Smallest microhouse in the world, assembled on the facet of an optical fiber by origami and welded in the µRobotex nanofactory

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Smallest microhouse in the world, assembled on the facet of an optical fiber by origami and welded in the μRobotex nanofactory

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In this study, the authors have demonstrated that it is possible to realize several three-dimensional (3D) micro- and nanostructures, by the fabrication of the smallest microhouse using a dual beam scanning electron microscope (SEM)/focused ion beam (FIB) Auriga 60 from Zeiss together with a six degree of freedom robot built with SmarAct components. In this new type of nanolab, cutting, etching, folding, assembling, and then welding thin membranes of silica on top of a cleaved optical fiber SMF28, or production of micro- and nanostructures, like the microhouse, are possible. The authors have experimentally shown that FIB can be used, in this new generation of micro/nanofactory, in combination with SEM, and gas injection system, in order to fabricate three-dimensional microstructures: a microhouse in this study, with ultrahigh accuracy assembly down to 10 nm. By using the theory of sputtering, the authors are able to propose a model of folding thin membranes of numerous materials such as metals, polymers, or crystals, i.e., silica, silicon, potassium tantalite, or lithium niobate. This method is usually described as origami in the literature [W. J. Aroa, H. I. Smith, and G. Barbastathis, Microelectron. Eng. 84, 1454 (2007); W. J. Aroa et al., J. Vac. Sci. Technol., B 25, 2184 (2007); and K. Chalapat et al., Adv. Mater. 25, 91 (2013)]. The experimental results indicate that the introduction of a microrobot inside the SEM vacuum chamber will provide the means to enlarge the scope of clean room facilities to build complex and smart 3D microsystems with heterogeneous materials, especially on the facet of an optical fiber in the lab on fiber new field. The authors propose a new way to easily manufacture many kinds of optical functions for light trapping based on nanoantennas, nanophotonic crystal, axicon or lattice, 3D biosensor with origami, and nanopatterning surfaces or carbon nanotubes, etc. Published by the AVS. https://doi.org/10.1116/1.5020128

I. INTRODUCTION

The micro- and nanofactory in dual beam SEM/focused ion beam (FIB)/gas injection system (GIS) is a new way to increase the field of clean room processes, especially with the introduction of several robots inside the chamber. By this new way, it is now possible to assemble several crystals as thin membranes in order to build new generations of real three-dimensional structures. The recent development of FIB process in three different types of action is a powerful way to pattern the different elements before assembly. The first patterning mode is high-powered membranes cutting mode with a high power of gallium beam, in the etching mode. With the combination of high current, from 200 pA to 20 nA, and xenon fluor, XeF₂, gas from the GIS, it is possible to etch very deep structures of more than hundreds of micrometers. The second way to pattern the structures leads to a process from 2003 by Origami.1–3 With a medium power of the gallium beam, from 50 to 100 pA, and with a specific process, it is possible to fold some crystals, metals, or polymers and some membranes in order to obtain self-folding of the membranes as patterning surfaces.4,5 Membranes for 3D structures could be processed in a clean room by lithography or directly patterning by FIB in the same vacuum chamber as the assembly process. The third way to use the gallium beam corresponds to the ion beam assisted deposition (IBAD) process. In this process, with a low current of the FIB beam from 5 to 20 pA, and in combination with one gas from the GIS, carbon, silica, or platinum, injected just near the bombarded surface, it is possible to realize the growing of a thin film in order to link, stick, or weld two different membranes or surfaces one with another. The step of assembly is carried out with the robotic six degrees of freedom arm before welding. Origami or micro-origami structures are also realized in atmospheric conditions in order to help robotic tasks such as microassembly, clinical microsurgery, cell manipulations, and biomedical engineering research.7,8

We can find many papers related to origami method, in SEM/FIB station, corresponding to the creation of three-dimensional microstructures.1–3 The authors discussed the fold radius as a function of ion implantation of gallium on a silicon nitride cantilever,1–4 the thickness of the cantilever, and the stress resulting of the ion dose. Others talk about tensile stress in the case of self-folding chromium layers on a silicon nitride cantilever. They also built a square nanobox of 800 nm in Ti/Al/Cr, just by self-folding but without

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linking the different faces at the end of the process. All these papers aim to study the self-folding origami process by Ga\(^+\) ion implantation. In the 2000s, many authors tried to build super structures by electron beam lithography (EBL) of FIB milling in order to enhance the properties of materials or metamaterials, but they were often limited by the manipulations or the characterizations at small scales under vacuum or in atmosphere. Other authors have fabricated nanobirds with plasmene nanoribbon of bimetallic gold and silver nanocubes. The thickness of their plasmene nanoribbons is in nanometer, but the scale of the birds is in millimeter. They used origami process by FIB milling to self-fold their 3D structures. Other origami structures in SEM were realized in CMI EPFL, in order to produce a “silicon nanoinsect hotel,” or polyimide foils corset, but the structures were realized without robotic assembly. One of the most important and common properties of the process of origami observed in bibliography is the end step of the self-folding process, when the cantilever is at 90°, that is, parallel to the ion gallium beam. In our study, we observe the same properties: when the walls are at 90° between the base of the house and the rest of the membrane, it blocks the ion beam and folding stops. However, in our study, we observe the same origami and self-folding process, but it does not depend on the ion implantation dose or on the tensile stress of the membrane. The recent development in nanorobotic, nanoptic, nanoprobe, or nanoantenna combines the properties of optical fibers in interaction with another nanosystem like bowtie nanoantenna, campanile near-field probes on the facet of an optical fiber. EBL and ultraviolet nanoimprint lithography (UV-NIL) in dual SEM/FIB station are often used to produce the optical nanosystem but the authors, especially the optician researchers, have difficulties to carry out the assembly of the two parts of a system with the right optical precision under vacuum because commercial solutions do not exist. The lab on fiber technologies (LOF) is an emerging field, from 2013, with a lot of applications in multifunctional photonic devices, but the actual systems are produced in clean room with conventional facilities, EBL, UV-NIL, SEM, FIB, without robotic actuators and not under vacuum. The development of the new \(\mu\)Robotex station with robotic SmarAct structure under vacuum will permit to have a reproducible solution to optical nanoassembly, because the SmarAct actuators have a centimetric strokes with a nanometric accuracy, and because, we use the SEM/FIB parameters to pilot the robotic arm. The new generation of nanofactory like \(\mu\)Robotex will become a very important way to produce optical nanosystem in the future, especially in the new lab-on fibers field. In this study, we show that the \(\mu\)Robotex nanofactory is able to assemble each type of integrated components such as mirrors, filters, photonic crystal, hexagon of less than 1 \(\mu\)m, on top of an optical stretched fiber, on the tip of an optical fiber, or on the facet of a cleaved fiber. The assembly properties correspond to all the geometrics parameters, angles, distances between the two faces, positioning between the center of the optical fiber, with an accuracy from 2 to 10 nm. In the \(\mu\)Robotex nanofactory, it is also possible to realize geometric assembly with the SmarAct robotic closed loop system with or without active optical way. Active optical way means that it is possible, under vacuum, to control the moving step of the robot in function of the properties of the transmitted light, losses, and phases.

The \(\mu\)Robotex station is a very powerful tool, especially for the LOF technologies due to the very high accuracy of the assembly and to the robotic solutions, which is necessary to control the position and the orientation of the center tool. The high accuracy of the robotic arm is necessary to set up the different components on the facet of the fiber, exactly at the optical center of the fiber, and exactly at the right position, or angle. Figure 1 shows a mirror of \(25 \times 25 \mu\)m pattern in a thin silica membrane of 1.2 \(\mu\)m, assembly on top of an optical fiber. We also have realized the assembly of a hexagon of \(760 \times 40\) nm of an optical crystal on top of a tip, grown by optopolymerization on the facet of an optical SMF28 fiber. The big vacuum chamber, the high stability of all the systems, and the low level of noise measured by the infrared method give exceptional conditions for all types of assemblies, especially in all optical fields because the accuracy is well below the wavelength of 1550 nm. The \(\mu\)Robotex station is a very powerful nanofactory for the LOF field. In this field, we often install the optical fiber on the arm of the robot, in order to place the z-axis of the fiber parallel to the z-axis of the FIB. In this position, it means that the facet of the fiber is exactly in the \((x,y)\) plane of the FIB containing the eccentric point. Then, we install the membrane, microhouse, the mirror, or the optical filter (Fig. 1), the pyramid, on the stage of the SEM, on a substrate holder with an angle of 54° so that to have the surface of the object to be assembled in the same \((x,y)\) plane of the FIB.

II. EXPERIMENT

A. Folding thin layers or thin membranes by origami

In this study, we have used silica as a substrate membrane for building the microhouse: base, walls, roofs. We have used a specific process of self-folding of the silica membrane in order to change the orientation of the walls from 0° to 90°, and each side of the roof at 35° ± 2°. The most
commonly used name for this process is origami. The parameters of this process are described in Table 1 for several materials. In this study, the membrane is a silica membrane of 1.2 μm thick covered on the upper face by a thin layer of 40–50 nm of chromium. When we fold the wall, we begin by etching the thin chromium layer in order to remove it.

B. Theory of the self-folding process by origami

Then, with the ion gallium gun, we etch the silica of the membrane, and after a short time, when the thickness of the etching area is thin enough (Fig. 2), the self-folding process starts. In all our tests, in all conditions, we observe that the self-folding process always starts for the same thickness of the silica membrane [Figs. 3(a)–3(d)]. If we change the materials to fold, then the thickness of the self-folding is different but always the same for the new materials, such as silicron, lithium, niobate, or alumina. The specific volume of self-folding corresponds to the etching area as a sputtering bulk. As it was explained in Fig. 2(a), by the sputtering mechanism of the ion beam, there is a transfer of momentum between the gallium ions and the atoms of the thinner etch membrane [Fig. 2(b)]. In fact, in the bulk of the last link between the two parts of the membrane, we can find two different areas: the upper value in white in Figs. 2(b)–2(d), the cold one, in which the gallium atoms pass across with the high speed. We also find the rear part in which the gallium atoms are reflected, in red in Figs. 2(b)–2(d), the hottest. The temperature in the upper area is lower than the temperature in the bottom one because the path of the gallium ions is longer and they change their direction of propagation. The gallium ions are reflected in the bottom part of the etching area. So, when the thickness of the membrane corresponds to the maximum of the thermal dissipation of all the gallium ions, which were all reflected, it appears in this small bulk, or areas in Figs. 2(c) and 2(d), a bimetallic strip with the hottest zone in the down part and the coldest one in the upper.

The properties of the bimetallic strip with the differential dilatation coefficient between the hottest and the coldest areas give rise to the self-folding of the moving part of the membrane, always in the direction of the top part [Figs. 3(a) and 3(b)]. It means that even if we want to cut a little part of the membrane and remove it, it always completes with a small part at 90° from the base [Fig. 3(d)]. By this specific process of self-folding, we are able to produce the real three dimensional micro- and nanosystems, with all types of materials, crystals, metals, semiconductors, polymers, metamaterials, or piezo-electrics. All the structures could be patterned before or after folding in function of the need. In the same vacuum chamber, we can, as in clean room but for smaller devices, in nanotechnologies, use the μRobotex station as a new generation of micro-and nanofactories or nanolab.

Before the real assembly, we have to simulate the result in order to prepare the assembly scene and the assembly tools. Figure 4 represents the conception assisted by computer (CAC) view of the conception step of the microhouse on top of the facet of an optical fiber.

### Table I. Parameters of the origami process for several materials such as silica, lithium niobate, and silicon.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Etching line</th>
<th>Step 1</th>
<th>Step 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Origami of silica</td>
<td>X = 10 μm</td>
<td>T = 600 s</td>
<td>T = 60 s</td>
</tr>
<tr>
<td>of 1.2 μm thick</td>
<td>Y = 1.7 μm</td>
<td>P = 30 kV 2 nA</td>
<td>P = 30 kV 200 pA</td>
</tr>
<tr>
<td>Origami of LiNbO₃ of 1.0 μm thick</td>
<td>X = 20 μm</td>
<td>T = 480 s</td>
<td>T = 45 s</td>
</tr>
<tr>
<td>Y = 1.5 μm</td>
<td>P = 30 kV 2 nA</td>
<td>P = 30 kV 200 pA</td>
<td></td>
</tr>
<tr>
<td>Origami of silicon</td>
<td>X = 25 μm</td>
<td>T = 360 s</td>
<td>T = 30 s</td>
</tr>
<tr>
<td>of 1.5 μm thick</td>
<td>Y = 2.0 μm</td>
<td>P = 30 kV 2 nA</td>
<td>P = 30 kV 200 pA</td>
</tr>
</tbody>
</table>

![Figure 2](image-url) (Color online) Theory of the origami process: (a) The first step of this process corresponds to a sputtering with gallium ions of the interesting area. (b) When the bulk of the link between the membrane and the panel to fold corresponds to a specific thickness, a bimetallic strip appears. (c) The effect of sputtering produces a differential dilatation coefficient into the upper part in white and the down part in red of the link. (d) The panel self-fold from 0° to 90°.
Zeiss. The Auriga 60 has a big vacuum chamber of 60 \( \times \) 60 \( \times \) 60 cm\(^3\), on the top of the chamber, and a SEM field emission gun column is installed with two detectors for retro diffused electrons: in lens and the energy selective backscattered one. The third electron detector corresponds to the secondary electrons, SE2 detector. The FIB column is produced by Orsay-Physics, and this column is positioned at an angle of 54\(^\circ\) from the z-axis of the SEM column. In this paper, we will always use the same GIS with three gases: xenon fluor for etching processes, naphthalene and cyclopentadienyl-platinum for vapor deposition processes, respectively, and carbon and platinum by IBAD.\(^6\) The GIS is produced by Oxford.
Instrument and the type is OmniGis II, in the family of Omniprobe. We can also work in electron beam assisted deposition mode with a better uniformity but for thinner films.

**B. Robotic under vacuum**

The robotic system is built with six individual actuators from SmarAct. The robotic structure is home assembled, and all the robotic arms are controlled in order to obtain a very high accuracy on the object position. The moving steps are controlled with a human/machine/interface (HMI), and the softwares are home made in order to combine the high accuracy of positioning and synchronization of all axes in the assembly processes [Fig. 5(a)]. The control of the robotic system is based on the kinematic model (KM) so as to calculate the value of the position of the object of interest in function of the value of the position of each axes. The parameters of each axis are used to build a CAC representation of the assembly scene in order to control the position of the elements under vacuum because it is impossible to see the elements in the chamber [Fig. 5(a)]. These structures are characterized by the possibility of decoupling the kinematic model in a complete geometric model calibrated in order to obtain information about the position of the assembled elements. The solution of this model, depending on a solving solution by iteration, is given whatever be the values of the geometric parameters, angles, and distances between successive axes, which are not conditioning by the structure. All the axes are synchronized every 500 ms in position and in acceleration in order to pilot with a high accuracy of the system. The axes are stick/slip type from SmarAct company. The combination of a high accuracy of the axis under vacuum and the SEM parameters for piloting the geometric model offer a very important scientific result in this study. One of the most important things in this type of control in the closed loop of the robot corresponds to the three ways to move the center tools of the robot. It is possible, by using the kinematic model [Figs. 5(a) and 5(b)], to control the robot in three different repairs: the repair \((x,y,z)\) of the SEM, the repair \((x_0,y_0,z_0)\) of the FIB, or the native repair of each axis of the robot.

**C. Substrate preparation: The fiber**

The substrate holder for the realization of the microhouse is an optical fiber, SMF28, which is cleaved after stripping the sheath. The fiber is then installed on a special tool of acrylate realized by 3D printing. A PVD process of the chromium layer coats all the systems, tools, and fibers. We sputter a thin film of less than 40 nm of chromium to pattern and observe the object in the SEM conditions. After deposition, the acrylate tool and the fiber are installed on the robotic arm inside the SEM vacuum chamber. The axis of the optical fiber is installed in the same axis than the \(z\)-axis of the FIB.
In this position, the top surface of the cleaved optical fiber is located in the (x,y) focalization plane of the FIB containing the eucentric point. The chromium layer is etched in the center of the fiber so as to let the optical signals lighting out of the fiber in working conditions. Four platinum plots of 1.4 μm were deposited, on the as-patterned area, at the four corners of the microhouse, in the center of the fiber.

D. Substrate preparation: The membrane for patterning the microhouse

The microhouse is made of a silica membrane of 1.2 μm thick, which is obtained by wet etching in potassium hydroxide of a silicon-on-insulator wafer in a clean room. We etch a square area of 200 × 200 μm of silicon on both faces of the wafer, and then the silica membrane is coated with less than 50 nm of chromium. After this step of preparation of the facet of the optical fiber, the fiber is retracted along the z-axis of the FIB in order to let the place to the membrane. The membrane is then installed inside the vacuum chamber on the stage of the microscope, at an angle of 54° in order to be patterned by FIB, in the (x,y) plane of the FIB [Fig. 6(b)]. Before assembly, the first action is to install the membrane at the eucentric point of the machine in order to be able to image the assembly scene by both SEM and FIB [Figs. 6(a) and 6(b)]. The gallium beam is used with a high power and a high beam current, 10 and 20 nA, to draw the cutting pattern by etching the membrane. We just kept a little link before folding the four walls and welding the base of the house on top of the optical fiber with GIS, naphthalene gas, and FIB [Fig. 7(a)].

E. Origami process to raise the four walls

After cutting the pattern, etching the doors and the windows of the microhouse, the four walls of the house are in the plane of the membrane [Fig. 7(a)]. We need to change the position of the walls by self-folding the membrane. Origami is often used for changing the angle of one part of

Fig. 7. Scale was the same for all the pictures. (a) Test of patterning of a membrane in silica without door and windows. (b) Optimization of the origami process or self-folding with three walls of a box like a house, view from the SEM. (c) The same optimization view by the FIB. (d) Origami of the four walls view by the FIB.

Fig. 8. (a) Origami process of the three walls of the final microhouse. (b) Origami process of the four walls and installation of the fiber in the background. We can see the diameter of the fiber, because the center of the house is installed in the center of the fiber in order to let the light throughout.
the membrane. In this paper, we study and develop a self-folding way [Table I] to install the four walls of the house at an angle of 90° from the original membrane plane [Figs. 7(b)–7(d)]. Figure 3(a) shows the different angles of folding in function of the different conditions of current of the FIB. Figures 7(b), 7(c), and 8(a) show the folding action of the first three walls and without the optical fiber, in the background, because the halls are dark. Figure 8(b) shows the origami at 90° of the four walls, and the installation in the background, just behind the membrane, of the fiber. The robot, with a special end tool, holds the fiber and allows the z-axis of the FIB repair to install the fiber in contact with the membrane, by learning and memorizing the position of the surface of the fiber before welding. This action required a very high accuracy, because if the fiber rises too far, the membrane explodes. The only way to control the position of the fiber relatively to the membrane is to observe through the dark windows which appear after the self-folding of the four walls of the house as it can be observed in Fig. 8(a).

Figure 8(b) also shows the welding points with the FIB and naphthalene gas from the GIS, which is used for linking or sticking the base of the house in contact with the top

![Figure 9](image_url)

**Fig. 9.** (a) After the step of welding of the base of the microhouse on the facet of the optical fiber, we must cut the thin last link between the base of the microhouse and the membrane. During the assembly time and the welding time, the stability of the system is very important. In the case of drift of the robot, the microhouse could be broken. (b) The link is cut, and we can extract the microhouse by the background direction. [(c) and (d)] After patterning and self-folding the first roof at 35°, we install the microhouse from the background like “mortise and tenon.” This step requires a very high accuracy less than 10 nm. (e) After welding the first roof, we install the second roof under the same conditions and we realize the welding step always with naphthalene gas. This step also requires accuracy and stability. (f) After welding the two roofs, we built the chimney. This picture was realized with the ESB detector. The other pictures were always realized with the SE2 detector.
cleaved surface of the fiber. The parameter for this process depends on the size and the thickness of the link to the plot. The first step consist of depositing a square of $5 \times 5 \mu m$ just near the membrane to increase the level of the surface of the plot, which corresponds to the thickness of the membrane, plus the thickness of the thin plots which were under the house, that is, a thickness of $1.2 + 1.4 \mu m = 2.6 \mu m$. Then, we deposit a new link of $7 \times 4 \mu m$ between the membrane and the plot of $2.6 \mu m$ thickness. After the last stick point, we expect that the base and the four walls of the microhouse are definitively stuck on the top of the fiber. Later, we cut the last link between the membrane and the microhouse [Fig. 9(a)]. By this process, we show that the house comes from the membrane, and after microassembly and stick process, it is fixed on top of the optical fiber by the robotic nanoassembly.

F. Ultrahigh accuracy assembly for tenon and mortise

Then, we pattern the two sides of the roof [Figs. 9(c) and 9(d)], the outer ribs and the mortise used for positioning the top of the walls before welding. Again, we use the origami method to get each side of the roof to the right angle of $35^\circ$. This process depends on the control of the beam power of the FIB and the thickness of the etching line. As we can see in Figs. 9(b) and 9(c), it is important to obtain the same angle for both sides of the roof. Again, we install the optical fiber with the base of the microhouse just in front of one side of the roof, by the backside of the membrane without breaking the as-assembled microhouse, linked on the fiber, and the first part of the roof, linked on the membrane. The ultrahigh accuracy of the SmarAct robotic in the HMI system is very useful for this assembly process, in all the (x,y,z) directions. We need 4 to 10 nm of accuracy in the plane of the roof, that is, in the (x,y) plane of the FIB, for tenon and mortise integration, and less than 50 nm in the z-axis of the FIB. For this part of the process, we need a very high accuracy, a few nanometers in combination with a very high stability of all the robotic arms. After assembly, we deposit naphthalene between the top of the wall and the mortise of the roof, starting from the bottom of the roof like tiles. The properties of the naphthalene coating present the advantages to grow into two directions, planar and perpendicular, in order to link the space between the walls and the roof, and to fix the roof onto the walls. This process time varies from 3 to 10 min for each plot, and during this “long” time, we need a perfect stability of the SmarAct actuators. We use the same process for the second part of the roof [Fig. 9(e)] and then, after depositing all the tiles, the house is finished, the chimney is built [Fig. 9(f)] and also because Besançon is just near the mountain of Jura, it snows in winter and it is cold (Fig. 10).

IV. SUMMARY AND CONCLUSIONS

In this study, we demonstrate the abilities of the new generation of micro- and nanofactories or nanolabs, with a lot of peripheral equipment, as FIB, GIS, and microrobotics structures inside the vacuum chamber, in our case SmarAct and Klein dieck. In the μRobotex station, we are able to set up other accessories as laser, ellipsometer, and spectrometer, electrical and optical bulkhead crossing, in order to test, excite, or align many samples in function of piezo answer or optical positioning. We are also able to introduce other gasses for mechanical link and traction test under SEM. By the building of the smallest microhouse in the world, which is in fact not even able to accommodate a mite, we demonstrate the high capability of the μRobotex station, the very high accuracy of the μrobot, and the high flexibility of the station. It is not possible to realize all these actions in a clean room with human accuracy, but now with the new type of big SEM vacuum chamber, with the high accuracy of the new generation of μrobot installed under vacuum and other accessories installed inside the chamber, we can pattern, cut, fold, assemble, weld, and stick many kinds of materials to produce real three dimensional micro- and nanosystems, for many kinds of new applications. We also submit an original theory to understand the folding effect of the thin membrane by the sputtering effect and the transfer of momentum. This new technology is an emergent one, which can be used for producing micro- and nanosystems for the future. In fact, it would have been impossible to realize such micro- and nanosystems without the new μRobotex station. The LOF technologies could be developed in our new nanolab.

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