MAG*I*CAL TEM Calibration Sample

Use Instructions

The MAG*I*CAL® calibration sample for transmission electron microscopy (TEM) has been developed to perform the three major calibrations of a transmission electron microscope: the image magnification calibration, the camera constant calibration for indexing diffraction patterns, and the image/diffraction pattern rotation calibration. The sample consists of an ion-milled cross-section of a silicon wafer on which a series of calibration marks appear. The spacings between these calibration marks are very accurately known.

On the MAG*I*CAL calibration sample, there are four regions that contain the calibration marks. These are marked by arrows in Figure 1 below.

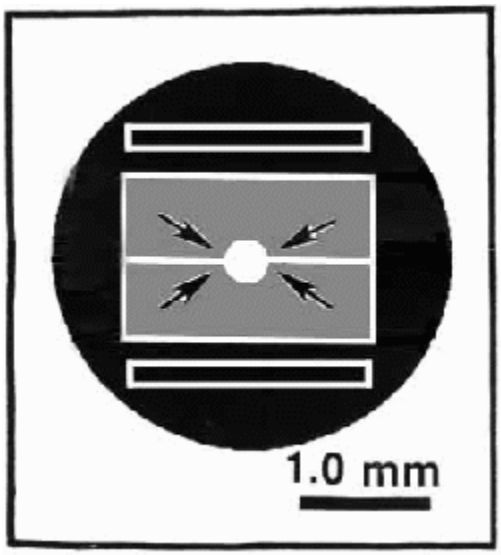

Figure 1: Diagram of the
Mag*I*Cal Calibration Sample

In the above diagram, the arrows indicate the four possible regions of interest on the sample where the calibration marks may be found. Note that this cross-sectional sample consists of two pieces of silicon wafer epoxied face-to-face and held in a titanium grid. To find the areas of interest at low magnification, look for the epoxy line on either side of the small ion- milled perforation (between the arrows in the above diagram).

One of the regions of interest (marked by arrows in Figure 1) is found by translating the sample in the microscope and focusing on the layered structure (Figure 2 below).

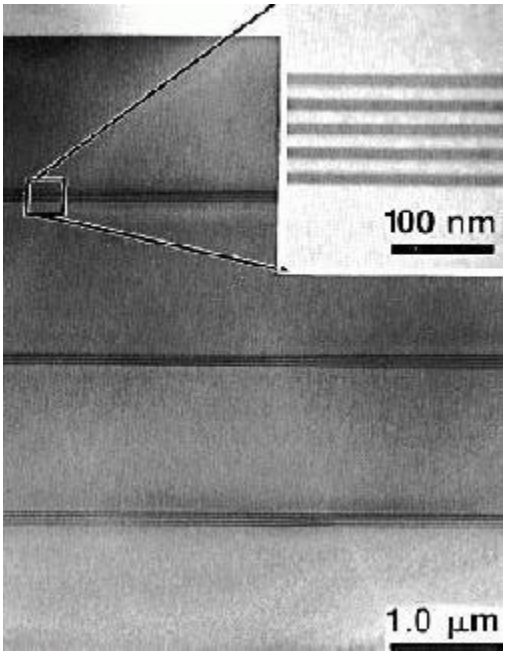

Figure 2: TEM micrographs of the MAG*I*CAL® calibration sample.

The sample consists of a series of layered structures whose thicknesses are accurately known, referenced to the (111) lattice spacing of Si. Inset shows a higher magnification image of one of the layered structures.

Aligning the calibration sample:

The MAG*I*CAL® calibration sample is made from a single crystal of silicon, and therefore has many very useful attributes when used as a TEM sample. To take advantage of these attributes, mount the sample in the TEM sample holder as shown on the instruction sheet and translate the sample to the region of interest. Now translate the sample a small distance away from this region, but stay close to the thin edge of the sample. Put the TEM into diffraction mode, choose the smallest camera length, and adjust the intensity control (condenser control) back and forth to find the proper value of over-focus that forms the striking pattern, called a Kikuchi pattern, which should look similar to Figure 3-b below, but perhaps rotated.

These Kikuchi bands are formed by elastic Bragg scattering of previously in elastically scattered electrons. Note that the intensity control can be adjusted so that either a diffraction spot pattern or a Kikuchi pattern can be observed. The sample should now be tilted to move this pattern so that the central "intersection" in this diagram where the largest number of bands intersect is centered on the brightest (zero order) diffraction spot in the electron diffraction pattern. It may be necessary to translate the sample to slightly thicker or thinner regions to get a clear Kikuchi pattern.

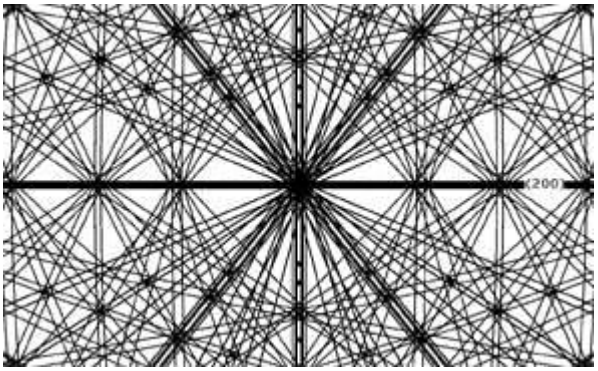

Figure 3-a: Calculated Kikuchi pattern of single crystal silicon viewed down the <011> zone axis. The broad horizontal band in the center of this figure is between the {200} Kikuchi lines.

Figure 3-b: Kikuchi pattern of the [011] zone axis of Si. The arrows indicate the broad band between the {200} Kikuchi lines. When the zero order beam in an electron diffraction pattern is centered anywhere along this band, the electron beam will be parallel to the layered structure , and result in accurate layer thickness values.

Image Magnification Calibration:

One of the regions of interest (marked by arrows in Figure 1) should be found by translating the sample in the microscope and focusing on the layered structure (Figure 2). The eucentric height of the microscope should then be adjusted. On microscopes with no sample height adjustment, the objective lens current must be monitored and kept constant at each magnification value. Switching to diffraction mode, the microscope should be adjusted to produce a Kikuchi pattern at the smallest available camera length (see above, "Aligning the calibration sample").

When using a double-tilt sample holder, the sample should be tilted so that the zero order diffracted beam (brightest diffracted beam) is at the center of the [011] zone axis Kikuchi pattern (Figure 3). With a single tilt holder , the sample should be mounted so that the epoxy line (between the arrows in Figure 1) is parallel to the length of the sample holder rod and the sample should be tilted so that the zero order diffracted beam is centered between the two {200} Kikuchi lines, in the region indicated by the two arrows in Figure 3-b. In either case, this will insure that the electron beam is parallel to the layered structure. The eucentric height should again be adjusted, and the microscope focused on the region shown in Figure 2 at the highest magnification range available. When performing a magnification calibration, a series of micrographs

or digital images at all magnification ranges should be taken starting at the highest magnification range and working down to the lowest. Also, the microscope should be initially over focused, then brought into focus. These two precautions will help to avoid lens hysteresis effects. Likewise, when making a measurement based on this calibration, the image should be focused at a higher magnification, then lowered to the magnification level of interest before producing the micrograph or digital image.

The magnification calibration at the highest magnification ranges (>400,000) can be accomplished by forming a lattice image of the Si below the layered structure (Figure 4), measuring perpendicular to a number of (111) lattice fringes then dividing by the number of fringes. This gives the measurement on the micrograph or digital image that corresponds to the (111) lattice spacing of Si (0.3135428 nm). Alternatively, one of the layered structures could be imaged, and the measured value compared to the "Layer Thickness Values". The microscope magnification can then be lowered through the complete range of magnification steps, taking micrographs or digital images and comparing these measurements with the actual thickness values, as given in the "Layer Thickness Values" table.

mag5 Caption: Figure 4: Lattice image of Si viewed down the [011] zone axis. Note the central diamond shape. The lines marked parallel to the upper two sides of the diamond shape indicate (111) planes, with an interplanar spacing of 0.3135428 nm. Note at the lower right of the diamond shape that measurements must be made perpendicular to the (111) planes, since measuring along a row of atomic columns will give an erroneous value.

Camera Constant Calibration:

After the sample is aligned parallel to the layered structure (preferably down the zone axis), translate the sample to a defect-free region below the layered structure, note the magnification value, and switch to diffraction mode. Use a selected area diffraction aperture and record silicon diffraction patterns at each magnification/camera length of interest. To arrive at the value of the camera constant, measure the length of a series of spots and divide by the total number of spacings (for example, measure on the diffraction pattern micrograph the distance from the (222) spot to the (222) spot and divide by 4; see fig. 5). This gives an accurate value of the diffraction ring radius R in mm. By then using the equation:

$$\mathbf{W\ L = dR}$$

and using the value for **R** from the above procedure as well as the lattice spacing value of **d**. (in this case the (111) spacing of Si, 0.3135428 nm), one can calculate the camera constant value **WL** in units of nm-mm. This value, calculated at all camera lengths of interest, is essential for indexing electron diffraction patterns. To now find

an unknown atomic spacing from a diffraction pattern, measure the unknown diffraction ring radius or a diffraction spot spacing in mm. Then divide the appropriate camera constant value by this spacing. The result is the unknown atomic spacing value in nm.

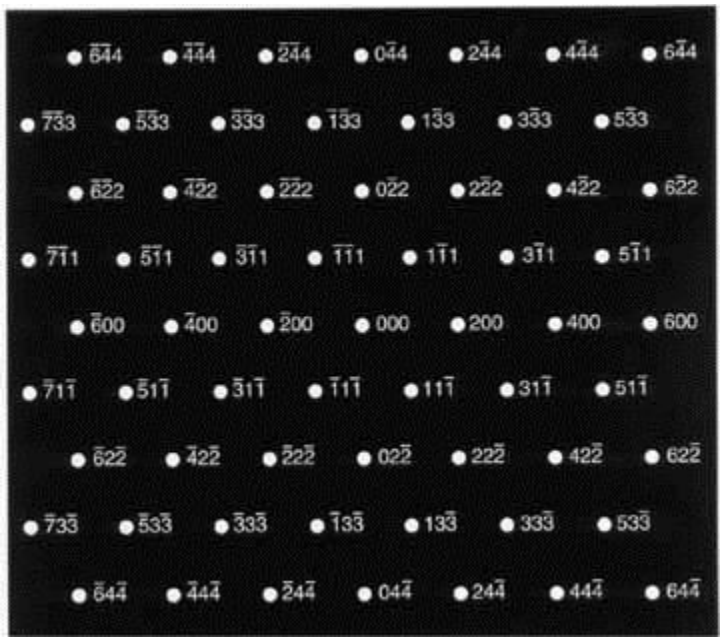

Figure 5: Electron diffraction pattern of Si viewed down the [011] zone axis, with the diffraction spots indexed.

Image-Diffraction Pattern Rotation Calibration

As magnetic lenses in electron microscopes produce a rotation between the image and the object itself and since the diffraction pattern is always produced with the same lens excitation, it is useful to know the various angles between the image and diffraction pattern at each magnification value and camera constant. This is useful for identifying Burgers vectors of dislocations in crystals and identifying the orientation of crystallites. This calibration sample is designed so that the (200) planes are parallel to the layered structure and to the surface. Silicon has the diamond cubic crystal structure, so on this calibration sample the [200] direction is perpendicular to the layered structure and to the surface. What is now required is to measure the angle between the [200] direction on the image and the (200) diffraction spot of the diffraction pattern on the micrograph by means of a series of double exposures.

To produce the image-diffraction pattern rotation calibration with the MAG*I*CAL® calibration sample, align the sample so that the incident electron beam is parallel to the layered structure (see the sheet titled "Aligning the calibration sample..."), then take a series of double exposures of the image and its diffraction pattern at each magnification value and camera constant value of interest. Now identify the (200) diffraction spot on the micrographs. On the diffraction pattern portion of the double exposures, there are two possibilities for the (200) diffraction spot 180 degrees from

each other (ie: which is the (200) spot and which is the (200) spot? See Figure 5.) To identify correctly the (200) diffraction spot, slowly translate the sample through the layered structure and past the top surface while viewing the diffraction pattern. As the sample is translated past the top surface into empty space, the diffraction pattern will fade and finally disappear first from the direction corresponding to the (200) diffraction spot. By referring to Figure 5, all of the diffraction spots can then be indexed.

To measure the actual rotation calibration values, draw a line from the zero order diffraction spot up through the (200) spot. Also draw a line from the zero order spot in a direction perpendicular to the layered structure. The angle between these two lines is the rotation calibration value for this magnification/camera length condition, and can now be used to identify crystal direction in unknown samples, when the images and diffraction patterns of the unknown material are taken at the same magnification/camera length condition.

Some Warranty Limitations

The MAG*I*CAL TEM Calibration Sample is very useful and easy to use. Even thought it has been ion milled and is therefore presumed to be quite fragile, it is nevertheless surprisingly rugged, but if the regions of interest are even slightly touched with even the finest tweezers they may cleave off and destroy that part of the sample. Please handle it carefully because SPI Supplies will not replace free of charge samples damaged by improper handling.

In some TEMs, where the grid cap is not so well aligned and fabricated to fit over the grid holder, to secure the grid, it is possible to over-tighten the grid holder cap, causing a level of compressive stresses that prove fatal to the MAG*I*CAL. Again, do exercise caution in this step as well because SPI Supplies will, similarly, not replace, free of charge samples damaged by over-tightening of the grid cap onto the grid holder.

SPI wishes you good luck in your work and using the MAG*I*CAL TEM Calibration Sample!