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.

Enabling *in situ* atomic-scale characterization of epitaxial surfaces and interfaces

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A custom designed sample handling system which allows the integration of a commercially available scanning tunneling microscope (STM) facility with a commercially available molecular beam epitaxy (MBE) facility is described. No customization of either the STM imaging stage or the MBE is required to implement this design. © *1998 American Vacuum Society.* [S0734-211X(98)01606-0]

Molecular beam epitaxy (MBE) has emerged as the technology pushing the frontiers of compound semiconductor device development. MBE uniquely provides the atomic layer control of the growth process necessary to produce compositionally abrupt interfaces and exact layer thicknesses required for band-gap engineering. At present, not all such novel structures can be routinely fabricated nor utilized in device applications. Progress in this area will require an improved understanding of the fundamental surface processes involved in MBE, such as diffusion, nucleation, and growth. Ideally, one would like to study these processes with spatial resolution down to the scale of individual atoms. In the last decade, scanning tunneling microscopy (STM) has matured to become a dominate tool for atomic-scale investigations. For this reason, it is not surprising that there is growing interest in integrating MBE and STM technologies into a single ultrahigh vacuum (UHV) system. Combining STM with MBE has been accomplished in only a few systems[1–3] and they have already demonstrated their ability to advance the fundamental understanding of crystal growth processes.[4–6] One limitation of existing systems is that they use custom microscopes, making the successful integration and operation of such systems limited to specialized experts in the STM community. In this Shop Note, we detail the modifications required to integrate a commercially available STM[7] with a commercially available MBE,[8] without customizing the microscope or deposition system.

Our approach for integrating MBE with STM is to connect two separate UHV chambers via a third commercially available ultrahigh vacuum transfer chamber (the MBE and STM are separated by ~2 m). One benefit of this approach is that it allows for the independent operation of the MBE and STM, while avoiding contamination and vibration problems, as well as electrical and thermal noise problems inherent to placing a STM inside a growth chamber.

The customized components required for this integration are associated with the sample mounts and transfers. We use both the standard MBE and STM sample mounts as our starting material, from which we fabricated new sample mounts that could still be transferred in the conventional way. The current MBE standard for sample mounting and manipulation is a circular 3 in. diam molybdenum wafer holder which will be referred to as a ''MBE moly block'' throughout this article. The current STM sample plate standard is a tantalum plate which is significantly smaller (15 mm×18 mm) than the MBE moly block. The MBE moly block is transferred and mounted using radially oriented pins, while the STM sample plate is held by an eye hole situated on the top side of the mount [see Fig. 1(b)].

Successful STM studies of MBE grown material require several factors to be simultaneously met. First, the substrate must be heated to temperatures in excess of 600 °C in order to remove the oxide from the semiconductor substrate. Second, the substrate surface must have glancing angle access to perform *in situ* reflection high-energy electron diffraction (RHEED). Third, the STM sample plate must be rigidly mounted to the STM imaging stage.

The core modification made to the standard STM sample plate which allowed the successful integration is the addition of a 1 mm thick tantalum over plate to the STM sample plate [see Figs. 1(a) and 1(b)]. The over plate has a square shape and is sized to match the vertical dimension (15 mm) of a standard sample plate. In addition, a rectangular cutout (7 mm×2 mm) centered on the bottom edge of the over plate allows a compression point contact to be made to the sample plate when mounted on the STM imaging stage. Once machined into the proper geometry, the over plate is easily spot welded to a conventional tantalum sample plate. The shape

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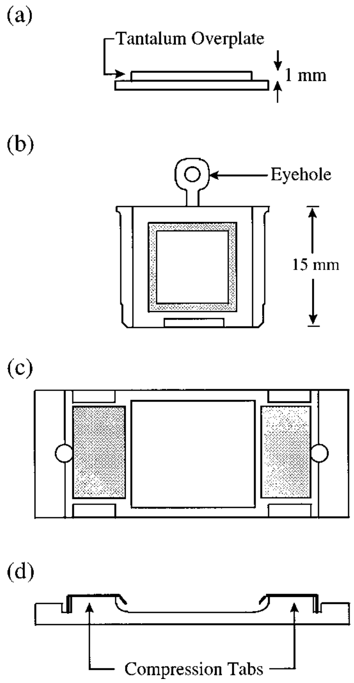

FIG. 1. Schematic diagrams showing the modified sample plate and mounting bracket. (a) Side view of modified sample plate showing 1 mm thick tantalum over plate. (b) Front view of modified sample plate. (c) Front view of standard mounting bracket used to hold the tantalum sample plate. (d) Side view of mounting bracket showing the compression clips which hold the STM sample plate.

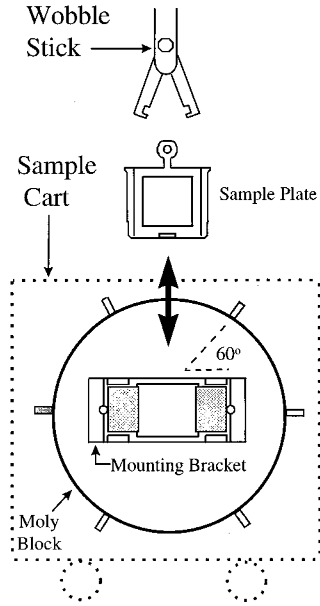

FIG. 2. A schematic explosion-style diagram showing the wobble stick, sample plate, and MBE moly block with a mounting bracket attached. The wobble stick moves along the face of the MBE moly block to position the sample on or off the mounting bracket.

of the over plate naturally provides a 1.5 mm wide lip on either side of the standard sample plate. This exposed surface is used to guide and hold the sample plate in all the mounts throughout the multi-chamber facility. No other modifications to the sample plate are necessary, since one could simply indium solder a substrate directly to the over plate. One can, however, make additional modifications for sample mounting as desired. For example, one could tap screw holes through both the over plate and the standard plate, providing 2 mm of thread depth to hold a sample to the over plate using clips. Our approach was to machine a 10 mm $\times$ 10 mm hole through the entire mount which is centered on the over plate, so we could radiatively heat the substrate. In addition, to help center the sample and stop it from rotating or sliding we cut a thin recess about 1 mm wide and approx. 0.2 mm deep all the way around the hole going through the mount [see shaded region in Fig. 1(b)]. Our samples measure approx. 12 mm on a side and are indium soldered to the 1 mm wide ledge surrounding the hole. The modified sample plate is maneuvered and positioned throughout the STM chamber using the standard transfer tools and mounts. For example, the standard manipulator inside the STM chamber has a compression-style mounting bracket for the sample plate, which is shown schematically in Figs. 1(c) and 1(d). By holding the eye hole situated at the top of the sample plate with a standard wobble stick, the plate can be slid underneath the two compression tabs on the mounting bracket.

Conveniently, the compression-style mounting bracket [shown in Figs. 1(c) and 1(d)] on the manipulator can also be used to hold the sample plate onto the MBE moly block (see Fig. 2). The only modification required is to tap two screw holes into the MBE moly block in order to attach the mounting bracket. In addition, we cut a hole through the center of the MBE moly block to allow the substrate to be radiatively heated by the filaments. Since the sample is mounted on top of the 1 mm thick over plate, the top surface of the sample is naturally elevated above the compression tabs and the supporting hardware of the mount allowing complete 360° RHEED access. Note, the surface of the sample is raised approx. 3 mm above the position where it would have been when mounted on a standard MBE moly block. Consequently, it is necessary to translate the entire MBE moly block and heater assembly inside the MBE chamber back 3 mm using the translation micrometers on the manipulator to get RHEED access.

The positions for the two tapped screw holes in the MBE moly block are chosen such that the center of the sample is in the center of the block, and such that the eye hole is vertical when the MBE moly block is mounted on the sample cart which shuttles the block between the MBE and STM chambers (see Fig. 2). A wobble stick is used to slide the modified STM plate on and off the MBE moly block, as illustrated in Fig. 2 (not drawn to scale). Since the eye hole on the sample plate is only 2 mm above the face of the MBE moly block, it was necessary to modify the wobble stick pincer assembly. On a standard wobble stick, the bolt which secures the two pincers together protrudes from the rear surface of the wobble stick and obstructs access to the sample plate. It is necessary to mill down by 2 mm the head thickness of the bolt that holds the pincers together and, in addition, to recess the head of the bolt 2 mm into the pincer assembly for a total reduction of 4 mm in the height of the pincer bolt.

A cross-sectional view down the axis of the transfer

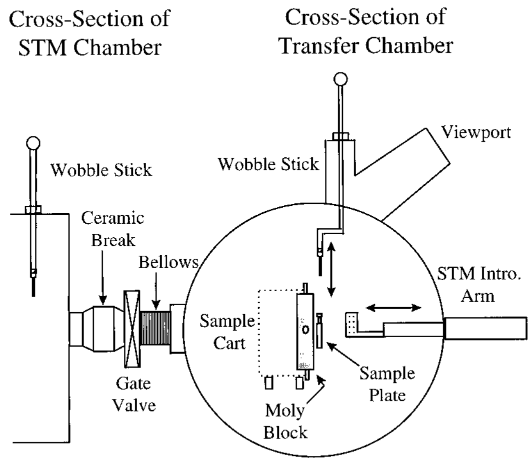

FIG. 3. Schematic diagram showing a cross-sectional view down the axis of the transfer chamber. This view shows how the wobble stick lifts the sample plate off the MBE moly block and positions it on the magnetic drive. Once the sample plate is on the magnetic drive it is moved underneath a second wobble stick inside the STM chamber, which removes the plate and positions it on the standard manipulator. In addition, the electrical break, gate valve, and vibration isolation bellows are shown.

chamber which shows the sample movement between the transfer chamber and the STM chamber is illustrated in Fig. 3. To remove the STM sample plate from the MBE moly block the wobble stick with the thinned and recessed pincer bolt is positioned precisely above the STM sample plate. To achieve this, a customized section of the UHV transfer chamber was manufactured. This transfer module is installed directly in front of the STM chamber, and the custom flange and viewport is positioned above the sample cart (see Fig. 3). After removing the sample plate from the MBE moly block, the cart is moved away. Next, a horizontally mounted linear-rotary magnetic transfer arm is positioned directly under the wobble stick and the sample is transferred to it. The magnetic drive is then used to pass the sample into the STM chamber, where another wobble stick removes the sample from the magnetic drive and places it on the conventional manipulator. Once the sample is positioned on the manipulator all sample handling and mounting proceeds via standard techniques. A ceramic vacuum break is installed between the transfer chamber and STM chamber (see Fig. 3) to electrically isolate the MBE and STM. An edge-welded bellows is installed in series with the ceramic break to reduce vibrational coupling between the MBE and STM system. Also, a gate valve is used to isolate the transfer chamber and the STM chamber for separate venting to atmosphere.

Successful integration of MBE with STM allows atomic scale imaging of the highest quality crystalline surfaces as illustrated by Figs. 4(a)–4(c). An image of a freshly prepared GaAs(001)-(2×4) surface grown at 580 °C using a deposition rate of 1 ML/s is shown in Fig. 4(a). This is a large scale image measuring 1.5 μm on a side. A (001) plane has been subtracted from the data so that each terrace is displayed with a separate shade of gray. The terraces are elongated along the $[\bar{1}10]$ direction and have an average width of 0.5 μm. A higher magnification image, measuring 250 nm on a side, of a region within Fig. 4(a) is shown in Fig. 4(b). At this magnification the 4-by rows along the $[\bar{1}10]$ direction which are separated by 1.6 nm are visible. A yet higher magnification image, measuring only 30 nm on a side, of a region within Fig. 4(b) is shown in Fig. 4(c). At this magnification, the 2-by periodicity along the rows is resolved.

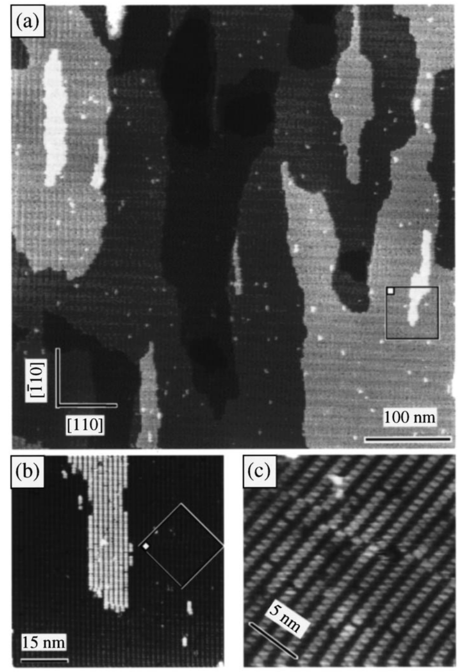

FIG. 4. Filled state STM images of a 1 μm thick GaAs film grown by MBE on a GaAs(001) substrate. (a) 1.5 μm$\times$1.5 μm area, with each gray level representing a change in height of 0.3 nm (a single GaAs layer). (b) 100 nm $\times$100 nm area zoom showing the 4-by periodicity of the (2×4) surface reconstruction. (c) 30 nm $\times$ 30 nm area zoom showing the 2-by periodicity of the surface reconstruction.

In summary, we have detailed the modifications required to integrate commercially available MBE and STM chambers into a single UHV system. Central in this challenge was the coupling of the MBE moly block with a modified STM sample plate in a manner fully compatible with the individual needs of both the MBE and STM chambers. Such integration of MBE with STM provides the unique ability to routinely perform *in situ* STM studies of epitaxial surfaces.

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[1]A. Madhukar (private communications).
[2]L. J. Whitman, P. M. Thibado, F. Linker, and J. Patrin, J. Vac. Sci. Technol. B **14**, 1870 (1996).
[3]C. A. Ventrice, V. P. LaBella, and L. J. Schowalter, J. Vac. Sci. Technol. A **15**, 830 (1997).
[4]P. M. Thibado, B. R. Bennett, M. E. Twigg, B. V. Shanabrook, and L. J. Whitman, Appl. Phys. Lett. **67**, 3578 (1995).
[5]P. M. Thibado, E. Kneedler, B. T. Jonker, B. R. Bennett, B. V. Shanabrook, and L. J. Whitman, Phys. Rev. B **53**, R10481 (1996).
[6]P. M. Thibado, B. R. Bennett, M. E. Twigg, B. V. Shanabrook, and L. J. Whitman, J. Vac. Sci. Technol. A **14**, 885 (1996).
[7]STM II, Omicron Associates, Denver, CO 80209.
[8]Riber 32P, Riber, Inc., France.