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Positioning, Handling and Measuring inside a Scanning Electron Microscope

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Abstract
In this paper we present our latest developments in high precision positioning and handling systems operating inside Scanning Electron Microscopes (SEM). This work is motivated by the growing need of new instruments for the in-situ analysis of materials. In the medium terms, these instruments will permit more laboratories to accomplish complex and repetitive tests on several samples and with various tools, without the need to open the SME chamber, increasing thus productivity and improving the reliability of the analysis.

The concept of a complete manipulator system, working inside a SEM chamber (Lab-In-SEM: LIS) is proposed. Then the performances of a miniature-indenter -scratch (Miniature-indenter -scratch for SEM chamber: MISS) are described and compared with the commercial instrument MTS-XP. Examples of nano-indentation and scratch operations will be described. Then we present a five degrees-of-freedom manipulator (Ztilt-5). The Ztilt-5 has a resolution of 100nm, a workspace of several cubic centimeters and can lift weight up to 300 g. Its geometrical model has been implemented on a PC-based controller allowing telemanipulation, and joint or task spaces control. The Ztilt-5 is powered with a new type of piezo-actuators.

1 Introduction

The emerging techniques used for the fabrication of microscopic mechanical parts, eg. LIGA, micro-stereolithography, micro- electrodischarge machining, electron beam induced deposition, etc., require new, advanced systems for their characterization (mechanical and chemical properties, geometries, etc.) [1, 2]. There are at the moment no standard methods for nanometer-scale engineering techniques, especially for nano-mechanical testing. The properties of nano-materials greatly depend on processing conditions and generally will differ from those of macro-materials. The existing instruments do not offer the required ranges in terms of positioning and force resolution and/or workspace [3, 4, 5]. Often their operation is not easy for non-expert and they do not offer enough flexibility to perform all necessary tests. Therefore dedicated instruments have to be developed to respond to this new demand. Our laboratory at EPFL, in cooperation with several European partners, is developing new advanced handling systems to make such tests easier and more reliable. Moreover, the systems can operate inside a Scanning Electron Microscope allowing in-situ nano-mechanical testing and therefore opening new possibilities for nano-mechanical testing that was not possible to achieve so far [6].

2 Lab-In-SEM: LIS

A concept of a complete manipulator system, working inside a SEM chamber, is proposed (Fig. 1). The system must adapt to various applications, thus be easy to reconfigure. It is essentially composed of four parts:

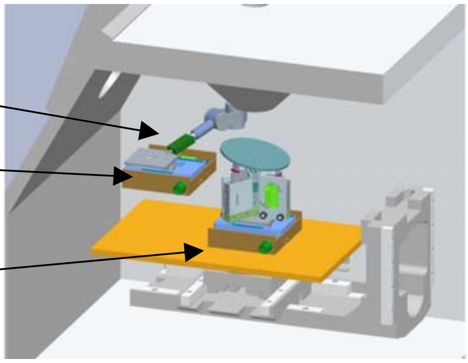
- Tasks manipulation device (TMD):
high accurate positioning in 1 to 6 DoF:
its tasks are: nano-scratch, nano-indentation, samples imaging, cells handling, micro-parts assembly, etc. These tasks are accomplished in the field of view of the SEM, in a relatively small work volume (typically 1 mm^3). The position feedback is given by the SEM image for very high accuracy (few nm). Internal position sensors are not necessary, however, they can improve the dynamic performances of the system and facilitate the manipulator control. The TMD is task dedicated.

- Samples manipulation device (SMD):
long range fine positioning in 2 to 3 DoF:
the samples, the tools and the micro-parts are put on an array to be pick-up by the conveyor manipulation device (CMD) to the tasks manipulation device (TMD).
The SMD brings the micro-objects in the filed of view of a CCD camera or of the SEM in order to be picked-up by the CMD. This operation requires alignment accuracy in the micron range between the objects and the gripper of the CMD. This operation is greatly simplified if the SMD is equipped with internal position sensors (the object being hided by the micro-gripper).

- Conveyor manipulation device (CMD):
long range, coarse positioning in 3 to 6 DoF:
its tasks are: samples and/or tools handling, micro-parts and cells handling, etc. CMS does not require very high accuracy (few microns) but large working volume (several cm^3) to be able to move the samples and the tools between the TMD and the SMD in the SEM chamber. It can be done either by a mobile platform equipped by a manipulator or by a fixed manipulator

(eg. fixed to the SEM column). The coarse position can be given by CDD cameras with an accuracy of a few tens microns. The fine positioning measurement (better than one micron) is provided by the SEM image.

- Centralized controller:
The complete system can have up to 15 or even more degrees-of-freedom, various sensors (force sensors, position sensors, CCD cameras or SEM images). Therefore a complex controller is required. In our case we are using a PC-based controller with a real time operating system developed in cooperation with MoveIt Automation SA [13]. This solution offer a maximum of flexibility and the system can evolve as new elements are added. Complex kinematics models have been implemented. The system can operate either in closed loop or in telemanipulation. A basic user interface has been implemented and is continuously adapted and improved as the requirements evolve.



CMD: Conveyor Manipulation Device:
(eg. Kleindieck GmbH product; MM3)

SMD: Samples Manipulation Device:
(eg. X-Y stage actuated by stick-slip actuators, EPFL)

TMD: Task Manipulation Device:
(eg. Ztilt-5, description in paragraph 4, EPFL)

Fig. 1: Proposed set-up for a Lab-In-SEM.

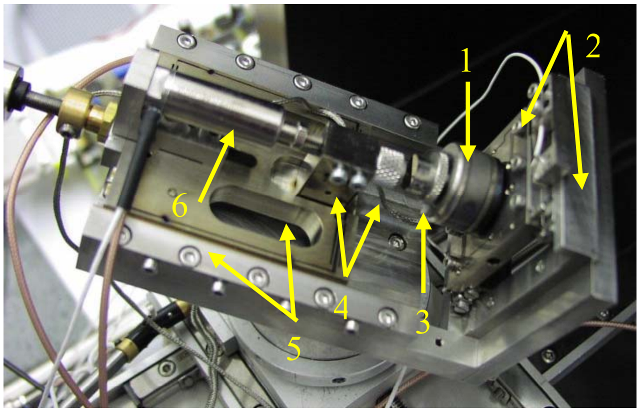
The proposed system allows making various tests on several samples using different tools, without opening the SEM chamber. A better reliability and a higher productivity are thus expected.
The main application fields are for in-situ nano-mechanical testing (nano-scratch, nano-indentation, EDX, nano-surface structuring or application in biology (3d imagery, …).

3 Miniature-indenter and -scratch for SEM chamber: MISS

3.1 Device description

The prototype was designed to realize in-situ Micro-indentation -scratch inside a SEM (Fig. 2). The indentation head (4) is composed by a parallel mechanism (flexure hinges) that holds the diamond tip. The head is driven by a stack piezo (6) with 20 µm range and build in displacement sensor (strain gauge). The indentation head and its actuator are assembled on a macro-positioning stage (5) (also a flexure mechanism) and driven by a fine pitch precision screw, remotely controlled with a cable connected to a knob installed on the SEM chamber's door. The stage is fixed on the main body by a dovetail sliding bearing. This mechanism allows testing samples with diverse heights using different load cells (with various sizes).
A X-Y stage (2) holds the load cell (1) and the sample (3). A stick-slip actuator (see paragraph 4.1) drives the X-axis that is guided by a set of linear bearings. The range is 10 mm and the

nominal step size is 200 nm. This axis can also be used to scratch the sample with maximum driving force of 200 mN. The Y-axis also has non-motorized 10 mm coarse range and a fine adjustment with a stack piezo (not visible on the picture). This allows scratching the sample in the Y direction on 20 μm range, with high forces and high resolution.



1 Load cell
2 X-Y positioning stage
3 Sample holder
4 Indenter head
5 macropositioning table / dovetail
6 Piezo actuator

Fig. 2: Miniature-indenter and -scratch for SEM chamber: MISS, main components.

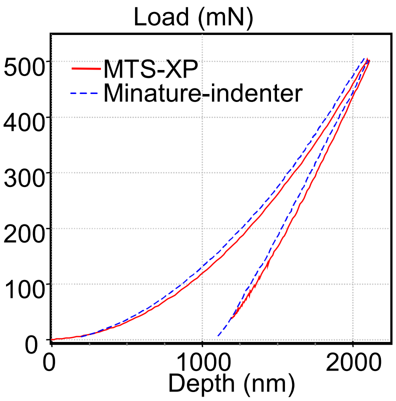
3.2 Operation

Samples up to 10x10x10 mm^3 can be tested. Depending on the force involved different load cells ranges can be used. For high loads a miniature load cell with maximum range 10 N (minimum detectable force around 3 mN) is used. For better resolution a 500 mN range load cell is available. The operator realizes all the setup manually and using the SEM image as a feedback system. The manual tip approach using the knob is a delicate operation but not difficult since the SEM can provide a good and precise visual feedback. Once the point to be indented is chosen, the indentation starts following the input parameters like indentation velocity, maximum depth or load, holding time and unloading velocity [15]. A real time force versus displacement curve (FxD) is generated given the opportunity for the operator to correlate any changes on the FxD curve and the SEM image. For scratching, the operation is similar but there is no measurement system for the lateral force neither for the scratch velocity. So far, it is used for qualitative purpose since it is possible to control only the normal force/indentation depth.

3.3 Validation

To validate the results two samples (Quartz – E = 72 GPa and Sapphire - E=420 Gpa) were measured using a commercial Nanoindenter MTS-XP (the reference) and the Miniature-indenter. For calibration purposes the same indentation tip was used in both indenters. Since its tip area function is known, the unique missing parameter for the Miniature-indenter calibration was its stiffness. So the first output of these measurements was its effective stiffness. This parameter was later used for adjusting the data points and consequently the FxD curve, providing a direct comparison between the curves obtained by reference instrument (The MTS XP nanoindenter) and the prototype. The tests showed that the

Miniature-indenter structure has a stiffness of 480 mN/µm. Additional experiments pointed out that the main source of compliance is first the load cell, followed by the X-stage (that is assembled over a set of linear bearings). To accurately determine the real stiffness is very important. For instance a 5% error in instrument's stiffness can bias the measured Young modulus by 10% for Quartz and up to 30% in Sapphire (it is more critical as the Young modulus increases). The graph below (Fig. 3) shows for material with low Young modulus the good correlation between the curves obtained by the reference instrument and by the Miniature-indenter (after calibration). Although initially was found a good correlation it is still necessary to realize more measurements (more loads, materials) to confirm those values.



Sample: Quartz (E=72 GPa)

Tip: Berkovich

$E_{\text{Miniature-indenter}}$: 70.6 Gpa

$E_{\text{MTS-XP}}$ 71.9 Gpa

Fig. 3: Comparison between results from the reference instrument and the prototype.

3.4 Conclusions

The Miniature-indenter -scratch device allows the operator to precisely choose a specific point for indenting and observe the indentation with SEM image and graph output at the same time (Fig 4).

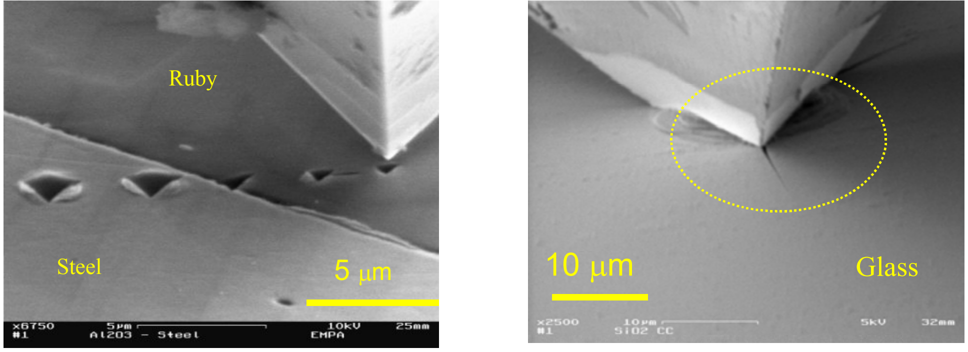


Fig. 4: Left: indentation with constant load on a sample composed of two different materials (steel and Ruby). Right: indentation in glass.

This can help to understand some singularities on the FxD graph like drop in loads/displacement, plateaus, pop-ins, etc. and as a result a better comprehension of the analyzed material. The tests showed the necessity to improve some of its position systems for providing a better usability (case of the Y-axis) and the improvement/replacement of some sub-systems (like the linear bearing, load cell), to enhance stiffness, providing accurate results when testing material from low to high Young's modulus.

4 Five degrees-of-freedom manipulator: Ztilt-5

The Ztilt-5 is a 5 degrees-of-freedom positioning system with 3 translations (x-y-z) and two rotations (θx-θy). It is equipped with embedded position sensor with 100 nm resolution and is actuated by 5 stick-slip piezo-actuators. The Ztilt-5 is compatible with SME environment. It has been developed as a tasks manipulation device (TMD, see paragraph 2).

4.1 Stick-slip piezo-actuators

Stick-slip piezo-actuators are extremely simple and can be made compact. They have been extensively described in several publications [7-12]. The operation principle is as follow:
An inertial mass (slider) is supported and guided by deformable legs (Fig.5). Two modes of operation are defined, viz. stepping- and scanning-modes.
In the stepping-mode each step consists on a slow deformation of the legs followed by an abrupt jump backward. During the slow deformation the mass follows the legs because of friction (stick), whereas it cannot follow the sudden jump because of its inertia (slip). The stepping-mode allows long displacements at a relatively high velocity (typically 5 mm/s). The resolution is limited to a step (typically 200-400nm). Once the position is within less than a step distance of the target, the legs are deformed slowly until the final position is reached. In this mode, called scanning-mode, the resolution is a fraction of a step (typically < 5nm).

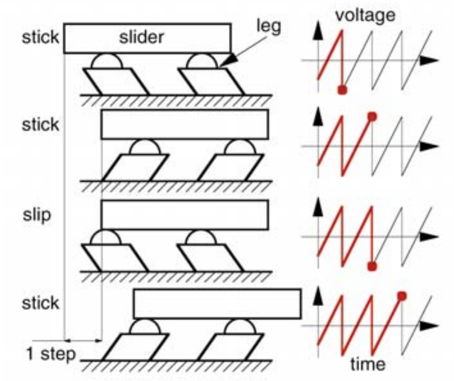


Fig.5: Stick-slip piezo-actuator: operation principle.

To increase the driving force and allow vertical motion, a preload between the slider and the legs is necessary. A common solution is to use permanent magnets [8]. In order to make the system SEM compatible and to increase the driving force, we propose in this paper a preload obtained with a spring system as described in the next paragraph.

4.2 High thrust force linear actuator (HiFla)

The HiFla has three legs, composed of piezo-actuators working in shear-mode. Two legs are rigidly fixed, while the third one is mounted on a compliant mechanism (leaf-spring) acting as the preload system. A commercial linear encoder is embedded in the housing. It has a grating pitch of 20 µm. A 50 folds interpolator gives a final resolution of 100 nm. If the SEM image is used as position feedback sensor, one can expect a final resolution of the device as good as 5nm or even 1nm, depending on the SEM image quality.
This new actuator can be integrated as a standard motor in various manipulators with several degrees of freedom.

Main characteristics:

- maximal thrust force:	1.6 N
- travel:	8 mm
- maximal velocity:	5 mm/s
- overall dimensions:	23 x 25 x 8 mm^3

(the size is essentially given by the embedded optical incremental sensor).

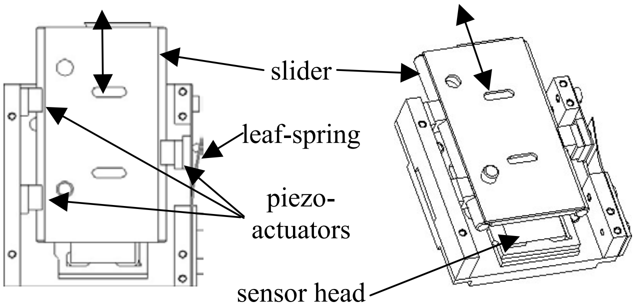


Fig. 6: Schematic view of the High thrust force linear actuator (HiFla) (without the cover plate).

Figure 7 right shows the vertical upward velocity at 5 kHz in relation with the preload when the actuator is loaded with 25, 50 and 100 g.

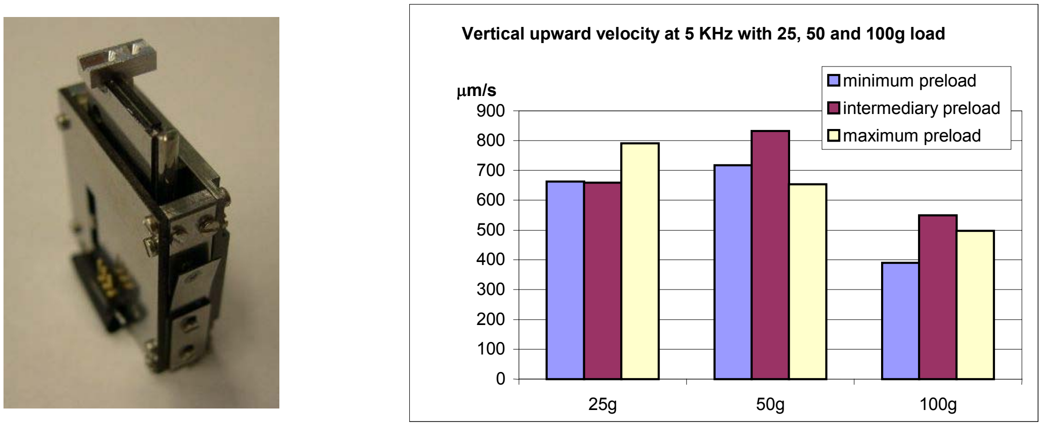


Fig. 7: Picture of a HiFla (left). Vertical upward velocity at 5 kHz (right).

4.3 Z-tilt5 manipulator

The HiFla have been integrated into a five degrees-of-freedom manipulator called Ztilt-5 (Fig. 8 right). It is composed of a X-Y stage (Fig. 8 left) and a parallel kinematics with three degrees-of-freedom (Z, θx, θy).

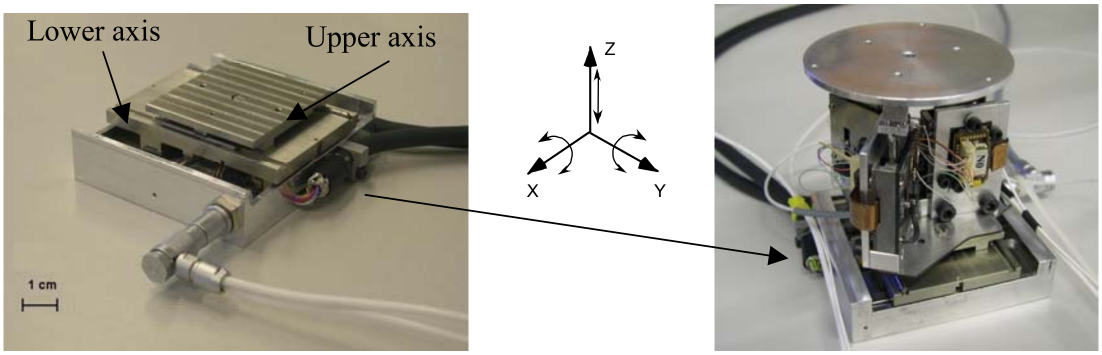


Fig. 8: Pictures of the X-Y stage (left) and of the complete Ztil-5 (right).

The main characteristics of the Ztil-5 are:

- resolution (limited by the sensor): 100 nm
- velocity: 5 mm/s
- recommended load on the platform: 300 g
- tilt angle: ±15°
- translations: 20 x 20 x 8 mm^3 (depending on the tilt angle)
- overall dimensions: 56 x 52 x (46-54) mm^3.

4.4 Control

A PC-based real time controller, being developed at EPFL in cooperation with the company MoveIt Automation SA [13] is used to control the Ztilt-5 in tele-manipulation or in automatic modes. The inverse kinematics model of the Ztilt-5 has been implemented. The position and orientation vectors (Cartesian positions and Euler angles, respectively) of the end-effector are given into the task space by the operator. All axis being linearly interpolated, complex trajectories can be generated.

As an illustration of the possibilities of the system a typical operation is described below.

A micro object is arbitrarily laid on the Ztilt-5 platform that is working under a microscope (light microscope or SEM). Using a joystick, the operator moves the object to bring it into a desiderated position (X-Y) and in focus (Z). Then a specific feature of the object is selected. The Ztil-5 can then be automatically controlled in order to keep the object's feature immobile while the platform is tilting. Such procedures are typically useful for pin-in-hole operations or to make three-dimensional imagery.

5 Conclusions

In this paper we have described nano-positioning and nano-handling systems that are SEM compatible. We have presented a concept of a complete system to be placed inside a SEM chamber to accomplish various experiments either automatically or in teleoparation mode. It is believed that such instruments will be a technological breakthrough in the near future for nano-material testing and will open new possibilities to investigate the fundamental phenomena at the nano-scale.

The proposed concept is not limited to nano-material testing, but can also be employed for micro-assembly tasks or micro-machining. We are currently investigating carbon nanotubes assembly, electron-beam induced deposition, 3d-structuring by proton-beam and micro-milling.

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