# Gold foil thickness measurment

## Sample preparation

Gold foil thickness was measured using a FESEM technique. A gold foil that was manufactured in the same batch as the target foils was mounted to a silicon substrate. For the thinnest foils, static electricity was sufficient to adhere the foil to the substrate. For the thicker foils, Aerodag carbon suspension was used as a conductive adhesive, and was either sprayed on the substrate then the substrate set on the foil or the foil was set on the substrate and a drop of the liquid suspension was applied to the foil around the edges. The substrates were then cleaved using a curved Exacto knife at the edge, and the foils were cleanly broken, exposing the cross sectional edge of the foil. The thicker foils sometimes didn’t break with the cleaving, so we had to ??? (Mamun?)

## Measurement

Once the substrate had an edge available, it was loaded into a FESEM(ref). Images were taken at different magnifications, between 10k and 150k depending on the foil thickness and the ability to get the thickness into the frame of the image. The working distance was adjusted until a crisp focus was achieved, and this working distance varied between 10 and 14 mm. Images were made at one at a single location for each sample prepared. For two of the samples, to test uniformity, the sample was translated and 3 or 4 spots were measured along the edge of a sample. Additionally, in two instances, two samples were prepared from a single target foil, one near the center and one near the edge, and both were analyzed. Finally, the tilt dependence of the mounting in the FESEM was studied for one foil.

## Image analysis

The software program “ImageJ” was used to determine the foil thickness from the FESEM images. For each image, the measurement gradation line was used to set the scale between nm and pixels, and measurements of the distance between the top and bottom of the foil were made using the built in measurement feature of the software. Depending on the quality of the image, the enhancement features of the software, including “edge find” and “sharpen”, as well as rotation correction were used to assist in the process of determining the edge of the foil in the image. An analysis for the effect of sample tilt was also performed, intentionally varying the angle of the sample holder in the FESEM by angles of -1.7 through +2.5 degrees and measuring the variation due to that change.

## Results

The summary of the measurements of the foils are listed in table 1. The foils from Lebow are each labelled with a unique 4 digit identification of the batch in which they were made. Typically four foils were purchased from each batch, each nominally with the same thickness, and designated with the letters A through D following the numerical batch identifier. These are referred to as sibling foils. One of the foils, 50 nm foil 6845, did not have a sibling foil available for analysis. Two of the target foils, ladder positions 8 and 14, were both siblings of the same measured foil, 5613A.

## Error sources and error analysis

Uncertainties for the foil thickness measurements can come from the following sources: (discussion follows for many of the less obvious sources)

1. Inherent limitations of the sample preparation and measurement in the FESEM
   1. FESEM resolution: 1.2 nm machine specifications
   2. Image tilt
   3. Uncertainty in magnification/working distance
2. Limitations on analyzing the image
   1. Pixel resolution: ± 4 pixels estimated, varies from ± 2.5 – 40 nm depending on scale
   2. Reproducibility between different analyses of same image
   3. Differences between images that should be the same
   4. Differences between images that should be similar (tilt, translation)
3. Errors or problems introduced in the preparation of the samples
   1. Sample mounting flatness, uniformity
   2. Any deformation during cleaving? Did it snap nicely or pull?
4. Uncertainties that have to be bounded by Lebow specifications
   1. Uniformity across entire sample: quote <= 2%
   2. Uniformity between samples within a batch: quote <= 5%

Discussion of uncertainty contributions:

### FESEM measurement uncertainties

1b: Tilts (pitch only) of up to ±5° were intentionally introduced. The images where the tilt angle (pitch) of the image was not zeroed out or recorded all seem to be within this ±5° range – we don’t appear to have any images at extreme angles. For a tilt of 0°, we’d expect to measure 100% of the full foil thickness in the image. For a tilt of ±5°, we’d expect to measure 99.6% of the actual thickness of the foil. Therefore, we have up to a 0.4% error factor for the uncertainty in tilt to all the foils to bound this error.

1c: When tilts (pitch) adjustments were made, the working distance to the foil will change slightly. This can be calculated using cosθ=(L-d)/L, where θ is the tilt (pitch) adjustment angle, L is the distance from the pivot to the front edge of the foil and d is the change in the working distance for the FESEM, which affects the focus and magnification calculations. For tilts up to ±5°, and with a distance to the sample stage pivot overestimated at 25 mm, we find that the difference in the working distance is (1-cosθ)\*L or less than 0.1 mm, which is the resolution of the working distance focus adjustment in the system. For working distances approximately 10 mm, this could be up to a 1% correction factor in the magnification and therefore the scale of the image.

1d: For many of the images, particularly the higher magnifications, they are not in sharp focus. This implies that the corrections for working distance weren’t fine enough or there weren’t adequate iterations of working distance adjustment to get the sharpest focus. This can be quantified as a ±1-2 “clicks” on the focus adjust knob uncertainty, or again with resolution in the 0.1 mm in the working distance knob and typical working distances of about 10 mm, as a 1-2% effect. (calculated as 1 gradation of focus knob, which is 0.1 mm, divided by recorded working distance, on the order of 10 mm).

### Image analysis uncertainties

The image analysis, conducted with the imageJ software package, requires that the scale be set by the user from the length scale bar on the image, then will capture the length of lines drawn by the user to across the image to determine the thickness of the foil. Uncertainties can be introduced in several ways:

2a: the lines drawn require that the edge of the image be precise – after multiple analyses, the user estimates that these line lengths are correct on each end to no better than 2 pixels, giving up to a ±4 pixel uncertainty on line length. Additional care was taken to get the line length for the scale factor on each image good to within one pixel – this should also be accounted for here and can shift entire images slightly. The pixel to nm conversion is provided in the software. \*\*Note – this has been assumed constant across images with different magnifications and still needs to be addressed\*\*

2b: The same image was analyzed multiple times. These have different methods employed – some used edge find, some used evenly spaced lines for measurements, some made as many measurements as were deemed good. The weighted average of these measurements is used for each sample to give a reproducibility error. (the average percent difference for this has been used right now)

2c: Some of the foils have multiple images of the same area at nominally the same tilt and location: perhaps focus or magnification was changed, but these should yield the same value since it is the same foil. The standard deviation distribution of these is accounted for in 2c.

2d: Some of the foils have images made of different spots. These are either from different foil samples, for example an area of the foil near the center and an area of the foil from near the edge, or from translations along one foil sample. The different samples are designated as “center” or “edge” and the different positions on sample are denoted by “spot 1”, “spot 2” etc.. The spread in these measurements gives an estimate both of the uniformity of the samples, which should be quite uniform given the less than 2% foil thickness uniformity as quoted by Lebow, and also gives a measure of the reproducibility of the measurements of a foil with the FESEM technique. We’ll break down the reproducibility to translation, different samples, and sample tilts to get a same foil, different method reproducibility.

3a: Sample mounting flatness and uniformity need to be considered and assigned an uncertainty. Not sure how to quantify this

3b: if we are systematically stretching or pulling the foil when we try to cleave the gold along with the silicon substrate, we should assign a value to this: it will only make them thinner, and we see a trend toward all the thicker foils measuring thinner than the Lebow quote. This systematic could affect the fit of the line.

4a: Lebow quotes the non-uniformity across each foil at less than 2%. If we are getting non-uniformities in 2d greater than 2%, it is likely our technique rather than the foils and we need to reconcile these numbers.

4b: Lebow only guarantees that sibling foils are identical to less than 5%. We could have cases where this could be our leading cause, and if we find elastic rates that deviate significantly from the FESEM measurements, this could be a factor.

Quantifying sources of measurement uncertainty for each foil thickness (all data in nm)

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| foil | Lebow (nm) | Meas (nm) | 1a: FE-SEM res. | 1b: tilt (0.4% of meas thick) | 1c: working distance due to tilt | 1d: working distance or focus – 0.1 mm/ WD | 2a: pixel res. | 2b: re-analy. same image | 2c: diff btwn similar images | 2d: varia. btwn images of a foil | 3: ??? stretch, foil mount issues | 4a: lebow foil uniform 2% | 4b: Lebow batch uniform. 5% |
| 5385 | 1000 | 943.7 | 1.2 | 3.7 | 9.2 | 8.5 | 40.0 | 31.0 | 29.0 | 6.5 |  | 20.0 | 50.0 |
| 3057 | 870 | 836.8 | 1.2 | 3.3 | 8.3 | 8.3 | 8.0 | 13.8 | 7.1 | 5.6 |  | 17.4 | 43.5 |
| 5134 | 750 | 774.6 | 1.2 | 3.1 | 7.7 | 6.9 | 10.0 | 13.2 | 9.1 | 9.1 |  | 15.0 | 37.5 |
| 7028 | 625 | 561.2 | 1.2 | 2.2 | 5.6 | 4.6 | 8.0 | 12.6 | 8.0 | 8.0 |  | 12.5 | 31.3 |
| 5275 | 500 | 487.6 | 1.2 | 2.0 | 4.9 | 4.3 | 8.0 | 8.4 | 3.4 | 2.9 |  | 10.0 | 25.0 |
| 5613 | 355 | 389.4 | 1.2 | 1.6 | 3.9 | 3.4 | 8.0 | 6.6 | 4.5 | 4.5 |  | 7.1 | 17.8 |
| 7029 | 225 | 215.2 | 1.2 | 0.9 | 2.2 | 1.9 | 2.6 | 2.7 | 1.9 | 1.9 |  | 4.5 | 11.3 |
| 6809 | 50 | 52.0 | 1.2 | 0.2 | 0.5 | 0.4 | 2.6 | 3.2 | 2.3 | 2.3 |  | 1.0 | 2.5 |

Combining sources of error (all measurements in nm)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Target ladder position | foil | Meas (nm) | FESEM (all from section 1) | image anayl (all from section 2) | Lebow (from section 4: larger only) | Total uncertainty (nm) |
| 15 | 5385 | 943.7 | 13.1 | 58.7 | 50.0 | 78.2 |
| 3 | 3057 | 836.8 | 12.3 | 18.3 | 43.5 | 48.8 |
| 4 | 5134 | 774.6 | 10.9 | 21.0 | 37.5 | 44.3 |
| 2 | 7028 | 561.2 | 7.7 | 18.7 | 31.3 | 37.2 |
| 5 | 5275 | 487.6 | 6.9 | 12.5 | 25.0 | 28.8 |
| 8, 14 | 5613 | 389.4 | 5.5 | 12.1 | 17.8 | 22.2 |
| 1 | 7029 | 215.2 | 3.2 | 4.6 | 11.3 | 12.6 |
| 13 | 6809 | 52.0 | 1.4 | 5.3 | 2.5 | 6.0 |

This fit is from Excel – probably not as good as Daniel’s root fits.

|  |  |
| --- | --- |
| ToF cut: | Gaussian fit of target ToF peak, cut made between -2 sigma and 2 sigma |
| E cut: | gaussian fit of ToF-cut energy spectra, cut made between -1 sigma and 2 sigma |