More on the Calibration of TEMs

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There was a recent thread on the microscopy listserver by John McCaffrey (who hosted the discussion on TEM calibration at the 2001 M&M facility managers session) and Richard Beanland, dealing with the calibration of TEMs. This discussion was prompted by a calibration question from John Basgen, who was looking for more precision and more long-lived calibration specimens. The discussion complements and extends the one of the M&M 2001 managers meeting on EM calibration (Microscopy Today, January/February 2002, Issue #02-1), and we are running this separately from that meeting discussion. (MT - ed.)

Richard Beanland: We use cleaved edge specimens of (atomic group) III-V multilayer specimens to calibrate our TEMs. We can measure layer thicknesses in-house to a good accuracy (~0.1%) using high resolution X-ray diffraction, whereas the MAG*I*CAL calibration specimen is (I believe) only guaranteed to ± 2%.

Measurement errors from TEM negatives puts the error in our calibration up to about 0.5%. The thin areas on the samples are minuscule and they are very robust. A drawback is that one needs to be able to tilt the sample to 45 degrees. As discussed some time ago on the microscopy listserver, one needs to be careful to eliminate lens hysteresis effects to make the measurements accurate.

John McCaffrey: Carbon grating replicas, particularly the cross grating replica samples, are probably still the best choice for magnifications between 3,000X and 20,000X. A common cross-grating replica has a line density of 2160 lines/mm, which would give a line spacing on the TEM negatives of 1.39 mm at 3,000X and 9.26 mm at 20,000X. This length would allow measurement of tens of spacings at all of the magnification ranges, and cut the uncertainty down to reasonable levels. While the edge definition of the grating replicas is not the greatest, and while they may need to be replaced every year or two, these calibration samples are still among the simplest to use, as well as being reliable and reproducible. They are also inexpensive.

Another option is latex spheres, which are in the size range that would be useful for these magnification ranges. The downside of these samples is that the spheres have a stated average value, so measurements of many spheres are required to lower the uncertainty to the desired level. Also, there is some anecdotal evidence that these spheres change size when exposed to higher electron doses. If the calibration requirement included the entire set of magnification ranges of a TEM, the MAG*I*CAL sample is still the best bet. Disclaimer: I "invented" the sample, but will attempt to stay objective about it's merits! Since this sample, and all other samples based on crystal lattice spacings, rely on fundamental constants of nature, they are the most accurate and precise samples currently available. However, in spite of this glowing pedigree, crystal-based calibration samples still cannot give measurements with better than ~1% uncertainty.

The comment from Richard Beanland claiming 0.1% accuracy for cleaved III-V multilayers is a bit misleading. His layer thickness confidence is based on X-ray diffraction (XRD) measurements. XRD gives very accurate layer thickness measurements, but these measurements are averaged over enormous surface areas in TEM terms - the diameter of the x-ray beam,

which can be significant fractions of a square mm. This is a very useful measurement for a layer thickness, but does not translate directly into uncertainty in a TEM. When a semiconductor multilayer is viewed in cross section, there are a series of contributors to the layer measurement uncertainty. The state-of-the-art TEMs have resolutions of approximately 0.2 nm. The (002) lattice spacings in most semiconductors (the atomic layers parallel to the surface in most wafers) are a little below 0.3 nm. This means that any measurement of a thicker semiconductor layer in a TEM is going to have a "TEM uncertainty" of approximately one atomic layer at each interface. Producing a semiconductor crystal interface that is perfectly, atomically abrupt (even over the relatively small volume of material used in high resolution TEM) is not trivial, and rarely claimed. Any additional atoms from layer A mixed into layer B, or vice versa, will tend to blur the interface when viewed in cross-sectional TEM. This "epitaxial layer" uncertainty is also approximately one atomic layer at each interface. Highly competent crystal growers can minimize, but not eliminate this interfacial intermixing, allowing the combination of the "TEM uncertainty" and the "epitaxial uncertainty" into one atomic layer at each interface. Semiconductor multilayers can only be grown up to a certain critical thickness, at which point the differences in lattice parameter between the adjoining layers becomes so great that the crystal "relaxes", with the generation of misfit dislocations at the interface. These dislocations need to be avoided in a calibration sample, but the contrasting layer needs to be grown as thick as possible to minimize the influence (*i.e.*, the percentage) of the two points above.

Over an entire semiconductor wafer, there will be systematic thickness variations. That is, layers may be slightly thicker in the middle of the wafer than at the edge. This is a fairly small, but still significant variation, typically less than 1%. Therefore, it becomes important to know where on the wafer a piece of material comes from. So, why can't the "God-traceable" crystal-based TEM calibration samples give measurements with better than ~1% uncertainty? From the combination of uncertainties given above! The thinnest SiGe marker layers in the MAG*I*CAL sample are approximately 10.0 nm thick, and considering their two interfaces to be uncertain in the worse case to approximately one atomic spacing each, this interfacial contribution to uncertainty is about 0.6%. Coupled with the <1% uncertainty across the entire MAG*I*CAL wafer, this gives a total uncertainty of ~2%, with a safety margin built in. Richard Beanland did correctly imply the important point that, if one wishes to internally calibrate one's own sample against a known lattice spacing on that same sample, the uncertainties will indeed be less. However, 1% uncertainty is a better estimate of the 'best' uncertainty available in a TEM calibration sample, even for a sample based on a fundamental constant of nature.

Richard Beanland: I would like to add a couple of points. First, I'd like to make it clear that I do think that the MAG*I*CAL specimen is probably the best commercially available calibration sample. I didn't mean to be disparaging! It's much better than diffraction grating replicas or other commercially available calibration specimens. It beats diffraction grating replicas in that one can use the crystallography to eliminate specimen tilting and is also more stable. Also, both this and the small angle cleavage technique have the advantage that one doesn't need large tilts and can be used in a single tilt holder. The extent to which operators calibrate their microscopes depends on what they use them for - a very accurate relative calibration can be gotten by taking pic-

tures of the same region at different magnifications. We are then only limited by how accurately we can measure features on the negative (0.05 mm in 5 cm is readily achievable with the right specimen and a loupe, which gives 1% relative accuracy). Once we get down to layers below 100 atomic layers thick, then of course it becomes impossible to get better than 1% accuracy with HRTEM. Lack of contrast between the different layers can make things even worse at larger thicknesses for some samples. The 0.1% layer thickness accuracy I quoted was for X-ray diffraction, and for the right sample XRD does beat TEM every time in terms of accuracy. However, most device structures can't be measured using superlattice fringes in XRD and that is where TEM comes in.

Of course, we're only measuring thickness because it is a measure of device performance. Really thin layers, below 100 atomic planes thick, are usually only used as quantum well structures, delta-doping layers, or etch stops, the effectiveness of which can usually be assessed by means more relevant to the device performance than layer thickness (*e.g.*, PL, sheet carrier density, over etching). (I am unaware of any applications which need high accuracy from thin layers apart from microelectronics.)

I do XRD with a 1 mm spot on the sample I use for calibration and I make a cleaved-edge specimen from a region as close as possible to that position. With a 1% thickness variation over a 3" wafer, I'm happy that this is truly representative. With lattice matched III-V layers, they can be grown as thick as desired, and better contrast is obtained in dark field 002 images. The downside is that the samples are less stable chemically and physically than Si/SiGe, which doesn't lend itself to commercial supply (I *certainly wouldn't like to put 100 InP cross sections in the post* with a bet that more than 50% would arrive unscathed).

John P. McCaffrey: I now work for the NRC Institute for National Measurement Standards, which is the Canadian equivalent of your NPL, American's NIST, the "world's" BIPM, *etc.*, so issues of traceability and ISO 17025 take up a lot of my time now. The fact that there is no officially traceable, high-magnification TEM calibration sample available has caused me (and probably most microscopists) some extra headaches. I maintain that TEM calibration samples based on crystal lattice spacing are fundamental constants of nature and hence do not require the blessing of a National Measurement Institute. It does occasionally take a bit of persuasion to convince the bureaucratically-minded that the whole point of traceability is not to refer to SI units through a National Measurement Institute, but to refer to nature through all of the above! As you implied in your original posting, having a crystal as the basis of the sample allows self-calibration at better than the 1% level. Backing that measurement up with XRD is even more convincing. You have a nice advantage in your calibrated measurements in that you can lattice-match your alloy layers, and avoid worries about layer thickness variations as a result of strain. A potential problem with the SiGe marker layers in the MAG*I*CAL sample was that they were strained layers and hence had a slight variation in lattice parameter. We worked around that problem by self-calibrating the thickness of the individual SiGe layer(s) against the pure silicon crystal substrate. Silicon and TEM are a marriage made in heaven. The low atomic number of silicon makes the sample still useful for TEMs with accelerating voltages less than 200 keV. You are also correct about the robustness of Si relative to InP. In my experience, InP crystals will gratuitously cleave under any kind of stress, and usually in exactly the region of interest!

Itís good to discuss the subtleties of TEM calibration. The topic doesn't work it's way into social conversation very often! ■