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Optimizing the environment for sub-0.2 nm scanning transmission electron microscopy

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Abstract Sub-0.2 nm probes can now be readily obtained on Schottky field-emission microscopes. However, environmental instabilities are proving to be the limiting factors for atomic resolution spectroscopy and distortion-free annular-dark field imaging. This is a result of the long acquisition times and the serial nature of the scanning system where instabilities result in image distortions rather than reductions in contrast. Troubleshooting the most common environmental problems is discussed here. In addition to the expected sensitivity to mechanical vibration, electromagnetic interference and temperature variations, air-pressure fluctuations are found to have a significant impact on microscopes with side-entry goiniometers.

Keywords STEM, EELS, environment, magnetic fields, pressure, atomic resolution

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Introduction

The basic mechanical design of commercial transmission electron microscopes has not changed dramatically since the mid-1970s. However, considerably more stability is expected of these instruments, as detector electronics and gun designs have improved. As a consequence, sub-0.2 nm probes can now be readily formed on microscopes equipped with Schottky field-emission guns (FEG) [1]. Such performance, particularly for spectroscopy, was previously limited to dedicated scanning transmission electron microscopes (STEM), such as the former Vacuum Generators HB series. (Examples are given by Batson [2], Browning *et al*. [3] and Muller *et al*. [4].) As semiconductor devices continue to shrink in size, the need to characterize smaller and smaller features on a routine basis grows. By 2001, the gate oxide, which is the smallest feature on an integrated circuit, was thinner than 2 nm. Electron energy loss spectroscopy (EELS) has proved invaluable in probing the interfaces of this thin layer [5,6]), but atomic scale stability is required for these measurements.

The addition of aberration correctors to dedicated STEMs will allow sub-angstrom diameter electron probes to be generated [7]. Because aberration correctors correct only aberrations, and not instabilities, this will only reinforce the need for quieter rooms and more robust instrument design. Atomic resolution spectroscopy and distortion-free annular-dark field imaging are particularly sensitive to environmental instabilities as a result of their long acquisition times (comparable to those required for energy-filtered imaging or inline holography [8]). The serial nature of the image acquisition in STEM makes the instabilities appear as image distortions, while the parallel recording in conventional TEM results in a loss of contrast and ultimately resolution.

The current generation of TEM/STEMs was optimized for TEM performance, which has led to increased environmental sensitivity in STEM mode. In particular, instabilities and deflections of the gun and condenser illumination system in a TEM will only damp the coherence envelope. This is not a particularly serious problem for thermionic sources, although it will impact the sub-0.2 nm performance of a FEG-TEM. However, gun and condenser stability is critical to forming a small probe in STEM mode (as critical as is the coupling between objective and imaging lens in TEM). The condenser lenses are often designed with large gaps, making them vulnerable to stray fields, if not properly shielded. The gun is placed on the top of the column, the point most sensitive to mechanical sway. Consequently, satisfactory operation of a thermionic source TEM is no guarantee that a STEM placed at the same location will perform as well.

As an illustration of the different environmental response between TEM and STEM, Fig. 1 shows a sequence of STEM images recorded at different points in the installation process as the room environment was gradually improved. The earliest STEM image (Fig. 1a) shows weak 0.3 nm fringes in one direction only. This image was recorded after the magnetic fields

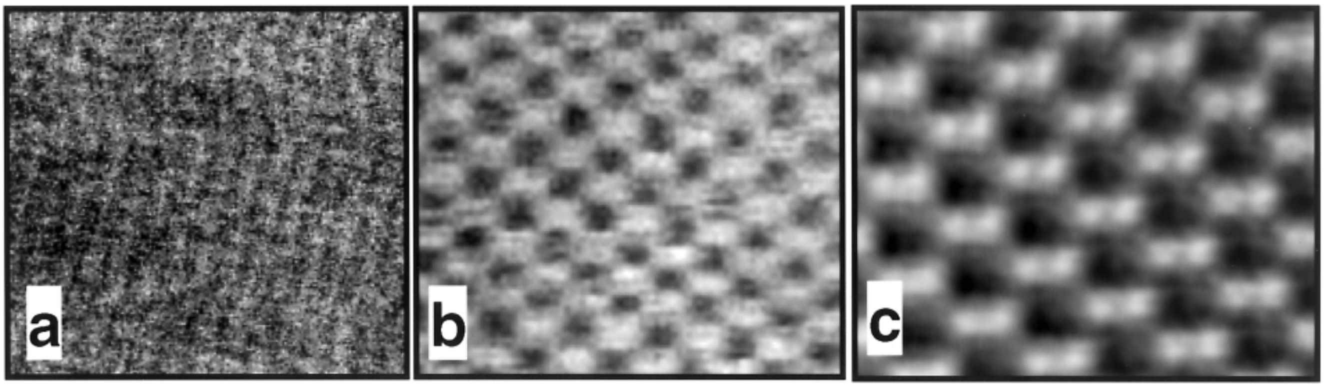

Fig. 1 Annular dark-field STEM images of [110] oriented silicon, recorded (a) when the microscope was first installed, (b) after 6 months and (c) after 1 year. The improvement in image quality from (a) to (b) was from improvements in the instrument, and from (b) to (c) was due to improvements in the room environment.

had been reduced by a factor of three from the conditions experienced by the previous microscope in the room, which was a conventional TEM of similar size and beam voltage. This conventional TEM had demonstrated a 0.2-nm information limit. While this was worse than the 0.14-nm information limit the TEM was theoretically capable of reaching, it is still far better than that of the STEM image in Fig. 1a. Another lesson to be learnt here is that if the previous instrument is not performing perfectly, don't expect the replacement to do so either. Figure 1c shows that there was a happy ending to this story (about 18 months later).

As is always the case, a great deal of pain and wasted time can be avoided by careful planning. It is much easier to fix the room before the microscope is installed than afterwards — imagine trying to cut an isolated slab with the machine in the room. It is always easier to fix a problem at its source than shielding at the instrument. Shielding attenuates, rather than removes, a disturbance. Removing all old cabling, pipes and ducts in the room is cheaper than trying to find bad ground connections later (days vs months of a professional electrician's time).

It is worth bearing in mind that the room is really part of the microscope and one should budget accordingly. In many cases, a new building may be cheaper than a new spectrometer, and it is easier to add a spectrometer later. While the manufacturers can survey a potential site to see if it meets all their specifications, they cannot be expected to find the problems if it does not. (It took us 6 months to find and remove all the ground loops in our wiring system before we met specifications.)

It is tempting to think that all remaining problems can be fixed with active cancellation systems, but these are effective only up to a point. In many cases the microscope will be a more sensitive monitor of the disturbance than the sensor used in the feedback loop. Active vibration compensation systems work best at low frequencies (10 Hz and below). The most common vibrations at industrial sites are at 30, 60 and 120 Hz from AC motors, and active systems provide little improvement over passive systems at 30 Hz and above. Building an isolated slab beforehand is a more effective (and often a cheaper remedy).

Electromagnetic (EM) field cancellation systems can only correct one point on the microscope. This means choosing between the spectrometer and the gun (i.e. choosing between spatial or energy resolution). These cancellation systems are then most effective in large rooms where the correcting field is most uniform, and where the external field is uniform across the column (i.e. it is probably from a remote source rather than a wiring problem in the room itself).

The best understood environmental problems (where the manufacturer's specifications will be most complete) are mechanical vibrations and AC interference. As the mechanical design of the column, in terms of mass and shape, has changed little in the past quarter century, a good starting point for understanding vibration control is Anderson's book chapter [9]. Some examples specific to FEG-TEMS are given by Turner, O'Keefe and Mueller [8] and Hetherington *et al.* [10]. (All these articles also cover other environmental issues such as EM fields.) While a necessary condition for good performance, vibration control will not be discussed further, as it is well covered in these three previous articles.

The four key environmental challenges discussed here are the control of

(i) EM fields to less than 0.1 mG r.m.s.,
(ii) temperature changes to less than 0.1°C h^{-1},
(iii) airflow across the column to less than 30 feet min^{-1}, and
(iv) air pressure changes to less than a few Pascal min^{-1}.

These are not just STEM problems: through-focal series require an even higher stability and energy filtered images require successive exposures lasting 10 s or more. The issues discussed here are relevant for all side-entry goiniometers and any system having a viewing chamber or other flux-leakage points. The examples given are for a 200 kV JEOL 2010F FEG-STEM with Gatan imaging filter (2 k × 2 k), but from discussions and visits with colleagues, the numbers and underlying physics appear to be quite general.

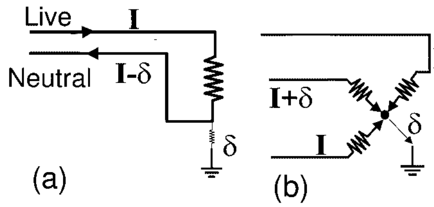

Fig. 2 The origin of ground currents in (a) a single phase circuit and (b) a three-phase circuit. The net AC field generated by the conductors will be proportional to the current, δ, lost to ground.

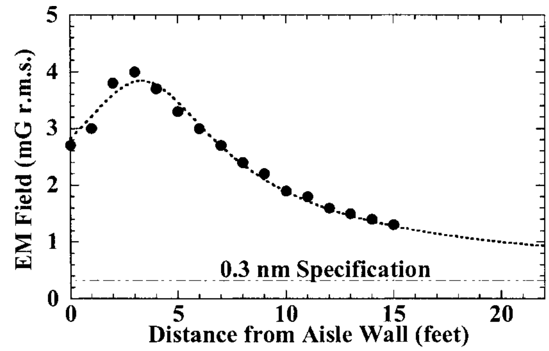

Fig. 3 The measured magnetic field from a power bus located 3 feet below the floor (closed circle). The fit (dotted line) to Ampere's law is for 1.5 mA current flowing at x = 3.3 feet and 2.9 feet below the floor. The bus was rerouted.

Electromagnetic interference

Electromagnetic interference can cause beam deflections in both the scanning system and the spectrometer [10]. These are most easily dealt with before the machine is installed, as substantial rewiring may be necessary. There is little that can be done about quasi-DC fields, such as from elevators and nearby trains and buses. Major sources of AC electromagnetic interference are unbalanced electrical loads. For a straight conductor, the magnetic field B (in mG) is given by Ampere's law as

$$B = \frac{2I}{d} \tag{1}$$

where I (in mA) is the current in the conductor and d (in m) is the distance from conductor. The direction of the field is given by the right-hand rule — point the thumb of your right hand along the wire, and your remaining four fingers curl in the direction of the field. This is particularly useful when hunting down ground loops.

As a practical matter, a 0.3 mG r.m.s. field can be detected in a 0.3-nm STEM image. Less than 0.2 mG r.m.s is needed for clean 0.2-nm STEM images. (In converting from root-mean-square to peak-peak measurements, multiply by ~3.) Equation 1 puts this in practical terms. At a 1 m separation from a straight wire carrying just a 0.5 mA, the magnetic field reaches 1 mG, enough to degrade 0.3-nm STEM performance. This does not mean that turning on a 60 W light bulb should blur out all STEM images.

If as much current leaves through neutral as enters through the live wire in a conduit, the magnetic fields from the 2 wires will cancel and there will be no net field. It is only when some of the return current finds another path to ground that a field will be generated. This is the case for a common 2-phase wiring mistake where the neutral and the ground lines are accidentally bonded at conduit junctions or at the load, instead of only at the source. This is illustrated in the circuit diagram of Fig. 2a. In our case, the T-junction blocks for the neutral lines were accidentally bonded to ground. This happened because the screw holding the junction block in place was too long, and had penetrated the insulator, contacting the case, which was at ground. These mistakes are easy to fix, but can be difficult to isolate.

The analogous problem for three-phase supplies can arise from unbalanced loads (common in old three-phase motors) where the unbalanced portion flows to ground (Fig. 2b). As most big motors are three phase, the unbalanced current to ground is often not small. Safety dictates that the case of the motor must be kept at ground potential. Part of the ground current will then flow through this case, which can be connected to air conditioning ducts, water pipes, wiring conduits, lightning grounds and metals beams. Microscopes tend to be in basements, and right in the path of all these currents to ground.

Both the single and three-phase problems can result in large ground currents flowing through metal conduits, pipes and air ducts in the room. Adding dielectric breaks to the water pipes and air ducts, and using plastic conduits (with ground wires inside) can isolate the microscope from other people's mistakes. However, there is no substitute for large rooms ($1 / d$) and removing all old wiring. (A shielded room only works if it completely encloses the microscope on all six sides, and is large enough that the fields generated by the microscope are not significantly enhanced.)

Handheld low frequency gauss meters with 30–300 Hz bandwidth and 0.1 mG r.m.s. sensitivity are invaluable for tracking down the offending sources and cost less than $100 (Fig. 3 shows how Ampere's law and a meter were used to pinpoint a problem wiring outside the room). These meters need to be calibrated with a search coil, as factor of three variations in sensitivity is not uncommon. Suppliers such as www.technitool.com and www.cole-parmer.com sell these meters over the web.

Post-column spectrometers are particularly vulnerable to changes in magnetic field that enter through the unshielded camera and viewing chambers. Until viewing chambers (which are obsolete on STEMs anyhow) are removed, there is little that can be done to shield the spectrometer. Any material with a high magnetic permeability moving through earth's magnetic field will cause those field lines to deflect, becoming a source of quasi-DC noise in the spectrometer. The closer the

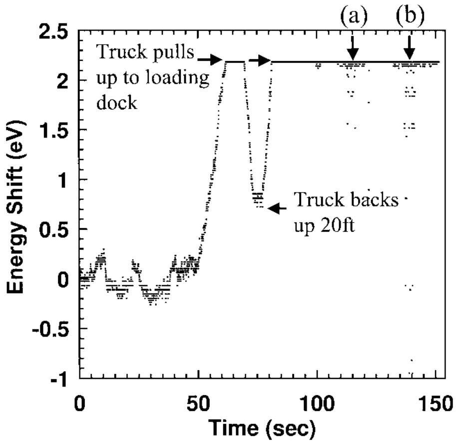

Fig. 4 Deflection of the zero-loss peak on the post-column energy loss spectrometer as a truck pulls up to the loading dock outside the microscope room. The signal is clipped at 2 eV. The truck is first detected at about 50 feet away (t ~55 s). It pulls up to the loading dock, and then backs up to come in straight. The spikes at (a) and (b) correlate well with the driver opening his door (a) and dropping the tailgate of the truck (b).

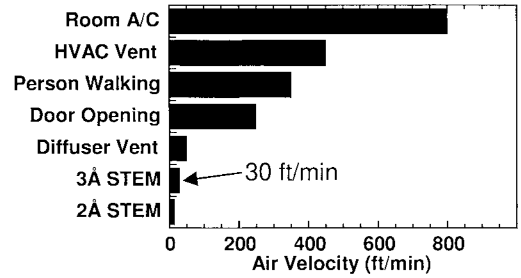

Fig. 5 Maximum airflow across the microscope column that can be tolerated for sub 0.3 and 0.2 nm STEM compared with common causes of drafts.

object is, the smaller it can be and still cause trouble. A penknife waved in front of a spectrometer causes shifts of an eV or so. The iron wheels or axles of a typical office chair will cause similar shifts when moved (such as when the operator leans over to record a spectrum). We have replaced the chairs in the microscope room with all-wood furniture. Plastic pool furniture works as well as the Amish-made chairs we have, although with a noticeably shorter lifetime. Sources outside the room can also be a problem. Recording DC fields at the site of the new column with a chart recorder for a few days may be worth doing if the room is near a busy corridor, elevator or road. Large electric furnaces or NMR machines do not make good neighbours.

Figure 4 shows the deflection of the zero loss peak (ZLP) on the spectrometer of our microscope when a truck pulled up to the loading dock located 20 feet from the column of the microscope. With the help of JEOL engineers R. Hynes and G. Griego, we have calibrated the effects at roughly 1 eV of ZLP deflection per litre of engine capacity at 20 feet (actually it should scale with the total mass of iron in the vehicle). At ranges beyond 50 feet, the spectrometer ceases to function as a truck detector.

Many older microscope peripherals can generate very large AC fields. Often the small black and white monitors used to display the signal from TV cameras can generate fields as large as 60 mG from the magnetic coils used to raster the electron beam across the phosphor screen. Computer monitors are also problematic. Even TCO-95 compliant monitors can still generate 10 mG fields in front of the display. Liquid crystal displays (LCDs) have no magnetic coils and should generate fields less than 0.1 mG a few feet away. Fortunately, LCDs are dropping in price. A 17 inch 1280 × 1024 pixel LCD with a picture-in-picture display for a TV camera can be purchased for less than $1500. Most new microscopes are shipped only with LCDs.

Some dedicated STEMs, such as those at Cornell and IBM Yorktown Heights, have been installed in shielded rooms made from mumetal. Slowly varying magnetic fields are not strongly attenuated. Instead, magnetic shielding works by providing a low reluctance path for external fields. Consequently, it is only effective if it closes the instrument on all sides. Placing shielding on only 1 wall is not very effective. As a rough rule of thumb, the magnetic field will penetrate roughly 5 times the size of any hole in the shielding. The shielded room also needs to be quite large, as any fields inside the room will also induce image fields in the walls. The amount of space required to house and service a TEM/STEM makes such rooms rather expensive. Simply choosing a large room (with a high ceiling) for the microscope may be as effective since most fields decay rapidly with distance. (One exception is the snow-melting coil that runs under the sidewalks encircling our building. To a good approximation, this can be treated as an 8-turn solenoid carrying 100 Amps of AC current surrounding the building. It is left as an exercise for the reader to calculate the strength, direction and spatial variation of the resulting field when it snows.)

As a final word of warning, it is often cheaper to avoid rooms with field problems than to correct them. In many cases when the large fields from bad wiring in the room are removed, it becomes clear that this was not the only room to be badly wired. The offending source could easily be the main power bus for the building located 3 labs away.

Airflow and temperature control

The most obvious way to transmit mechanical vibrations to the column of the microscope is through the floor. While floor vibrations can be a serious problem in a poorly chosen room, they are also relatively easy to identify, and each manufacturer has well-established limits for the maximum allowable vibrations. However, this is not the only source of mechanical vibrations. Pressure and temperature fluctuations from airflow across the microscope column can lead to random displacements in the image (especially over 1–30 s). The x and y drives for the stage are probably where the coupling occurs.

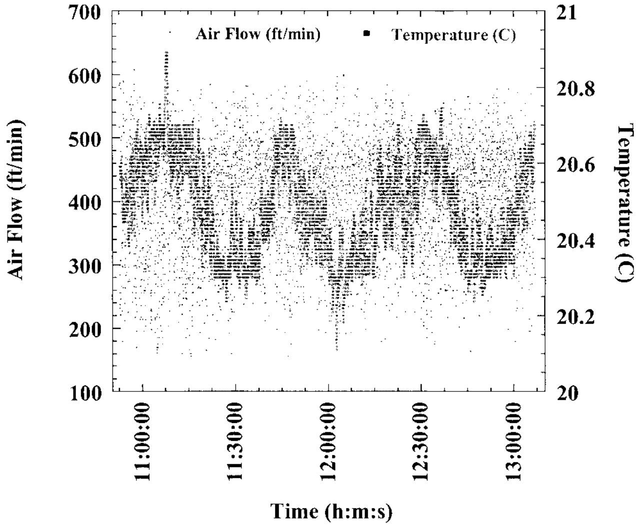

Fig. 6 Airflow and air temperature recorded at the air return vent of the microscope room. The air is well mixed so this temperature reflects the temperature changes in the room. The airflow rate and size of the return allowed us to calculate the mass of air moving through the room, and, hence, its heat removal capacity.

Airflow across the column must be 15 feet min^{-1} or less for 0.2-nm resolution STEM images. Figure 5 shows some common sources of air movement. Air movement can be recorded with a handheld thermoanemometer. These are expensive and 15 feet min^{-1} of air movement is at the lower limit of what they can detect. We used the thermoanemometer to calibrate a simpler series of tell-tales — if a 12×0.25 inch strip of toilet paper (single ply) deflects more than 1 inch, the airflow exceeds 20 feet min^{-1}. By covering the microscope, and the room, with these strips we were able to map out the airflow and identify trouble spots.

Figure 6 shows the airflow and temperature variations of the air at our air conditioning (A/C) return vent. The 400 feet min^{-1} flow rate corresponds to 96 air changes per hour in the microscope room. The heat load, q, in the room determines the airflow rate (from $q = mC_p\Delta T$). C_p is the specific heat of air, m is the mass of air moved and ΔT is the temperature gradient between supply and return. In our case, the heat load was about 4 kW and the temperature gradient was 3°C, which was not good. Most of this heat (~3 kW) was generated by the power supply and electronics rack. The most sensible way to reduce the airflow is to separate (and cool independently of the column and HT) the power supply and electronics racks. Room layouts that do this have been described previously [8,10].

As a low cost alternative, we found that constructing a shelter around the column on 3 sides prevents drafts blowing across it and greatly improves the stability on the 1–10 s time scale. Further, wrapping the column in bubble wrap or neoprene greatly damps thermal fluctuations (Fig. 7).

Most of these airflow problems are specific to forced air A/C systems, where heat is removed predominantly by convection.

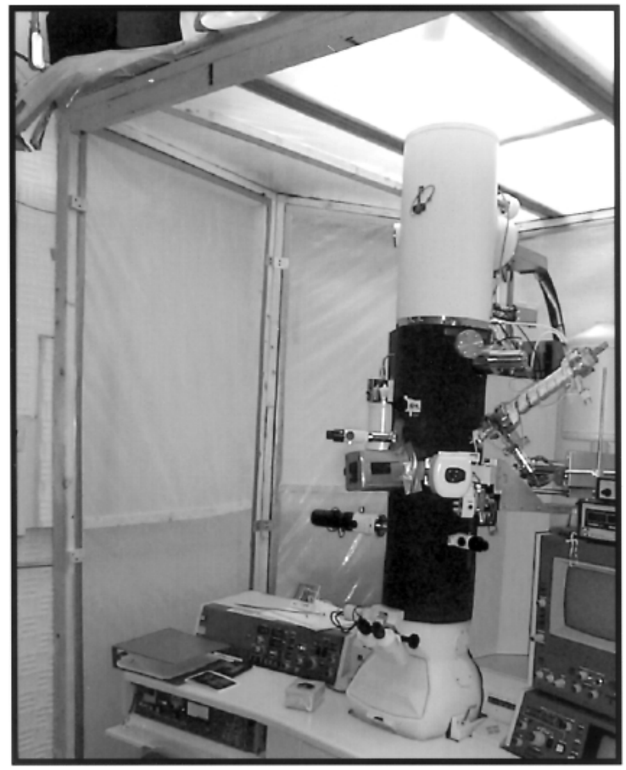

Fig. 7 A 'gazebo' shelters the microscope from cross-drafts produced by the air conditioning, and neoprene rubber around the column and stage damps out pressure and temperature fluctuations.

An efficient alternative is a radiant cooling system. This can be as simple as a large surface-area radiator on the wall with building chilled water flowing through it. If this removes most of the heat load, then the existing forced-air system can be slowed down, and used mostly to regulate humidity. More sophisticated systems are described by Roulet *et al.* [11]. These can regulate the temperature to better than 0.1°C, and as most of the cooling is by radiation, the return to equilibrium is rapid. The exceptional temperature stability is a second (and even greater) advantage of radiant cooling systems over forced air A/C. Figure 6 also shows the typical temperature fluctuations as the forced-air A/C system heaters and cooling coils cycle on and off to maintain an average temperature of 20.5 ± 0.25°C. Adding a reheat coil with feedback from a thermocouple near the column could reduce these fluctuations. However, a radiant cooling system will still be more stable.

Pressure fluctuations

Even when all the previous problems were addressed, we still noted persistent and serious instabilities in our images during the day. Figure 8b shows an image recorded while someone opened the outside door to the building (the room door was closed throughout). The microscope was also sensitive to changes in the weather leading to the saying, 'Drift to the right, rain tonight!'

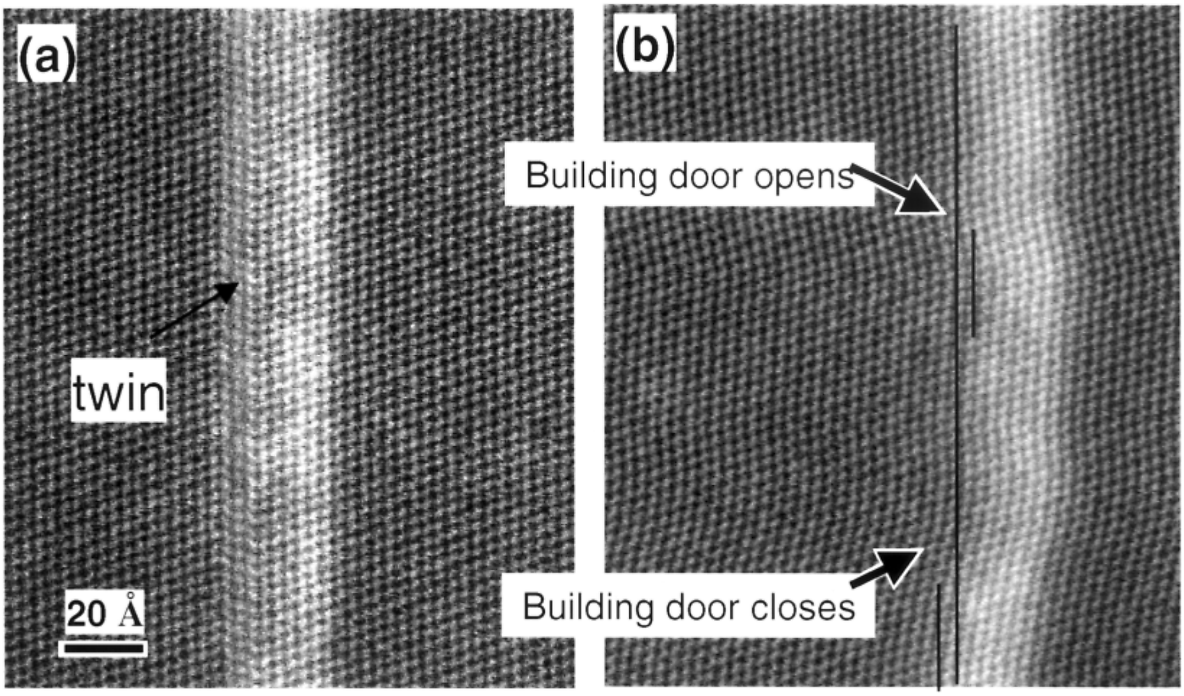

Fig. 8 Annular dark field images of an arsenic-delta doped layer grown on (111) Si, viewed along a [110] zone axis. (a) Recorded at 10 p.m. when there is no traffic in the building. (b) Recorded at 11 a.m. The image is undistorted until the main door to the building is opened, and the image settles down again when the door is closed. The air pressure change of a few Pascals is felt throughout the building and deflects specimen stage by a few angstroms. (Sample provided by O. Dubon, P. Evans and M. Chisholm.)

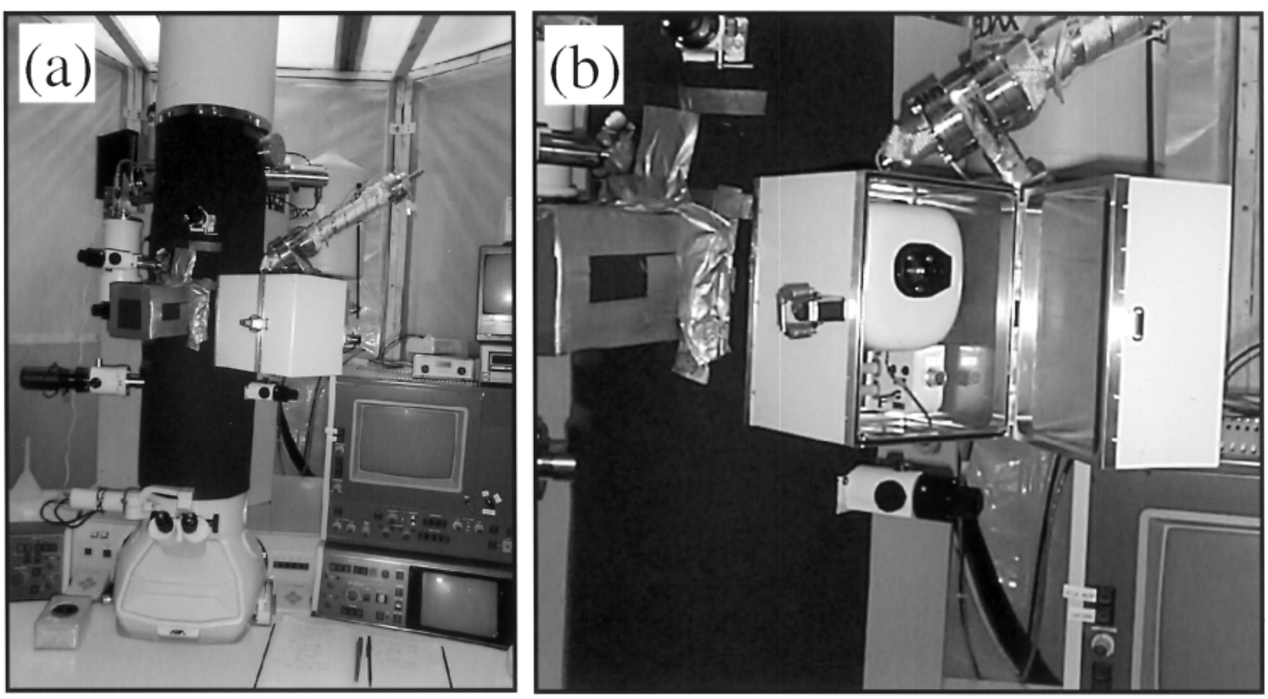

Fig. 9 Adding an airtight airlock cover reduced the pressure sensitivity problem of Fig. 8. (a) The airlock cover is closed. (b) The airlock cover is open, showing the O-ring seal needed to isolate the specimen rod from rapid pressure changes in the room. Note that the stage drive to the left of the airlock is covered with neoprene for improved thermal stability.

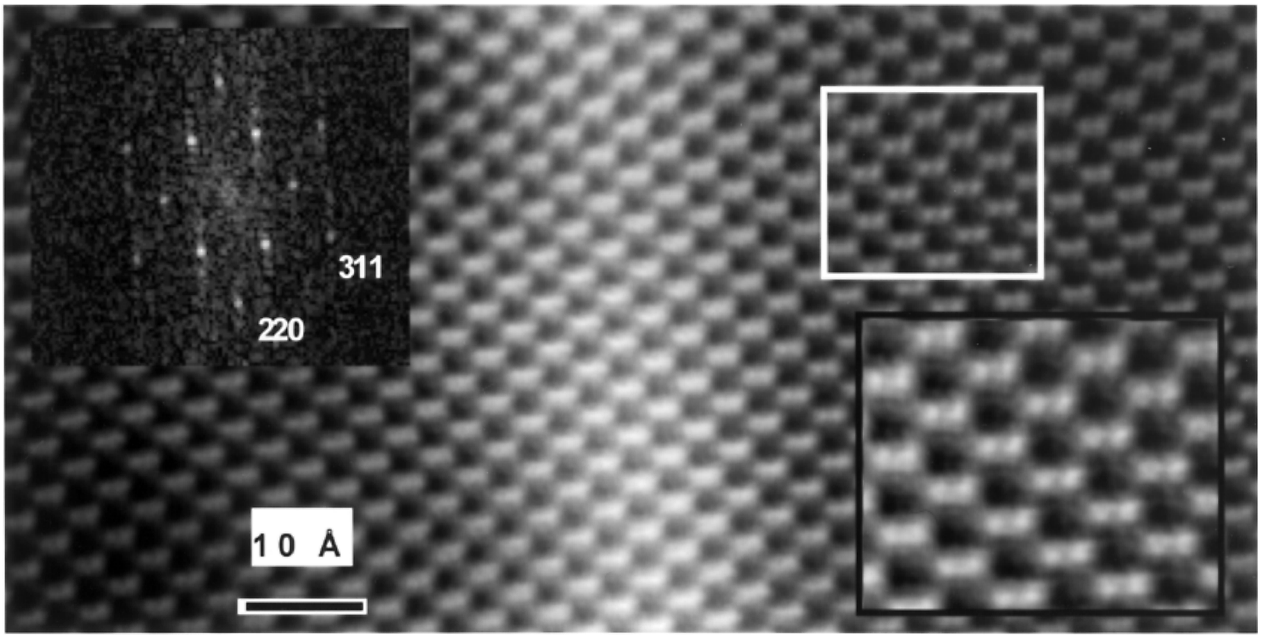

Fig. 10 An ADF-STEM image of a Sb δ-layer in [110] oriented Si, showing an information limit better than 0.163 nm and minimal drift during the 40 s acquisition. The peak Sb concentration is 20 atoms or about 0.2 monolayers. (JEOL 2010F at 197 kV, C_s = 1 mm, θ_o = 10 mr, and low-pass filtered.)

The problem was the side-entry goiniometer. The sample is under vacuum, typically 3×10^{-6} Pa, while the outside of the sample rod is at atmosphere ($\sim 10^5$ Pa). Using a high-precision barometer, we were able to calibrate the deflection of the sample rod as 0.1 nm Pa^{-1}. This is typical given the compliance of the metal rod, and should be common to all side-entry goiniometers, independent of manufacture.

As is quite common in American buildings, the A/C system overpressures the building by about 10 Pa. Opening and closing the door sends a pressure wave down the corridor, which is seen in Fig. 8b. In TEM mode, the instrument would be stable for a while, suddenly blur for one or two images then quieten down again. In STEM mode, this is recorded as a deflection. The weather changes are also quite simple. A low-pressure cell is typically 16 mbars (1600 Pa) lower in pressure than that of the air before the front. As the front passes through, 160 nm of drift results.

A daily drift forecast for the continental United States can be found at www.rap.ucar.edu/weather/surface/us_ptnd.gif. It is updated hourly. Note that 1 mb h^{-1} results in 10 nm h^{-1} of drift. The pressure changes on a weather map are averages. During a storm, microcells and gust of wind can create rapid pressure fluctuations of 10–30 Pa.

Once these problems were identified, JEOL provided us with a 'clamshell'. This is an airtight airlock cover, shown open and closed in Fig. 9. When the specimen rod is sealed inside the airtight case, the pressure fluctuations are reduced by a factor of 10. This made imaging during the day more practical. Figure 10 shows a sub-0.2 nm resolution image recorded slowly to demonstrate the minimal drift needed for successful atomic resolution spectroscopy.

It must be stressed that the key to the success of the clamshell was that it was airtight. Even a hair on the O-ring seal cancelled its effect, as very little air needs to flow to change the pressure by 1 Pa. A non-airtight cover could in fact make things worse — think of the sound that you hear when putting a seashell to your ear.

Concluding remarks

In addition to the expected sensitivity to AC fields and mechanical vibrations, pressure fluctuations and air movement can also degrade the performance of an electron microscope. Airflows of less than 15 feet min^{-1} are needed for 0.2 nm STEM work. While placing a curtain around the column helps greatly, better insulation of the stage drives and objective lens are needed. Air pressure changes of 1 Pa can result in stage deflections of about 0.1 nm. To prevent this, the sample must be decoupled from the outside atmosphere, either by a clamshell or a sample transfer mechanism. Post-column spectrometers are sensitive to stray fields that enter through the viewing port. For our spectrometer, this has been calibrated at 1 eV l^{-1} of truck engine capacity (or $\sim$1 eV mG^{-1} in SI units). For complete magnetic shielding, the viewing port and camera chamber should be eliminated.

When planning a new microscope installation, the microscope column and power racks should be kept in separate rooms to reduce the temperature gradient and airflow across the microscope column. A large microscope room with a high ceiling will not only improve the temperature stability, but also is a very effective way to isolate the column from electromagnetic interference. Radiant cooling panels can be retrofitted to an existing room and provide increased temperature stability and reduced the required airflow.

Acknowledgements

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