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Analysis and specificities of adhesive forces between microscale and nanoscale

Michaël Gauthier*Member, IEEE, Sébastien Alvo†‡, Jérôme Dejeu‡, Brahim Tamazdet‡, Patrick Rougeot* and Stéphane Régnier†Member, IEEE.

Abstract - Despite a large number of proofs of concept in nanotechnologies (e.g. nanosensors), nano electromechanical systems (NEMS) hardly come to the market. One of the bottlenecks is the packaging of NEMS which require handling, positioning, assembling and joining strategies in the mesoscale (from 100nm to 10µm, between nanoscale and microscale). It requires models of the interaction forces and adhesion forces dedicated to this particular scale. This paper presents several characteristics of the mesoscale in comparison with nanoscale and microscale. Firstly, it is shown that the distributions of charges observed on the micro-objects and meso-objects would have negligible effects on the nano-objects. Secondly, the impact of both chemical functionalisation and physical nanostructuring on adhesion are presented. Thirdly, the van der Waals forces are increased by local deformations on the mesoscale contrary to the nanoscale where the deformation is negligible. This article shows some typical characteristics of the mesoscale.

Note to Practitioners - Micro and nanorobotics covers a high range from nanometers to micrometres which represents six orders of magnitude. Most of the micro-assembly activities have been focused on micro-objects whose size is 10µm or more when nanohandling provides solutions mainly for nano-objects up to 100nm. The interest in the medium scale (mesoscale) has been growing recently. This article presents an analysis of the behavioral characteristic of the objects on this scale in comparison with the two others. It shows that specificities exist on the mesoscale and illustrates the requirement of original micro-nanorobotics at this particular scale.

Index Terms - Microscales, nanoscales, mesoscale, micro-nanohandling, modeling, interaction forces

I. INTRODUCTION

Micro and nanorobotics have provided a lot of handling, manipulation and assembly strategies on both micro and nanoscales during the last twenty years [1-21]. However, most of the micro-assembly activities have been focused on micro-objects whose size is 10µm or more when nanohandling provides solutions mainly for nano-objects which are smaller than 100nm.

On the one hand, top-down approaches based on contact manipulation and tweezers are proposed [1], [2], [3], [4]. The main challenge is to tackle adhesion which becomes typically predominant around several hundred micrometers [5]. In order to avoid adhesion, authors have proposed to use self-assembly processes based on capillary forces [6], [7], [8], magnetic principle [9] or dielectrophoresis [10], [11]. These articles propose new ways to perform micro-assembly of complex and hybrid microsystems [3], [4].

On the other hand, nanomanipulation is mainly based on self-assembly using chemical processes [12], [13]. Authors have also proposed to manipulate nano-objects and typically Carbon NanoTube (CNT) or macromolecules with nanotweezers [14], [15], [16]. The objective is mainly to build nanocomponents (e.g. based on CNT) or to functionalise surfaces (e.g. based on Self-Assembly Monolayer - SAM [17]).

On the mesoscale between 100nm and 10µm some new exploratory works have been reported based on contact handling [18], [19] and based on non-contact manipulation [20], [21].

If we compare manufactured objects to biological ones, this mesoscale between nanoscale and microscale is the dimension of biological cells: the elementary components of life. In the biological domain, cells create a bridge between DNA on the nanoscale and tissues on the microscale (see figure 1). This multiscale assembly enables the construction of biological tissues with highly advanced properties e.g. self-reparable materials, powerful actuation. In comparison, the advent of micro-assembly strategies on the mesoscale could be a high challenge in order to build a bridge between microsystems and nanotechnologies. Fulfilling the lack of methods and technology on the mesoscale should open the way to smart and adaptive materials in several applications: energy harvesting, security and Structural Health Management (SHM).

The study of mesoscale assembly requires a better under-
Standing of object’s behaviour in this scale which is significantly different to nanoscale and microscale. The objective of this paper is to present some characteristics in the behaviour of objects in mesoscale. It particularly focuses on the adhesion forces which are the most critical perturbations in contact micro-nanohandling (gripping, pushing). The next section emphasises the different current methods to model adhesion on the mesoscale and the nanoscale. The measurement of forces is essential to validate model and is presented in section III. Some typical significant coupling effects on the mesoscale are going to be presented in the following sections.

II. Modeling the micro-nanoworlds

Both ways to model adhesion are used in the literature: (i) based on nanoanalysis, adhesion forces are built as the sum of the interaction forces (e.g. van der Waals forces); (ii) based on microanalysis, where adhesion calculation is based on energetic models (e.g. JKR [22], DMT [23]). In both cases, the models propose analytic equations in the case of a contact between a sphere and a plane.

A. From the microscale modeling...

Top-down models are currently used to model adhesion. Energetic modeling based on continuum mechanics can be used to estimate the pull-off force between objects. The most famous models are the JKR [22] and the DMT [23] models which are limited to simple geometries. They are widely used to estimate the force required to break the contact surface between objects: pull-off force. However, they provide a global value of the pull-off force without modeling the impact of each elementary effects (capillary force, electrostatic force, van der Waals force). This kind of model is not flexible: adding a complementary force or changing the geometry is complex. This is one of the major drawbacks of this approach.

B. ... to the nanoscale modeling

Adhesion is induced by several phenomena as van der Waals, electrostatic and capillary forces [24]. Each of them depends on several parameters (materials, roughness, humidity, temperature, etc.) and are due to nanophenomena.

Concerning van der Waals forces, it is commonly assumed to be additive forces. Consequently the total van der Waals forces between two objects  are the sum of forces applied on atoms of  by atoms of  [25], [26], [27]. Adhesion is currently modeled by considering a minimal distance between both objects.  is usually set to 0.3 nm which is the equilibrium distance between two atoms in a vacuum. Throughout this paper, we consider interaction forces between a glass sphere of radius  and a plane. The van der Waals forces induced by this geometry are [1]:

\[ F_{vdw} = -\frac{A_{12}r^2}{6z^2}, \]  

where  for contacting objects and  is the Hamaker coefficient ( for glass-glass contact).

Principal electrostatic forces are due to surface charges added by friction or chemical treatments (e.g. cleaning, oxidation). The electrostatic force applied by a plane (surface charges 1) on a sphere (surface charges 2) is done by:

\[ F_{es} = 2\pi r^2 \frac{\sigma_1 \sigma_2 \overline{n}_1}{\varepsilon_3}, \]  

where  is the electrical permittivity of the environment, and  is the normal of the plane.

An electrostatic force appears also because of the equilibrium of the Fermi level when two conductive materials are placed in contact [28]. In this case, a potential difference  (typ. from 0 to 0.5 V) appears between both objects and the interaction force is calculated by:

\[ F_{ep} = \frac{\pi \varepsilon_3 r^2 V_F^2}{z_F^2}, \]  

where  is the interaction distance from which the tunneling effect starts which is typically 1 nm in the case of really smooth surfaces [28].

Capillary forces  are due to water meniscus created thanks to ambient humidity or adsorbed layers [29] are done by:

\[ F_{cap} = 4\pi r^2 \gamma_3 \cos \theta, \]
where $\gamma_L$ is the surface tension of water and $\theta$ is the contact angle between the liquid and the solids.

The models presented above can be used to model pull-off force on the microscale. Indeed, the most natural way to model pull-off force is to sum the elementary forces calculated when both objects are in contact.

The major interest of this approach is the fact that each phenomenon can be decoupled. This bottom-up model of pull-off force leads us to understand and compare the origins of the adhesion.

We are going to base our models dedicated to the mesoscale on this approach which enables to study different phenomenon separately.

III. FORCE MEASUREMENTS

Experimental force measurements are required to validate the proposed models. Our analysis is based on the force measurement performed by an Atomic Force Microscope (AFM) which is currently used to measure interactions between nanospheres (radius of the AFM tip) and substrates. In order to measure forces on the mesoscale, micrometer beads are fixed on AFM cantilevers (figure 2).

![Fig. 2. Measurements of the cantilever deformations in SEM: each color represents a deformation of the beam which has been measured using vision algorithms.](image)

The computation of measured forces on the mesoscale requires a careful study of the AFM where the force measurement is a function of the mechanical stiffness of the beam. From a mechanical point of view, the stiffness is directly linked to the mechanical boundary conditions at both ends of the beam. As the use of a micro-sphere in spite of the usual nanoscaled tip is able to significantly change the mechanical boundary conditions on the beam, we studied the mechanical behavior of the AFM beam in mesoscale measurements. To model the general behavior of a cantilever, we considered the deformation in the case of a clamped beam. The general boundary conditions include a force $F_y$ and a torque $T$ applied on the extremity of the beam. Considering a position $x$ along the beam, its deflection $y(x)$ classically verifies:

$$ y(x) = \frac{1}{E I} \left( \frac{F_y}{6} x^3 + \frac{L F_y - T}{2} x^2 \right), $$

where $E$ is Young’s modulus, $L$ is the length of the beam, and $I$ is the momentum of inertia of the beam section. The stiffness of the beam can be expressed by:

$$ \frac{F_y}{y(L)} = \frac{3E I}{L^3} \left( 1 - \frac{3}{2} \frac{T}{L F_y} \right)^{-1}. $$

The ratio between $T$ and $L F_y$ is driven by the boundary conditions at the extremity of the beam. For example, the clamped free condition is characterized by $T/L F_y=0$. The clamped-clamped deformation is defined by $T/L F_y = 0.5$. Between both cases, the stiffness is increased by a factor of 4.

The identification of the boundary conditions can thus be found by measuring the whole deformation $y(x)$ and by identifying the ratio $T/L F_y$. The whole deformation $y(x)$ of the beam has been measured in a SEM and computed using (5) in order to define the ratio $T/L F_y$ (figure 3). We have shown that the ratio $y(L)/F_y$ is negligible compared to 1 (figure 3): the beam follows the deformation of a clamped-free beam. Consequently, current force measurement principles based on AFM, usually used on the nanoscale can be used on the mesoscale.

![Fig. 3. Identified force momentum $F_y L$ and torque $T$ at the end of the cantilever in function of the deflection: $T$ is negligible compared with $L F_y$ which is a characteristic behavior of a clamped free beam.](image)

IV. SCALE EFFECT ON ELECTROSTATIC FORCES

The objective of this section is to present, in a concrete case, the scale effect of electrostatic forces compared to van der Waals forces.

On non-conductive materials, surface charges could appear with tribo-electrification and the level of charges is typically unpredictable. Indeed, no reliable models of tribo-electrification currently exist in the state of the art. Consequently, in order to evaluate the order of magnitude of the electrostatic force $F_{elec}$, experiments have been done on a silicon plane and a borosilicate sphere. Typical surface charges about $\sigma = 0.15 mCm^{-2}$ have been determined using (2) during experiments. This value gives an example of charges which can be induced by tribo-electrification.

This electrostatic force $F_{elec}$ induced by tribo-electrification can be compared with the electrostatic force $F_{elec}$ induced by contact electrification. We are considering the maximal Fermi potential difference $V_F = 0.5V$ which maximizes the force $F_{elec}$ [28].
Figure 4 represents the scale effect on the electrostatic force induced by tribo-electrification, the maximal electrostatic force induced by Fermi equilibrium and the van der Waals forces for a glass sphere. The first result deals with the Fermi equilibrium which induces a negligible force compared with the others. Secondly, the van der Waals forces are proportional to the radius of the bead (1), whereas the electrostatic force $F_{es}$ is proportional to its square (2). This difference induces different scale effects on both phenomena. In the case of glass, it can be seen in figure 4 that the influence of the electrostatic force induced by tribo-electrification has to be considered for objects whose size is greater than 1 μm. On the nanoscale, the electrostatic forces become negligible compared to van der Waals forces (see figure 5).

![Figure 4](image)

**Fig. 4.** Van der Waals (green dashed line) force, electrostatic force $F_{es}$ induced by tribo-electrification (red solid line) and maximal electrostatic force $F_{Vp}$ induced by contact electrification (black dashed line) calculated between a sphere and a plane according to the sphere radius. The electrostatic force $F_{es}$ is considered as negligible as it is lower than the tenth of the van der Waals forces (blue dash-dot line). Considered materials for van der Waals and $F_{es}$ calculations are borosilicate glass, with $A_{12} = 6.5 \times 10^{-20} J$, $z_0 = 0.3 \text{ nm}$ and $\sigma = 0.15 \text{ mC/m}^2$.

The limit of 1 μm is directly linked to the measured charge density used during our calculation. However, this result reports a global trend which shows that tribo-electrification can induce a significant force on the microscale whereas on the nanoscale it can be neglected. The mesoscale is characterized by the transition between both cases where both forces should be considered. In the case of non-conductive materials, the charge density can not be controlled and is unpredictable. Consequently, on the microscale and on mesoscale, the adhesion is difficult to predict and control for non-conductive micro-objects without controlling the electrical charge density. On the nanoscale, the adhesion is mainly induced by the van der Waals forces which are repeatable.

### V. Chemical Functionalisation and Electrostatic Forces

Based on the fact that electrostatic forces can have a predominant effect on the mesoscale, electrochemical effects can be exploited in order to control electrostatic surface charge density via chemical equilibrium between the surface and the liquid medium. Surface functionalisation of both objects and grippers can be obtained by different methods (physisorption, grafting, etc.).

The protonation of chemical functionalisation have been widely studied on nano-objects [30], [31], [32]. On nanoscale, the electrostatic force is characterized by an interaction range of several tens of nanometers. On mesoscale, the impact of electrostatic forces induced by surface functionalisations on beads has been observed experimentally in [33]. It shows the ability to obtain long interaction ranges (up to 100μm), but the experiments have not been compared with a model. At least, electrostatic forces induced by an external voltage between mesoscaled objects have been modelled in [34].

We have proposed a model of electrostatic forces induced by chemical functionalisation. Two chemical functionalisations have been tested: (i) the silane, 3 (ethoxydimethylsilyl) propyl amine (APTES); (ii) the silane, (3 aminopropyl) triethoxysilane (APDMES). Both chemical compounds (APTES, APDMES) used for surface functionalisation are amine functions $\text{NH}_2$ which can be protonated or ionised to $\text{NH}_3^+$ according to the pH. In acidic pH, the amine is totally ionised, then the ionisation decreases and is null in basic pH (between pH 9 and 12).

Our principle is also based on the protonation of silica, which enables the switch from SiO$_2$ to SiO$^{-}$ according to the pH. The combination of both effects enables to obtain a surface whose electrostatic charges switch from a positive value for low pH, to a negative value for high pH. These charges induce an interaction force $F_{es}$ which could induces controllable adhesion phenomena.

![Figure 5](image)

**Fig. 5.** Scale effect on electrostatic forces. On the nanoscale, electrostatic forces become negligible in front of van der Waals forces. On the microscale, electrostatic forces could become strong enough to lead adhesion phenomena.

![Figure 6](image)

**Fig. 6.** Molecules used for the silica functionalisation.

Some force measurements were done on a functionalised plane using a cantilever with a non-functionalised sphere. The results of the measurement are presented in figure 7. The pH significantly influences the forces between the cantilever and the surface. At a natural pH, the electrostatic force is attractive. Indeed a pull-in force is measured when the bead comes to the
surface (near -60 nN) and a significant pull-off force is also measured (-350 nN, figure 7(b)). When the pH increases the electrostatic force becomes repulsive. The maximal repulsive forces reach 280 nN and 770 nN at pH 9 and 12 respectively; and the adhesion forces disappear.

Fig. 7. Force-distance curve for the APTES functionalised substrate in wet medium at different levels of pH obtained with a tip whose spring constant is 0.3 N/m.

Moreover the interaction distance is typically several micrometers in mesoscale when the chemical electrostatic interaction force is only limited to tens of nanometers on the nanoscale [30], [31], [32]. Because of the size of the object, the interaction force on the mesoscale is significantly greater than on the nanoscale. It opens new methods in micro-nanorobotics: non-contact manipulation of mesoscaled objects in an electric field using surface charges controlled by chemistry can be considered.

The forces induced by chemical functionallisations have different properties in each scale. Indeed, on nanoscale it can be considered as a short range force whose interaction distance is smaller than several nanometers. On mesoscale it is a long range force able to induce interaction up to several tens of micrometer. On microscale, weight is becoming greater than chemical based force (sedimentation) and this effect cannot be exploited.

VI. IMPACT OF ROUGHNESS ON VAN DER WAALS FORCES

Another important parameter which significantly modifies the adhesion is roughness. The impact of the roughness on van der Waals forces have been firstly modeled by [35] on simple rough profiles. More recently, models which consider roughness as a repartition of nanospheres have been proposed by [36], [37]. Some more complex models are also proposed in [38], [39]. These models are difficult to validate experimentally, because roughness is usually a random phenomenon.

We have proposed to use nanostructures in order to control roughness on surfaces during the force measurement. It enables the impact of roughness on interaction forces to be studied properly, and also enables the roughness in an application case to be controlled.

Let us consider the nanostructure described in figure 8 which represents the position of self-assembled polystyrene (PS) nanospheres on a surface. These nanostructures were built by the EMPA institute, Thun, Switzerland [40], [41] using self-assembly methods. In an application case and also during force measurements, the location of the sphere on the cantilever up to the structured surface cannot be controlled precisely. The bead on the cantilever $r_2$ touches the nanospheres $r_1$ on a non-controlled position. We have shown that the van der Waals force is included between a minimum and a maximum which verify [40]:

$$F_{\text{min}} = \frac{A_{12}}{r_1 + r_2} \sum_{i,j} r_2 + z_0 + r_1 - 6l_{ij} (l_{ij} - r_1 - r_2)^2$$ (7)

$$F_{\text{max}} = \frac{A_{12}}{r_1 + r_2} \sum_{i,j} \sqrt{(r_2 + z_0 + r_1)^2 - (4/3r_1^2)}$$ (8)

where:

$$l_{ij}^2 = (r_2 + z_0 + r_1)^2 + 4r_1^2 (j^2 - ij + i^2)$$ (9)

$$L_{ij}^2 = (r_2 + z_0 + r_1)^2 + 4r_1^2 (j^2 - ij - j^2).$$ (10)

The comparison between values predicted by the model and the measurement, plotted in figure 9, shows a promising concordance. Moreover it shows a minimum of the interaction force which represents an optimum of adhesion reduction. Indeed, in the right-hand part of the figure (radius greater than 100 nm), in the sums (7) and (8), 1 and 3 nanospheres induce significant forces respectively. In this case, the surrounding nanospheres are too far from the sphere $r_2$ and induce negligible forces. In the left-hand part of the figure (radius smaller than 100 nm), the density of nanospheres is higher and the sum (7) and (8) include a lot of nanospheres. In this case, the smaller the nanospheres are, the higher the number of spheres in the sum is, the higher the total force is. In our experimental case, the optimal radius $r_1$ in order to minimize
the adhesion is between 45 and 100 nm. This optimal value depends on the radius \( r_2 \) and the nature of the sphere placed on the cantilever.

The proposed model can be used to determine the diameter of the optimal PS nanospheres to be placed on a gripper to minimize adhesion force with a grasped sphere. Moreover, using (7) and (8), the model can be used for different types of materials knowing its Hamaker constant and for different geometry \( r_1 \) and \( r_2 \). Some first tests on nanostructured silicon grippers [43] have been presented in [40] (see in figure 10).

VII. Influence of Deformations on Van Der Waals Forces

Van der Waals forces (1) are usually computed on non-deformed objects but local deformations are able to significantly increase their value on the mesoscale. Indeed, on the nanoscale the increase of the sphere radius \( r_2 \) induces a reduction of the force whereas on the mesoscale and microscale the increase of the sphere radius \( r_2 \) induces an increase of the force.

A. Coupling principle

Because of its short interaction range, the van der Waals forces are a surface force. In this model, we chose to replace this local force by an equivalent external load which induces a deformation. This deformation increases the contact surface and then the global van der Waals forces too. This coupled problem can be seen as an algorithm that sequentially uses two models (figure 11). The first one computes van der Waals forces according to the object shape. The other one computes deformation shape according to an external load. An iterative calculation is able to converge to the physical equilibrium.

B. Results

The coupling between van der Waals force and the local deformation can be illustrated on a contact between a sphere and a plane. The local deformation can be calculated by the Hertz deformation model, which enables the calculation of the radius \( a \) of the contact surface in function of an external force. In order to calculate the van der Waals force on the deformed sphere, we consider a simplified geometry. Indeed, we assume that the deformed sphere is a truncated sphere where the radius of the removed spherical hat is \( a \). It is then possible to calculate the van der Waals forces between the truncated sphere and the plane according to \( a \) (details of the calculation can be found in [44]).

\[
R_e = \frac{2E^2z_0}{(1-\nu^2)A_{12}},
\]

(11)

Fig. 11. Algorithm proposed for calculating the adhesion force \( F_{vdw} \) between two objects using the coupling between deformation and van der Waals forces. The algorithm starts with non-deformed objects so the initial contact radius \( a_0 \) is set to zero. The van der Waals forces \( F_{vdw} \) can be computed as the sum of a deformable dependent part \( F_{defo} \) and a non deformable one \( F_{vdw} \) [44]. The deformation model based on the Hertz theory is able to provide the radius of the contact surface \( a_{n+1} \) induced by \( F_{N} \).

The optimal radius \( r_1 \) which represents the frontier between both asymptotic behaviors is around the frontier (typ. 100 nm) between the nanoscale and the mesoscale. This example also shows behavioral differences between different scales and some specificities of the mesoscale compared to the nanoscale. Indeed, on the nanoscale the increase of the sphere radius \( r_2 \) induces a reduction of the force whereas on the mesoscale and microscale the increase of the sphere radius \( r_2 \) induces an increase of the force.

Fig. 10. Structured gripper by PS particles of 1 \( \mu m \): Joint work between EMPA institute, Thun, Switzerland, and FEMTO-ST institute, Besançon, France [40].
where $E$ and $\nu$ are the mechanical characteristics of the objects. In the case of glass objects, $R_c = 0.5 \, \mu\text{m}$. If the sphere radius is smaller than this critical radius $R_c$, the computed force tends to be the classical van der Waals forces presented in eq. (1). So, on the nanoscale, the influence of deformation on van der Waals forces becomes negligible.

The influence of deformations on the van der Waals force has only to be considered on the meso and micro-scales. We have shown in [44] that on the mesoscale, for a sphere radius $r_2 \gg R_c$ the adhesion force tends to be:

$$F_{dvdw} = -\frac{A^3 r_2^2}{48 \sigma_0 E^{z/2}}.$$  \hspace{1cm} (12)

The difference between both force models increases with the radius of the sphere.

The coupling between the local deformation and the van der Waals force is a function of the scale considered (figure 13). Indeed, on the nanoscale, the coupling is negligible and the van der Waals force is proportional to the radius $r_2$ of the sphere (1). On the microscale, the coupling is significant and the van der Waals force on the deformed geometry is proportional to the square of the sphere radius $r_2$ (see asymptotic behavior on figure 13 and equation (12)). Mesoscale is the transition between both asymptotic cases where the coupling should be determined with numeric calculations.

VIII. DISCUSSIONS

This analysis illustrates the wide range from 1 $\text{nm}$ to 1 $\text{mm}$ of micro-nano-scales which includes 6 orders of magnitude with high differences. Each order of magnitude has its own characteristics and consequently its own scientific problems. In order to differentiate each of them, we propose to use additional terms before micro and nano prefixes (see in figure 14). On the microscales, three different levels can be defined: iso-microworld (up to 10 $\mu\text{m}$), deca-microworld (for the medium scale) and hecto-microworld (down to 100 $\mu\text{m}$). These notations could be a solution to clearly classify scientific and technological works in function of their scale of validity.

CONCLUSION

Manipulation and assembly on the micrometer scale (mesoscale) appear to be a high challenge for the future integration of nanotechnology in materials or systems. The micrometer seems to be a critical size characterized by couplings between several physical effects. Four features of physics on the mesoscale were highlighted in this paper: (i) electrostatic forces disturb micromanipulation whereas it seems to be negligible on the nanoscale, (ii) surface functionalisation enables interaction forces to be significantly modified, (iii) the impact of the roughness is different on the meso and the nanoscale and (iv) the mechanical deformations strongly increase the van der Waals force only on the microscale and the mesoscale. This paper illustrates the paradigm of the mesoscale placed between micro and nanoscales.

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Sébastien Alvo received the M.S. degree and the Ph.D. degree in robotics and mechanical engineering from both University Pierre et Marie Curie, Paris, and University of Franche-Comté, Besanon, in 2008 and 2012, respectively. He is currently post doc at the Photonic and Nanostructures Laboratory, Marcussis, France. His research interests are related to force sensing and modeling at the microscale and are currently focused on force measurements in SEM.

Jérôme Dejeu received the Ph.D. degree in physical chemistry from the University of Franche-Comté in 2007. He held a Research Scientist position at the FEMTO-ST Institute before joining the faculty of the University Joseph Fourier (Grenoble 1) where is Assistant Professor of Molecular Chemistry Department. His research interest includes the physical-chemistry phenomena at the interface as interaction, adsorption, and the comparison to the experimental measurements with the theory by the development of models.

Brahim Tamadazte holds a Ph.D. in Automation and Computer Science from the "Université Franche-Comté" in 2005, in France. Before, he has also trained as an engineer in Automation Systems at the Université Mouloud Mammeri de Tizi-Ouzou” from Algeria. During his Ph.D thesis, he has developed different vision algorithms and vision-based control laws (image-based visual servoing and pose-based visual servoing and direct visual servoing) for the automation of MEMS handling and assembly. After a post-doctoral position at the TIMC-IMAG Laboratory (GMCAO team, Grenoble, France), where he is involved in computer-assisted medical interventions (surgical robotics), he recruited as CNRS researcher at the FEMTO-ST Institute (AS2M department, Besanon, France) where he is involved in microrobotics surgery investigations.

Patrick Rougeot is research engineer in the AS2M department of FEMTO-ST institute, France since 2000. He is working in the field of development and control of robotic equipment for 30 years. He works in the field of microrobotics and especially on Micro/Nanoforce measurement (SPECiMeN team). His objectives are developments and exploitations of measurement systems at micro- and nanoscopic scales. His research interests are focused on atomic force microscopes (AFM) for the characterization of the surface effects and on design and developments of tools for micromanipulations. The major activities of his research deals with the adhesion force measurement on structured and/or functionalized surfaces and polymer films.

Stéphane Régnier received his PhD degree in Mechanics and Robotics from the University of Pierre and Marie Curie, Paris, France in 1996. He is currently Professor at the Institute of Intelligent Systems and Robotics (ISIR), University of Pierre and Marie Curie, Paris, France. He is head of the micromanipulation team of ISIR since 2001. His research interests are focused to micro and nano manipulation, teleoperation and haptic feedback at the nanoscale, micromechatronics and biological cell characterization.