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SHOP NOTES

These are "how to do it" papers. They should be written and illustrated so that the reader may easily follow whatever instruction or advice is being given.

Enhanced positioning precision and *in situ* macroscopic contacts for shadow-evaporated nanostructures

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The authors present the design of a modular sample holder that offers the possibility of *in situ* fabrication of metallic nanostructures under ultrahigh vacuum. One of the crucial points is to bridge the gap between the macroscopic leads and the nanostructure itself. This problem is solved by using a set of two different masks. For a precise alignment of the two masks, a magnetic tripod connection system has been developed. With this new system, an alignment precision of 26 μm is obtained. As a result of the fabrication in ultrahigh vacuum, the nanostructures will be accessible to scanning probe techniques without surface contamination. First results show that electrical measurements are indeed possible. © *2014 American Vacuum Society*.
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I. INTRODUCTION

The size of the functional units active in computers continuously shrinks in size. Besides the top–down approach used in semiconductor industries, one possible alternative is the use of single molecules as building blocks of electronic functional units.[1] To contact single molecules on a surface, atomically clean contacts with a distance of the size of the molecules, typically a few nanometers, are an important requirement. Today, mainly two methods are used to fabricate clean atomic scale metallic contacts: mechanically controlled break-junction technique[2] and electromigration.[3–5] Both methods need predefined structures with a size from some tens of nanometers to a few micrometers. For such larger than atomic structures, electron beam lithography (EBL) is the state of the art fabrication method. However, it is difficult to preserve the high standards of cleanliness needed for atomic scale precision, since EBL relies on the use of organic chemicals during the fabrication process. It is often argued that the surface can be rinsed after the process and therefore appears to be clean, e.g., in scanning tunneling microscopy (STM) images.[6] However, mobile molecular remnants may not be detected with STM as they only lead to additional noise in the images. We have observed contamination of the sample surface similar to the carbon deposits observed during scanning electron microscopy (SEM) characterization.[7] On the other hand, cleaning by plasma-etching or sputtering is known to modify the chemistry and the structure of the surface and might also damage the sensitive nanostructures.

Thus, structuring the sample using mask evaporation in ultrahigh vacuum (UHV) has recently become an interesting alternative. In this approach, a prestructured mask is either brought to or fabricated close to the clean substrate and the metal is then evaporated onto the assembly. A mask integrated into the sample allows an excellent control of the half shadow but leads to some drawbacks for surface preparation if the mask is sensitive, for example, to heating.[8] This method also causes difficulties for scanning probe observation since the tip has to pass the mask to image the nanostructure. For movable masks, often referred to as stencil lithography,[9–12] the mask is moved close to or pressed onto the sample surface.[13,14] Often nanopositioning systems are used for this task, which offer the possibility to move a mask in the nanometer range. However, combining high precision with macroscopic contacts also remains difficult here. In this work, we introduce a new design of a modular holder system on the basis of a commercial sample holder plate (Omicron, Taunusstein, Germany). With our new design strategy, we are able to take advantage of complete *in situ* preparation with a much simpler design compared to nanopositioning systems. We can apply standard surface cleaning methods, such as direct current heating of the substrate without damaging the deposition masks. In addition, the flexible mask holder system not only allows the deposition of metallic nanostructures and macroscopic contact leads but also the mounting of a four-point contact holder for *in situ* electrical measurements.

II. EXPERIMENTAL SET UP AND METHODOLOGY

A schematic overview of the concept for sample preparation and electrical contacting of nanoscale bridges is given in Fig. 1. For the fabrication of metallic structures on the sample surface we use a two-step deposition process shown schematically in Figs. 1(a) and 1(b). In the first step,

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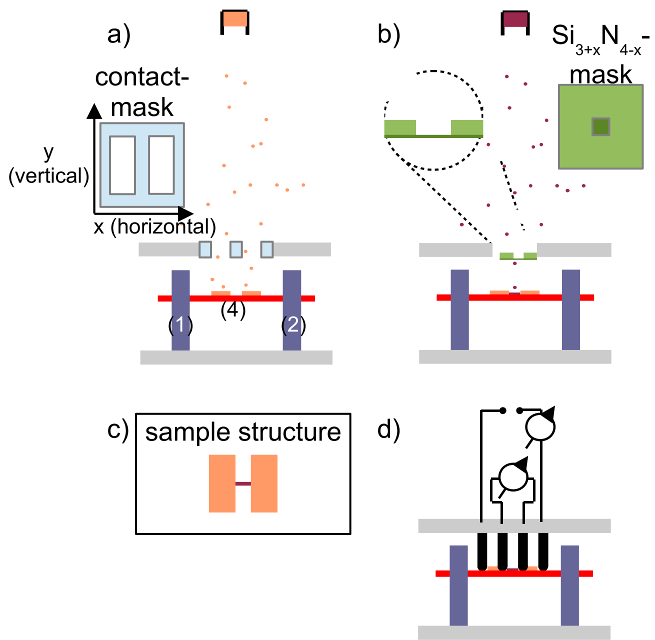

FIG. 1. (Color online) Principle of the modular holder system for which all preparations can be performed in UHV. The sample (4) is attached via posts (1,2) to the sample holder. (a) First mask holder for evaporation of macroscopic contact pads (contact mask). (b) Second mask consisting of a nanostructured $Si_{3+x}N_{4-x}$ membrane. (c) Schematic of the ideal nanoscale bridge structure. (d) Four-point contact for *in situ* electronic measurements.

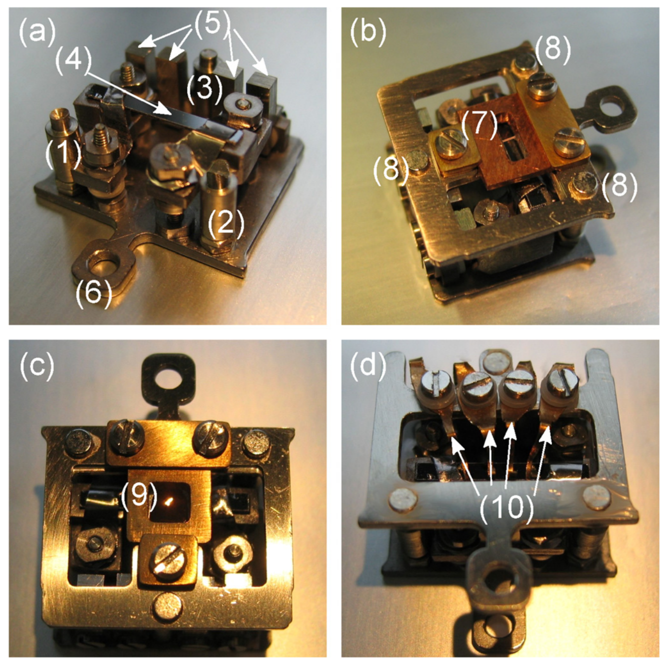

FIG. 2. (Color online) Photo of the modular holders: (a) sample holder alone, (b) sample holder with mask for macroscopic contact pads (first mask, TEM grid), (c) sample holder with nanostructured $Si_{3+x}N_{4-x}$ mask (second mask), (d) four-point contact holder attached to sample holder. (1), (2), and (3) tripod positioning system, (4) sample, (5) intermediate contacts for the four electrical contacts inside the UHV system, (6) manipulation system, (7) macroscopic mask, (8) magnets that attach to the tripod positioning system, (9) nanostructured mask, and (10) four-point contact springs contacting the sample surface.

macroscopic contact pads are made by evaporating metal through a large mask, see Fig. 1(a). Then, this mask mounted on a mask holder is removed *in situ* together with its holder and a second mask holder is mounted in front of the sample. This second mask holder contains a $Si_{3+x}N_{4-x}$ membrane mask used to make nanostructures by shadow evaporation, see Fig. 1(b). The ideal final sample layout is sketched in Fig. 1(c). For such a layout of the sample, it is important that the mask for the $Si_{3+x}N_{4-x}$ nanostructure bridges the contact pads. The dimensions and number of the nanostructures to be written are given by this requirement. Last also the second mask holder is removed *in situ* and a third holder designed for making an electrical connection to the sample surface is attached to the sample holder, see Fig. 1(d). Then, an electrical measurement can be performed.

In such a two-step deposition process, the alignment between both masks is crucial as has also been reported for similar processes.[15] Therefore, we introduce a magnetic tripod connection system inspired by the sample positioning system used for the tip and sample positioning in the low temperature scanning force microscopy described in Ref. 16. This tripod connection system is intended to enhance the positioning precision of the modular sample holder system.

Figure 2 shows a set of photographs of the modular holder system, i.e., of the sample holder, Fig. 2(a); the two mask holders, Figs. 2(b) and 2(c); and the four-point contact holder, Fig. 2(d). Three magnetic steel posts are attached to the sample holder, labeled 1, 2, and 3 in Fig. 2(a) intended to make the connection to the mask and four-point contact holders. In the mask- and four-point contact holders, three NdFeB magnets [(8) in Fig. 2(b)] are integrated. Magnetic steel half spheres are glued onto the side of the magnets oriented toward the sample holder. These three steel half spheres fit into three magnetic steel posts attached on the sample holder. Two of these steel posts are adjustable in height using screws. This height adjustment is used to precisely predetermine the height of the mask relative to the sample in air before introducing the holders into the vacuum system under an optical microscope. See supplementary material for shop drawings of the mask holder and the four-point contact holder.[17]

The magnetic tripod connection system consists of three steel posts with three different supports. It is intended to ensure a unique position of the mask holder even for a nonperfect mask holder with given fabrication tolerances. The layout of the steel posts is sketched in Fig. 3(a). After adjusting the height of the posts, the mask holders have two degrees of freedom for translation and one degree of freedom for rotation. The first support, a conical pit, establishes a center of rotation in the *x*–*y* plane for the mask holders. The second support, a V-shaped slit, fixes the remaining rotational freedom. Finally, the third support is flat and does not influence the before-mentioned degrees of freedom. In Fig. 2(a), the cone shaped post one is labeled 1, the V-shaped one is labeled 2, and the flat post is labeled 3.

In practice, a minimal motion remains possible since the long steel rods have a certain elasticity and therefore deviate

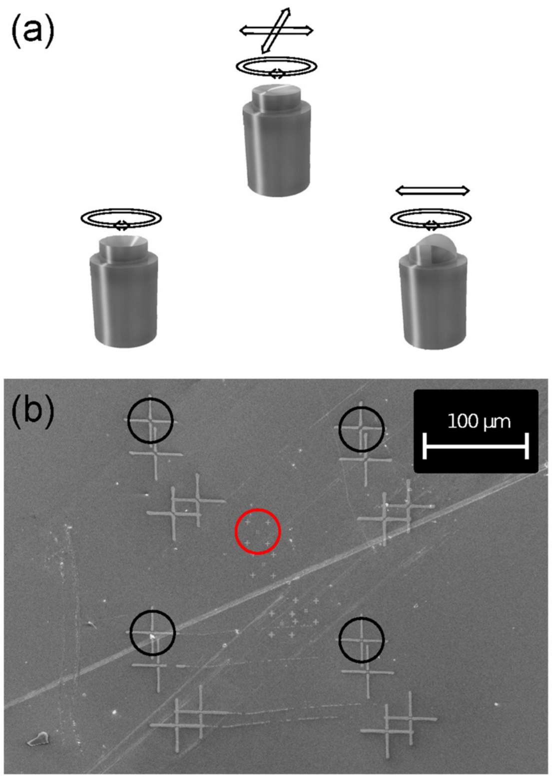

FIG. 3. (Color online) (a) Tripod connection system, which allows for the different degree of freedom in motion. (b) Test of the improving of the positioning accuracy. SEM image of the sample after attaching, covering with metal and removing of the $Si_{3+x}N_{4-x}$ mask for several times. The $Si_{3+x}N_{4-x}$ mask itself consists of four crosslike structures with a width of 50 μm (black circles) and an additional smaller copy of the same crosslike structure in the center (red circle).

from the ideal position perpendicular to the sample holder base plate. This variation is of the order of several micrometers. Indeed, a variation in the direction perpendicular of the nanostructure [in the *y*–(vertical) direction parallel to the gap] is of minor importance.

III. RESULTS

The mask used for making the macroscopic contact pads consists of two rectangles (approximately 2 mm × 4 mm) separated by a thin line (approximately 35 μm wide). This mask was made from a modified TEM-grid (Athene Polygrid G227) by removing some unneeded parts. After metal deposition through this mask two large contact pads are obtained with a gap of 35 μm width.

For the second mask used for nanostructuring, masks were fabricated with a commercial focused ion beam system, 2FEI Strata 400 STEM, on silicon chips that are covered by a 200 nm thick $Si_{3+x}N_{4-x}$. The chips provide a 500 $\times$ 500 μm^2 wide free-standing $Si_{3+x}N_{4-x}$ membrane. To avoid charging and cracking of the silicon–nitride membrane during the writing process, Cu layers were deposited on both sides (50 and 200 nm) of the chips.

A mask with symmetric crosses was used for testing the positioning precision. Therefore, the mask holder was repeatedly detached and reattached to the sample holder in UHV and each time the structure was transferred onto the substrate by metal deposition through the mask. The result after four repetitions is shown in Fig. 3(b). The analysis shows that a horizontal precision (*x*-direction) of 26 $\pm$ 14 μm and a vertical precision (*y*-direction) of 35 $\pm$ 29 μm was achieved. As mentioned above, the vertical precision is not of concern for bridging the contact pads. The horizontal precision, however, is critical. Using our original design up to 48 nanostructures had to be written onto the $Si_{3+x}N_{4-x}$ membrane mask to ensure that one of these nanostructures bridged the 35–μm-wide gap between the macroscopic contact leads.[18] This implies long FIB writing times, which reduce the lateral precision owing to thermal drift. With the new magnetic tripod, we are able to reduce the number of nanostructures by increasing the positioning precision of the $Si_{3+x}N_{4-x}$ membrane mask with respect to the contact pad mask. Only two 70 μm long nanostructures are now necessary to obtain precisely one structure bridging the gap. As a consequence, the FIB writing time is strongly reduced and nanostructures with smooth edges are obtained with negligible effects of thermal drift.

After metal deposition, the size of the half-shadow and the known geometry of the evaporator setup can be used to calculate the distance between mask and sample (see also the discussion in Ref. 18). Using this method results in an experimentally determined mask–sample separation of 8–16 μm, which is the resolution limit of our optical microscope.

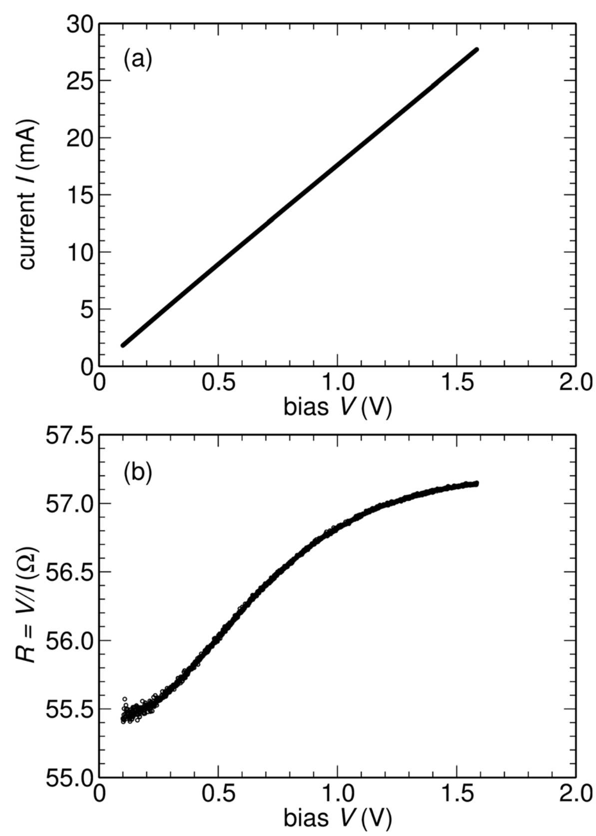

FIG. 4. (a) Measurement of the current as a function of voltage of a Au sample contacted by the method described above. (b) Calculated resistance as a function of voltage from the measurement shown in (a).

Figure 4(a) shows the measured current as a function of applied voltage for an Au nanobridge fabricated by the method described above. The sample has been fabricated for controlled electromigration thinning.[5] The measurement has been taken at the start of the thinning procedure, where the nanobridge had a resistance of 55.5 Ω. The results of the electromigration thinning procedure showed that contacts in the tunneling regime were formed and are discussed in detail in Ref. 19. In this publication, the results of the electromigration thinning procedure are found to be similar to results obtained by electromigration of nanobridges fabricated using standard electron beam lithography methods and on-chip microstructured $Si_{3+x}N_{4-x}$ membrane masks.

IV. SUMMARY AND CONCLUSIONS

We conclude that it is indeed possible to fabricate nanostructures with an increased alignment precision using a magnetic tripod connection system. This alignment precision allows for $Si_{3+x}N_{4-x}$ masks with less structures needed than previously for one nanobridge to span the macroscopic contact pads. These nanostructures are intended to be imaged by scanning probe microscopy methods without surface contamination in the future. In addition, they can also be contacted using a four-point contact holder. In the UHV-chamber, contact springs are installed that allow to establish a contact between the contact posts and the outside world in order to perform electronic transport measurements *in situ*. First results show that electrical measurements are indeed possible with this method.

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