# Gold foil thickness measurement

## Sample preparation

Gold foil thickness was measured using a field emission scanning electron microscope (FESEM) technique. A gold foil that was manufactured in the same batch as the target foils was mounted to a silicon substrate. For the nominally 50 nm foils, static electricity was sufficient to adhere the foil to the substrate. For the thicker foils, a commercial carbon suspension in alcohol, Aerodag™, was used as a conductive adhesive between the foil and the silicon crystal. The Aerodag 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 edge of the foil. The samples were then cleaved by applying pressure to the edge of the silicon substrate with a curved blade, cleaving the silicon and separating the gold foil to expose a thickness cross section for SEM imaging.

## Measurement

A Hitachi s-4700 FESEM model using an electron energy of 15.0 kV was used image the gold foil edges. Magnifications between 10k and 150k were used depending on the foil thickness. The working distance was varied until the image was in focus, and varied between 10 and 14 mm. Images were typically made at a single location for each sample prepared. For two of the samples, to test uniformity across a small area of the foil, the sample was translated and 3 or 4 spots were measured along the edge of a sample. Additionally, for two foils, two FESEM samples were prepared from a single target foil, one near the center and one near the edge, and both were analyzed. Finally, the tilt (pitch) dependence of the mounting in the FESEM was studied for one foil.

## Image analysis

The software program “ImageJ[[1]](#endnote-1)” 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. A systematic study was also performed to determine the effect of sample tilt, 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.

## Error sources and error analysis

#### Statistical Uncertainties

Uncertainties for the foil thickness measurements can come from the following sources both statistical and systematic uncertainties, as listed here.

Statistical uncertainties include the variation in the thickness measurement obtained from images of the same spot on a film, which may vary in magnification or working distance. Additionally, repeated analysis of the same FESEM image using different techniques in the ImageJ software, such as edge find on or off, evenly spaced or clearest positions only for the lines yielded another uncertainty for the reanalysis of the same image. These are quantified in Table 1, rows 3 and 4.

#### Systematic Uncertainties

Systematic uncertainties in the foil measurements include inherent limitations of the FESEM machine, uncertainties introduced in the image analysis for foil thickness calculation, and uncertainties or inaccuracies introduced through the foil mounting technique.

The inherent resolution of the FESEM is 1.2 nm, and this uncertainty is the first systematic uncertainty considered in row 5 of Table x.

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. The precision of assigning endpoints to the line measurements is no better than ~2 pixels at each end of the measured line, adding a ±4 pixel uncertainty on the measurement of the thickness of the foil, as shown in Table 1 row 6.

The tilt uncertainty of the measured FESEM images is shown in row 7 of Table 1. To measure the full thickness of the gold foil, the image must be made with the sample edge perpendicular to the incoming SEM electron beam (with normal incidence defined as 0°). This was not tightly controlled in the images of the first few foils measured, and some of the images appeared to have a non-zero angle. To test the dependence of this effect, tilts (pitch only) of up to ±5° were intentionally introduced for two of the measured foils. Comparing the intentionally tilted samples to the inadvertently tilted samples, none of the inadvertently tilted samples exceed this ±5° tilt. Therefore, due to tilt, the measured thickness of the foil would be 99.6% of the actual thickness of the foil. We have added a 0.4% error factor for the uncertainty in tilt to all the foils to account for this measurement uncertainty.

For the FESEM, the working distance is adjusted to best focus. To account for inaccuracies in the focus of the images, a systematic error of 0.1 mm (the resolution of the working distance adjustment) out of the 10 mm typical working distance adds a 1% uncertainty due to the instrument focus resolution, shown in Row 8.

Rows 9 and 10 of Table 1 give the manufacturer’s specifications for the thickness variation across a single foil and the thickness variation possible between sibling foils. Translation studies, where a sample was measured at different translation of the same FESEM sample, and larger studies where different FESEM samples were prepared from near the edge and the center of a gold sibling foil, were consistent within the resolution of the measurements with the assertion that the FESEM thickness varies by no more than 2% across the sample. Since studies were not performed for all sample, the manufacturer’s estimate of no more than 2% variation across the foil was used for this estimate.

The target foils used in the Mott study were manufactured in the same batch as the samples mounted for FESEM measurements, but there is a guarantee only that these sibling are consistent with the target foils to 5%. This comprises the largest systematic uncertainty in the target foil measurement at 5%, listed in Row 10 of Table 1.

#### Additional uncertainties

Uncertainties that have not been quantified are any difference between the thickness of the foil as mounted and any difference introduced by the mounting and cleaving process. This could be significant, but comparison difficult to quantify. Additional extrapolation with rate rather than measured target thickness is done in section \_\_\_ to help account for any systematic discrepancies in the foil mounting, cleaving, imaging and measuring process.

## Results

The summary of the measurements of the foils are listed in Table 1. The target foils are each labelled with a 4-digit batch identification, and “sibling” foils from the same batch are distinguished by suffixes from A-D. 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.

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  |  | Au\_5385\_B | Au\_3057\_C | Au\_5134\_B | Au\_7028\_B | Au\_5275\_C | Au\_5613\_D | Au\_7029\_B | Au\_6809\_B |
|  | *nominal thickness (nm)* | *1000* | *870* | *750* | *625* | *500* | *355* | *225* | *50* |
|  | **mean thickness (all data, nm)** | **943.7** | **836.8** | **774.6** | **561.2** | **482.0** | **389.4** | **215.2** | **52.0** |
| Stat. | Stdev, nom. identical data (nm) | 29.0 | 7.1 | 9.1 | 8.0 | 9.7 | 4.5 | 1.9 | 2.3 |
|  | stdev image reanalysis (nm) | 22.5 | 7.7 | 9.4 | 7.5 | 4.0 | 2.7 | 1.8 | 2.1 |
| Syst. | Image analysis: ± 4 Pixel | 20.0 | 8.0 | 10.0 | 8.0 | 8.0 | 8.0 | 2.6 | 2.6 |
|  | Resolution (1.2 nm inherent) | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 |
|  | Tilt (0.4%) | 4.6 | 4.2 | 3.9 | 2.8 | 2.5 | 1.9 | 1.1 | 0.3 |
|  | Focus (1%) | 9.4 | 8.4 | 7.7 | 5.6 | 4.8 | 3.9 | 2.2 | 0.5 |
|  | Different spots (Lebow: 2%) | 18.9 | 16.7 | 15.5 | 11.2 | 9.6 | 7.8 | 4.3 | 1.0 |
|  | Sibling difference (Lebow:5%) | 47.2 | 41.8 | 38.7 | 28.1 | 24.1 | 19.5 | 10.8 | 2.6 |
| Totals |  |  |  |  |  |  |  |  |  |
|  | stat uncertainty (nm) | 36.7 | 10.5 | 13.1 | 11.0 | 10.5 | 5.2 | 2.6 | 3.1 |
|  | syst uncertainty (nm) | 55.6 | 46.7 | 43.8 | 31.9 | 27.7 | 22.9 | 12.2 | 4.1 |
|  | **total uncertainty (nm)** | **66.6** | **47.9** | **45.7** | **33.7** | **29.6** | **23.5** | **12.5** | **5.1** |

1. ImageJ software site [↑](#endnote-ref-1)