexibility needs to be controled by round-trips with micromanipulator, to avoid a sudden release of elastic energy of the drop of glue which could induce the break of the tip of pipette or sticking of the thin film on the glue.

When conditions (shape and size of the drop of glue) are satisfied, we successfully detached from the substrate region a selected thin film thicker than 10 layers. The same approach applied to thinner regions allows to detach only the part of the graphite that is in direct contact with the glue, as in Fig.8.

The shape of the torn thin film is unpredictable. It is necessary to adapt movements of the pipette based on visual feedback to increase the dimensions of the detached thin film. This shape influences the release step since the thin film should be placed on the substrate from the free extremity to the glued one. The time of the complete task (take off and release) is around 1 or 2 min. It mainly depends on the take off task (pull on drop of glue and take off the thin film). Trenches, created by engraving, do not enable to detach predictable shapes (Fig.9).

Area of thin film, released on TEM grid, has not been maximized. Only a few parts of detached thin film has been released to the grid. Area of the original thin film was  $32 \times 12 \ \mu m^2$  and area of released thin film onto TEM grid is only  $4.8 \times 11 \ \mu m^2$ . The grid is very flexible and fragile, and can be broken up by the tip of micropipette and glue. Sutter micromanipulator has three degrees of freedom (two movements are in plane and the third is vertical). Thus, it is difficult to control the contact between tip of micropipette and grid. Micropipette needs an additional degree of freedom like tilt, to avoid this contact and improve release of thin film. Increase degrees of freedom of setup could ensure an easy release of thin film.

## VI. Conclusion

In order to characterize thin films using specific equipments such as AFM or SEM, it is necessary to be able to manipulate selected



Fig. 8. Hole created by a drop of glue on a thin film composed of less than 10 layers. Membrane is dark and light grey, substrate is very light grey



Fig. 9. On the left, trenches have been made on selected thin film before gluing, to control the shape of detached thin film. On the right, the pipette with the detached thin film. The shape of the detached sample remains unpredictable. Surface of detached thin film (Fig.9(b)) is  $32 \times 12 \ \mu m^2$ .

parts of thin films without altering their properties by chemical residues. To reach this goal, a technique based on local gluing is proposed in this paper. This method is validated by experiments on graphite. Thin films of  $4.2 \times 4.7 \ \mu m^2$  to  $70 \times 12 \ \mu m^2$  with a thickness of around 15 nm have been manipulated. These transfers have been succeeded to two different substrats, pyrex and TEM grid. This work is the first step towards selective transfer of thin films. Some stages must still be improved to get a full control of the shape and the number of layers of the released thin film. Future developments include the automation of the system, the increase setup's degree of freedom and the reduction of the number of manipulated layers .

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