Concept for support and heating of plate-like samples in the ultra-high vacuum

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We present the concept for a sample holder designed for mounting and heating of plate-like samples that is based on a clamping mechanism for easy handling. The clamping mechanism consists of a U-shaped bracket encompassing the sample support plate from the rear. Two spring wires are fixed in the walls of the bracket spanning the sample to secure it with only two point contacts. This enables the sample to freely expand or contract during heating and cooling. To accommodate for a large variety in sample size, shape, and quality, we introduce two designs differing in the generation of the clamping force: One pressing the sample against the spring wires, the other one pulling the spring wires onto the sample. Both designs yield an automatically even alignment of the sample during the mounting process to achieve an even load distribution and reliable fixation specifically for brittle samples. For high temperature treatment, the sample holders are enhanced by a resistive heating plate. As only the sample and a small fraction of the sample holder are heated, temperatures of 1300 °C are reached with only 8 W heating power. The sample support and heating components are mounted on a 11 mm × 13 mm base plate with a handle that can be transferred between the sample entry stage, the preparation stage, and surface science experiments in the ultra-high vacuum system.
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I. INTRODUCTION

For high resolution scanning probe microscopy studies, flat layered and soft materials such as mica[1,2] and graphite,[3] refractory metals such as tungsten,[4] metal oxides[5,6] such as sapphire or nitrates such as highly oriented pyrolytic boron nitride (HOPBN)[7] are well appreciated substrates, where specifically the preparation of some oxides requires very high temperatures.[8] Such samples are either prepared by cleaving[9] and subsequent heating in the ultra-high vacuum (UHV) for surface cleaning or are prepared by cycles of sputter cleaning and high-temperature annealing[10] to yield a clean and well ordered surface. Specimens available often have the forms of small plates with one surface cleaved or mechanically or chemically polished.

Here, a sample holder is introduced that allows for easy handling and preparation of small samples by sputter-anneal cycles. Opposite to previously published designs,[10–12] our new design allows for a variability in size and shape and the preparation at highest temperatures. The clamping mechanism provides an even load distribution and a precise force generation ensures the rigid fixation even of brittle crystals. As the clamping force is exerted by point contacts without lateral confinement and the sample can expand or contract freely, thermal stress and the risk of fracture is effectively reduced. Furthermore, the design is optimized for easy sample exchange and allows the incorporation of a heating element for high temperature sample treatment at minimum heating power. Therefore, parts are manufactured from high temperature compliant materials such as tantalum and molybdenum. The point contacts mechanically fixing the sample can also be used to establish an electrical contact if required. Basis for mounting the clamping mechanism is a 11 mm × 13 mm metal plate with a handle that allows for a transfer within the UHV chamber.

II. UNIVERSAL DESIGN FOR THE SUPPORT OF FLAT CRYSTALS

Design I of the sample holder is especially suited for layered materials such as highly oriented pyrolytic graphite (HOPG)[3] or Mica[1] which often come as plates. As layered samples are often soft and delicate in handling, we devised a clamping mechanism holding the sample with adjustable force as shown in the details of Fig. 1. The sample is mounted on a support bridge affixed to the base plate where the bridge is encompassed from the rear by a U-shaped bracket. Two spring wires with a diameter of 0.3 mm are inserted through the top most ridge of the vertical side walls of the bracket spanning across the bridge. The distance between the wires is set according to the sample size and orientation. In between the spring wires, the side walls of the bracket are lowered to allow for access over a large solid angle as is required, for instance, for scanning force microscopy and angle resolved electron emission experiments. A slight downward bending of the spring wires provides the contact to the sample at one point contact only. The clamping pressure is generated by pulling the bracket towards the sample holder base plate by a hexagon socket bolt that is screwed through a thread in the base of the bracket and presses against the bridge from the rear. The globular tip of the screw is received by a depression in the rear side of the bridge allowing the bracket to tilt and adjust for an even force distribution between the two spring wires. Thus, the sample is fixed by fastening only one screw allowing very precise control of the clamping force

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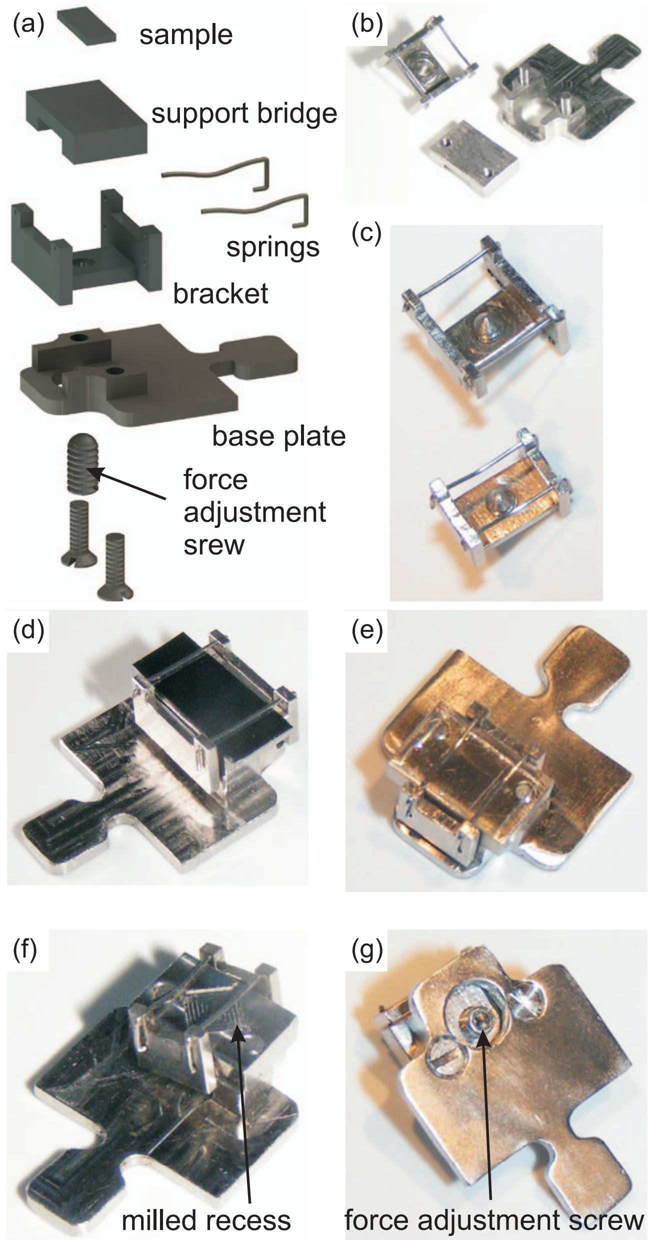

FIG. 1. Sample holder design I. (a) Exploded drawing of the sample holder. (b) Photograph of the disassembled components. (c) Close up of brackets designed for different sample sizes by adjusting the spring wire distance. (d) Photograph of an assembled sample holder with adjusted wire distance for a wide sample mounted and (e) of an empty sample holder designed for a narrow sample. (f) Assembled sample holder with milled recess for very small samples. (g) Rear view showing access to the screw for adjusting the spring force.

determined by the stiffness of the springs and the bolt turn position. To prevent the spring wires from turning away from vertical alignment when loaded, they are bent backward and reinserted into the walls of the bracket. Sample insertion is most easily accomplished sideways by unscrewing the bolt and lifting the bracket. As the clamping mechanism fixes the sample only at two well defined points in the outer regions, the sample orientation can be chosen freely. Small rectangular samples can, for instance, be mounted rotated by 45° to allow best access to the surface. For very small samples, a milled recess tailored to the sample shape is introduced into

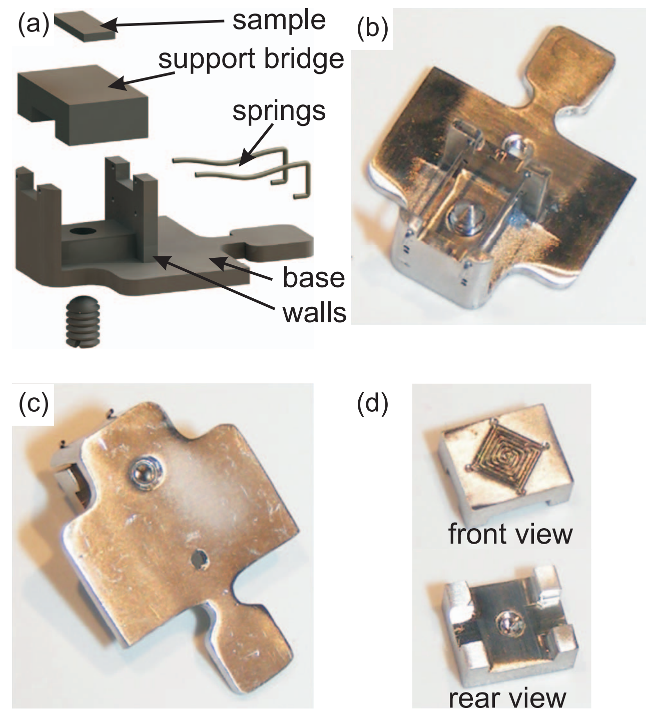

FIG. 2. Sample holder design II. (a) Exploded drawing of the sample holder. (b) Assembled base plate without support bridge. (c) Rear view showing the embedded fixation screw. (d) Support bridge with milled recess for precise placement of very small samples. A depression in the rear side acts as reception for the globular tip of the fixation screw.

the support plate to allow for a precise positioning of the sample as shown in Fig. 1(f). Thereby, the fixation points can be located on the very outer edges of the crystal without the risk of the sample slipping away but preserving as much as possible sample surface accessible.

For surface preparation where moderate sample warming is required, for instance, to remove volatile surface contaminations or excess charge, the entire sample holder is placed on a resistor heating plate of a preparation stage in the ultrahigh-vacuum system. A good electrical contact is established by the spring wires to the topmost sample surface layer which is crucial for some experiments such as scanning tunneling microscopy.

III. DESIGN FOR FIX VERTICAL SAMPLE SURFACE POSITION

For design II, the clamping mechanism is inverted so that the vertical position of the sample surface is determined by the spring wires, which are fixed on two vertical walls on the sample base plate as shown in the details of Fig. 2. The sample support implemented as a movable plunger is placed underneath the spring wires. Thus, the vertical position of the sample surface relative to the base plate is the same irrespective to the sample thickness. In this design, the clamping force is generated by a screw threaded through a fortification of the base plate pressing the plunger with the sample crystal against the spring wires. Thereby, the support position is compensated by the engagement depth of the screw. The increased thickness of the base plate is necessary to embed the screw,

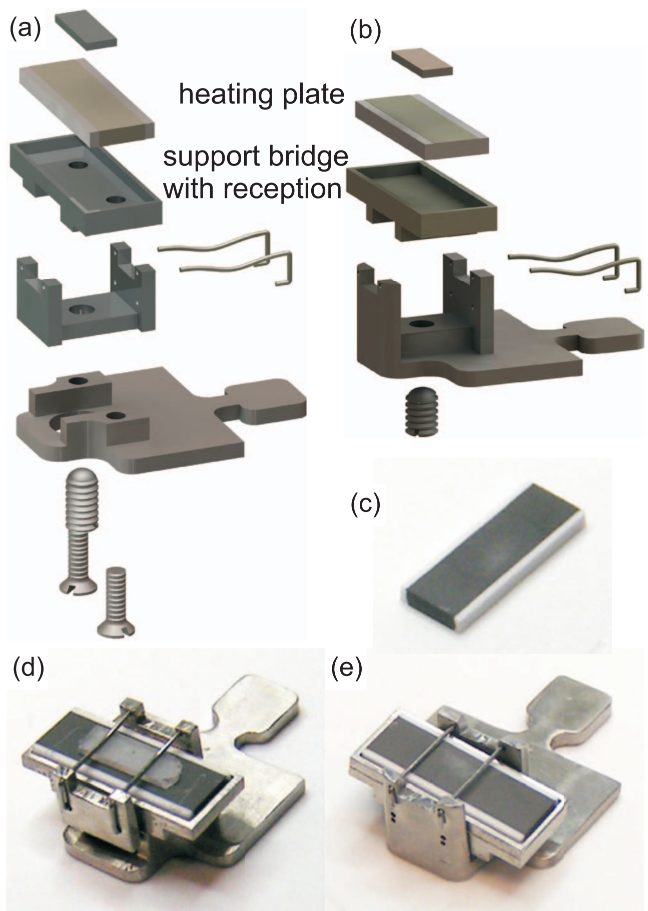

FIG. 3. Exploded drawing of a sample holder (a) in design I and (b) in design II remodeled for direct current heating. (c) Alumina heating plate with a tungsten strip directly fired to the substrate used as heating element. (d) Assembled direct current heating setup in design I with sample. (e) Assembled direct current heating setup in design II without sample.

so that it does not stick out of the rear of the sample holder. The globular tip of the screw rests against the rim of a cylindrical depression in the backside of the plunger. This allows the plunger to slightly tilt to adjust for an even force distribution between the two spring wires. The plunger features guiding rails on the rear side to keep it centered when the screw is released.

IV. DESIGN FOR HIGH TEMPERATURE SAMPLE PREPARATION

For use at moderately elevated or at low temperatures, the sample holder can be made from steel for its good grindability or aluminum for light weight and high thermal conductance. However, for high temperature sample preparation, materials are chosen to comply with the extreme application conditions and a slight modification of the design is required to incorporate a heating element in direct contact with the sample. Details of the respective construction are shown in Fig. 3. The metallic components of the high temperature sample holder are manufactured from tantalum or molybdenum to establish high temperature compatibility while their simple design complies with limitations in machinability. A highly temperature resistant tungsten alloy is chosen for the spring wires as it stays elastic even at highest temperatures and prevents spot welding the spring wires to the sample surface. Localized heating using low power to generate high temperatures is best accomplished by direct current heating in closest proximity to the sample. To incorporate a heating plate into the sample holder, the sample support is remodeled for the reception of a custom made ceramic heater inlay (CeramTec GmbH, Marktredwitz, Germany) as shown in Fig. 3(c). This heating plate is an alumina substrate of 11.0 mm × 4.5 mm × 0.5 mm for highest temperature stability which guarantees a minimum of deformation during heating and is metalized with a 4 mm wide and 25 μm dimension thick tungsten stripe for generating the heat. The precisely fabricated metal layer assures for a homogeneous heat distribution reducing thermal strain and minimizing the risk of dunting. The small conductor cross section allows for using relatively low currents for reaching the required temperature. Aside from its high melting point, tungsten is chosen as the conductor layer as it does not spot weld to any sample material. The alumina substrate likewise acts as thermal and electrical insulation from the sample holder. The tungsten coating encompasses the ceramic inlay in U-form contacting the plunger or the support bridge only on one side. Therefore, it is possible to either contact the tungsten heating layer with two sliding contacts on both sides or to use only one sliding contact and the base plate as the second contact. The fraction of thermal energy conducted through the heating stripe is calculated to estimate the effective thermal insulation effect of the heating plate. The heat flow is given by

$$\dot{Q} = \frac{\lambda\, A\, \Delta T}{l}, \tag{1}$$

where the cross section A and the thickness l are given by the dimension the alumina plate ($A_{\mathrm{Al_2O_3}} = 11\,\mathrm{mm} \times 4.5\,\mathrm{mm},\ l = 0.5\,\mathrm{mm}$) and the tungsten heating stripe ($A_{\mathrm{W}} = 4\,\mathrm{mm} \times 25\,\mu\mathrm{m},\ l = 0.5\,\mathrm{mm}$) and the thermal conductivities λ are given by $\lambda_{\mathrm{Al_2O_3}} = 28\,\frac{\mathrm{W}}{\mathrm{K\,m}}$ and $\lambda_{\mathrm{W}} = \frac{170\mathrm{W}}{\mathrm{K\,m}}$. The heat flows through the materials are calculated to $\dot{Q}_{\mathrm{Al_2O_3}} = 2.77\,\frac{\mathrm{W}}{\mathrm{K}}\Delta T$ and $\dot{Q}_W = 0.03\,\frac{\mathrm{W}}{\mathrm{K}}\Delta T$. As the thermal heat flow through the metal is negligible compared to the heat flow through the alumina, the insulating effect of the alumina plate is not affected.

This setup is perfectly suited for the preparation of electrically insulating samples as there is no electrical shortcut by the direct contact of the sample with the tungsten stripe. To exploit the presented fixation concept even for metallic samples, an electrically insulating heating plate can be used as shown in Fig. 4. Incorporating a graphite conductor path as resistance heater into a custom made pyrolytic boron nitride (PBN) ceramics element (Ceramisis, London, UK) allows for highest sample preparation temperatures while electrically insulating the heater. This modification is best realized using design II as it obliterates the implementation of metal–ceramics junctions. The heating element is molded in form of the plunger. The conductive graphite path is wound in the center at the position of the sample to increase heating power and homogeneity. The conductive path of the heater is uncovered by ceramics at both ends for electrical contact. To protect the thin graphite layer from wear due to the sliding contacts,

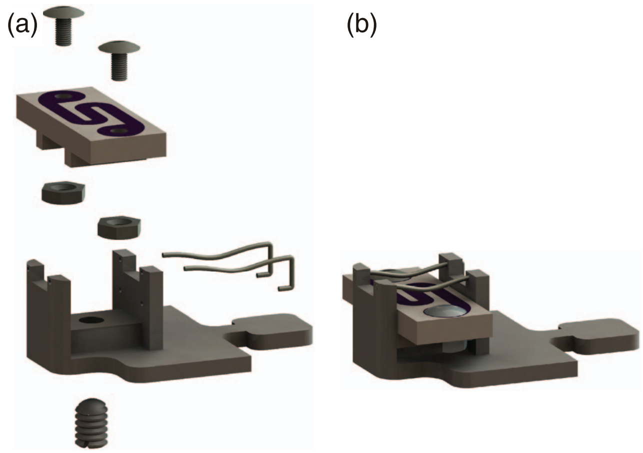

FIG. 4. (a) Exploded drawing of the sample holder designed for electrically conducting samples heated by a PBN heating element. (b) Illustration of the complete assembly without sample.

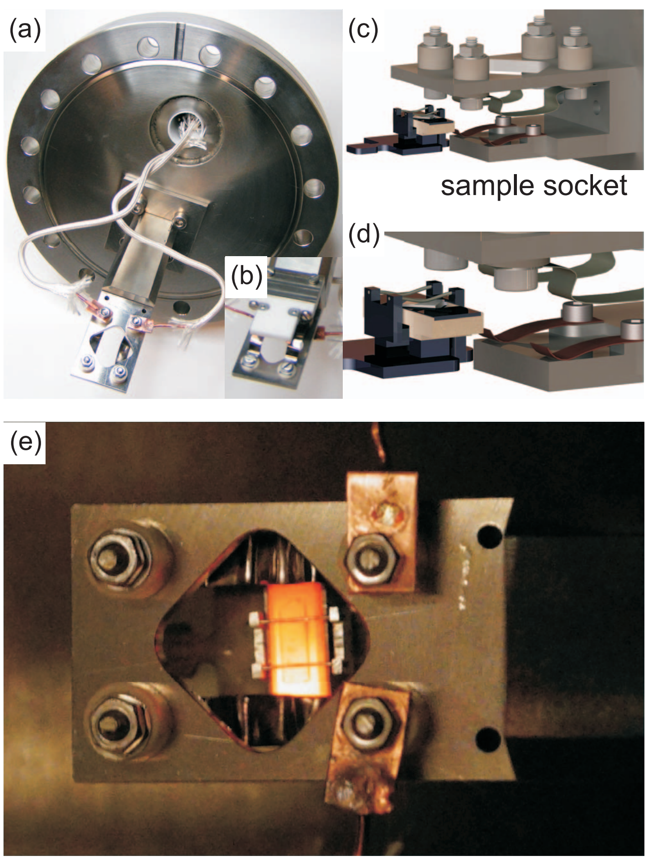

FIG. 5. Sample heating stage supporting the sample holder and establishing the electrical contact to the heating element by two sliding contacts made from 125 μm thick tantalum foil. Both contacts are insulated from the mounting frame by ceramic slit bushes. The sample socket is made from Macor and copper-beryllium springs are used for the fixation of the sample holder. A cut-out in the upper base plate allows for direct line of sight to conduct temperature measurements with a pyrometer. (a) Photograph of the heating stage mounted on a NW100CF flange. (b) Photograph of the rear side of the sample heating stage showing the sample socket made from Macor. (c) Illustration of the sample holder and the heating stage. (d) Close up illustration showing the sample holder, the sliding contacts, and the fixation springs. (e) Sapphire sample crystal heated to 1000 °C with a heating power of 5.8 W.

molybdenum bolts are screwed through the plunger. A reliable electric contact to the sample for STM experiments or a NC-AFM bias connection is formed by the tungsten spring wires connecting the sample surface to the sample holder base plate.

V. HEATING STAGE AND PERFORMANCE TEST

The sample holder is supported in the UHV system by the heating stage shown in Fig. 5 that also supplies the electrical power for heating. The electrical contact is established by two sliding contacts that are made from 125 μm thick tantalum foil. The contacts are formed as bows to facilitate sliding during the insertion of the sample holder and to reduce scratching on the tungsten metalization on the heating element. The tantalum contacts are insulated from the mounting plate by alumina split bushes. The sample socket supporting the sample holder is made from Macor because of its good machinability and for electrical and thermal insulation. Copper-beryllium flat springs are used for the fixation of the sample holder. A cut-out in the upper base plate allows for direct line of sight to conduct temperature measurements with a pyrometer.

The sample holder of design II and the heating stage were tested with a 2 mm × 8 mm × 0.5 mm sapphire sample. We measured the sample temperature with a pyrometer (Metis MS09, Sensor Therm GmbH, Sulzbach, Germany) having a measuring range from 650 °C up to 1800 °C and the emissivity was set to 0.3 to match the emissivity of tungsten. The heating power was stepwise increased by regulating the current from 0 A to 4 A. In the observed temperature range up to 1300 °C the tungsten stripe exhibited a resistive behavior according to Ohm's law with a constant resistance of 2 Ω. The sapphire sample could be heated to 1300 °C in a time of less than 1 min without cracking the sample being testament of the low internal strain during heating. The photograph shown in Fig. 5(d) was obtained at 1000 °C sample temperature with a heating current of 1.7 A at a voltage of 3.4 V equaling a dissipated heating power of only 5.8 W.

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