The Colors of Silicon

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When silicon samples are thinned and backlit, they display a series of colors; from deep red in the thicker (~5 – 10 μm) regions, to orange and yellow in thinner regions, to effectively transparent in the thinnest regions. This colorful effect can be used as a technique for monitoring the thickness of cross-sectional samples prepared for transmission electron microscopy (TEM).

As seen in Figure 1, a cross-sectional TEM sample of a thin, indium tin oxide (ITO) film on glass was being investigated. A sample was prepared for low-angle ion milling by the Barna method[1,2], with the addition of an adjacent piece of silicon for a thickness indicator. The colors of silicon could then be monitored to indicate when the ITO/glass sample was thin enough for TEM examination. In the figure, the arrow indicates the region of interest on the ITO/glass sample. This figure was produced by photographing the entire TEM sample through an optical microscope, backlit with a standard quartz-halogen illuminator.

The correspondence of colors to different silicon thicknesses has recently been directly measured[3,4], and a plate displaying this color/thickness correspondence is given in Figure 2. Please note: color reproduction is difficult at the best of times, so consider these colors to be approximations. Exact color coordinates are given in references [3] and [4]. This particular sample was backlit using a standard quartz-halogen illuminator, operating at the manufacturer's recommended intensity (color temperature). This is an important detail to note when relying on the variations in silicon color to monitor thickness. The colors can shift for different thicknesses of silicon with variations in the color temperature of the light source, *i.e.*; varying the intensity or the type of your light source can cause minor color changes. The good news is that these variations produce maximum shifts of only approximately 1 μm in thickness for most light sources that are commonly available. By operating illuminators at the recommended intensity, the silicon thickness/color comparison remains a useful technique.

Another related, but more qualitative, thickness-monitoring technique involves the observation of optical interference fringes in the thinnest (<2 μm) regions of the thinned silicon sample. Interference fringes are produced by constructive and destructive interference between transmitted light and light that has undergone multiple internal reflections, as illustrated in Figure 3. Many rays enter the silicon wedge through the first air-silicon surface. While some rays pass completely through the wedge, others undergo multiple internal reflections. When the addition of all phases of all exiting rays of a particular wavelength cancel each other out (interfere destructively), a dark band (*i.e.*, no color) will appear. When these rays interfere constructively, a colored band will appear. Note that green, red and yellow bands can be seen in the thinnest regions of Figure. 3. Because standard microscope illuminators produce a range of wavelengths, relating fringe spacings to thickness is difficult because of the uncertainty of knowing which wavelengths are responsible for which fringes. Single-wavelength laser light could be used as a measurement tool, but is not practical for standard TEM sample preparation. However, the presence of interference fringes is still a reliable indicator that the sample is, or is very close to being, electron transparent.

Monitoring the color of the light transmitted through a silicon sample and observing interference fringes can be helpful techniques for monitoring the ion milling progress of cross-sectional TEM samples. It can be particularly useful when trying to prepare TEM samples of selected areas, where stopping the ion-milling at a critical point is essential. The colors are also a delightful and welcome addition to the typically grey-scale world of TEM microscopists!

For more information on the sample preparation technique shown in the first figure, please see references [1] and [2]. For information on colors and interference fringes see references [3] and [4]. You are also welcome to email john.mccaffrey@nrc.ca for further info or reprints.

1. Barna, A. (1992) Topographic kinetics and practice of low-angle ion beam thinning. In: *Specimen Prep. for TEM of Materials - III*. Mat. Res. Soc. Symp. Proc. Vol 254, eds. R. Anderson, B. Tracy and J. Bravman, pp. 3-22.
2. J.P. McCaffrey, A. Barna. "Preparation of TEM samples for low-angle ion milling", Microscopy Research and Technique, 36(5), pp. 372-377 (1997).
3. J.P. McCaffrey, B.T. Sullivan, J.W. Fraser, D.L. Callahan. "Use of color to calibrate the thickness of silicon samples during etching and thinning processes". Micron, 27(6), pp. 407-411, (1997).
4. J.P. McCaffrey and J. Hulse. "The use of transmitted color and interference fringes for thickness determination of silicon", Micron 29(2-3). pp. 139-144 (1998).

Figure 1: Cross-sectional TEM sample of indium tin oxide (ITO) on glass, prepared with an adjacent piece of silicon used as a thickness indicator

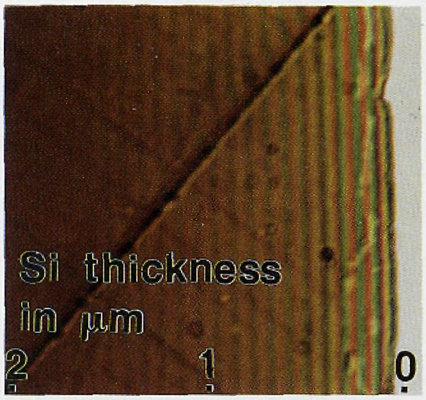

Figure 3: Interference fringes in the thinnest region of a thin, backlit wedge of silicon.

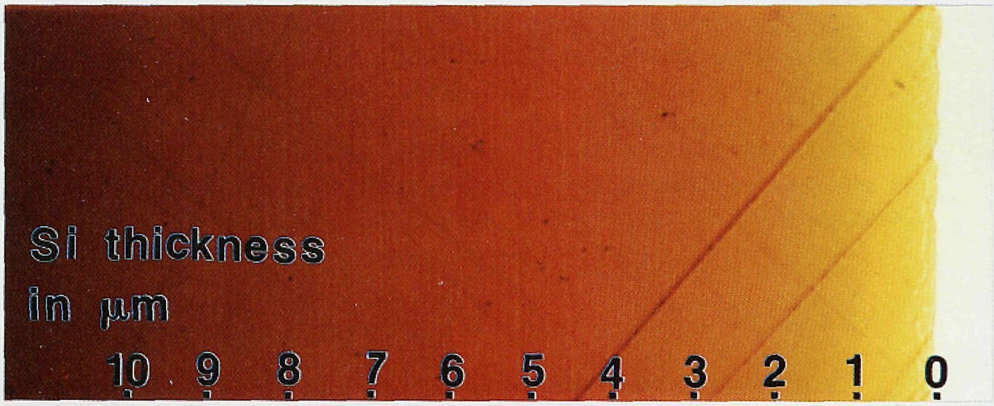

Figure 2: The colors of a thin, backlit wedge of single-crystal silicon. The dark, diagonal lines are cracks along silicon (III) crystal planes. The lighter lines and spots are polishing scratches and dust particles, respectively