August 26, 2019 – 4D STEM session for sample MV\_1.5\_9 (1.2° TBLG)

Summary:

**Probe Size Measurements**

The first half of this session was spent determining the probe size under various conditions at 60 kV. The goal was to begin generating a table of probe sizes (FWHM and 80% of probe) based on convergence angle, aperture size, spot size, etc. The probe images can be found under the folder “Probe images 60 kV”. At 1 mrad convergence angle for spot 9 and a 40 um aperture, a minimum probe size of 1.6 nm (FWHM)/2.8 nm (80%) was achieved. Under these same conditions but using a 1.5 mrad convergence angle, the minimum probe size was 1.15 nm (FWHM)/ 2 nm (80%).

**Diffraction Measurements**

Since the moiré unit cell size for 1.2° is 11.7 nm, we started with the 1 mrad convergence (~1.6-1.8 nm FWHM, 2.8-3.1 nm at 80%). The uncertainty in probe size comes from the fact that at these larger convergence angles, even a small (50-100 nm) shift out of focus can significantly change the probe size. The thought was that if we can resolve these disks, then we could try with even larger Bragg disks (smaller probe) next time if we need better real space resolution.

We collected three sets of data. The first (file 1) was a test to see how long data collection would take when binning by 1. File 2 is probably the most useful file collected. We used the same conditions as in File 1, but increased the exposure time to improve signal:noise and increased the scan area. File 3 would have been the best data set since we increased the exposure time again and decreased the spot size (higher electron flux), *but* we realized that the probe was elliptical after collection. The resulted from changing the spot size and not having time to recheck the beam stigmation. For now, we can work with File 2, which had a circular beam. During the next session we can try again with a higher electron flux and long exposure.

**Collection Parameters for File 2:**

Acc. Voltage: 60 kV

Condenser Aperture: 40 um

Convergence Angle: 1 mrad

Spot Size: 9

Exposure Time: 1 s

Binning: 1

Probe Size: 1.6 – 1.8 nm (FWHM), 2.8 – 3.1 nm (80%)

Step Size: 3 nm

Moiré Unit Cell Size: 11.7 nm

Field of View: Two full rings included in pattern, with beam stop

**Observations:**

Due to the significant overlap in the graphene disks, interference patterns are observed in the overlap regions. Small, uniform crescent moons may be visible in the areas where there is no overlap (particularly in the outer ring). These could allow for individual disk detection; however, it may be to our advantage to see what information comes from these regions with interference. The patterns are not uniform around the rings, and there is an apparent shift from pattern to pattern – maybe because of strain(?).

**Follow-up – here are some routes to consider based on what we find with this set of data:**

1. If we need better real space resolution, we can decrease the probe size by:
   1. Increasing the convergence angle – this will increase the size of the Bragg disks but electron flux doesn’t change
   2. Increasing the spot size – this changes probe size less rapidly, but it also decreases electron flux
2. If we need better signal to noise, we can decrease the spot size and continue increasing exposure time. However, decreasing the spot size does increase probe size, so again we may have to adjust convergence angle.