Silicon Consortium Project: No-Go on Moiré Interferometry for Measuring SEI Strain as a Probe for Calendar Life Testing

Team: *Josefine McBrayer (Sandia National Laboratory, University of Utah), Darwin Serkland (Sandia National Laboratory), Kyle Fenton (Sandia National Laboratory), Christopher Apblett (Sandia National Laboratory), Shelley Minteer (University of Utah), and Katharine Harrison (Sandia National Laboratory)*

Background and Motivation

Silicon is a promising candidate as a next generation anode to replace or complement graphite electrodes due to its high energy density and low lithiation potential. When silicon is lithiated, it experiences over 300% expansion which stresses the silicon as well as its solid electrolyte interphase (SEI) leading to poor performance. The use of nano-sized silicon has helped to mitigate volume expansion and stress in the silicon, yet the silicon SEI is still both mechanically and chemically unstable.[1] Identifying the mechanical failure mechanism of the SEI will help enhance calendar and cycle life performance through improved SEI design. *In situ* moiré interferometry was investigated to try and track the in-plane strain in the SEI and silicon electrode for this purpose. Moiré can detect on the order of 10 nm changes in displacement and is therefore a useful tool in the measurement of strain.[2] As the sample undergoes small deformations, large changes in the moiré fringe allow for measurements of displacement below the diffraction limit of light. **Figure 1a** shows how the moiré fringe changes as the sample grating deforms. As the sample contracts or expands, the frequency of the moiré fringe changes, and this change is proportional to the strain in the sample.

Approach and Results

Model thin foil electrodes were fabricated by spin-coating photoresist on a silicon wafer and then evaporating 50 nm of SiO_2, 50 nm of Si, and 500 nm of Cu on the top of the wafer. Si gratings with a 4 µm period were patterned on the Cu. The in-plane strain is very small due to constraint from the copper current collector on the silicon thin film, so the current collector was made as thin as possible without losing structural integrity. The foils were released by dissolving the photoresist using acetone. The free foils were then adhered to stainless steel washers as mechanical supports using silver epoxy. The SiO_2 layer served as a protective layer between the silicon and photoresist. The samples were cleaned by O_2 plasma and then the SiO_2 layer was removed using 1% v/v HF. The cyclic voltammogram of a successful electrode is shown in **Figure 1b**. The electrodes were used in a house-made *in situ* cell for strain measurements (**Figure 1c**).

The moiré interferometer was built in-house. Both geometric (reference grating projected onto the sample) and interferometric moiré (interference of light used to generate the reference grating) were explored. The moiré interferometer is shown schematically in **Figure 1d**.

Initial experiments using geometric moiré seemed promising as there was a change in the perceived strain as a function of voltage. However, it was determined that the measurement was convoluted by changes in height of the foil relative to the objective. The setup was then changed to a moiré interferometer to achieve a more uniform fringe pattern with changes in height. Despite achieving more consistent measurements with changes in height and a decrease in static noise (**Figure 2a and 2b** respectively), *in situ* variability continued. Through Gaussian beam simulations, it was

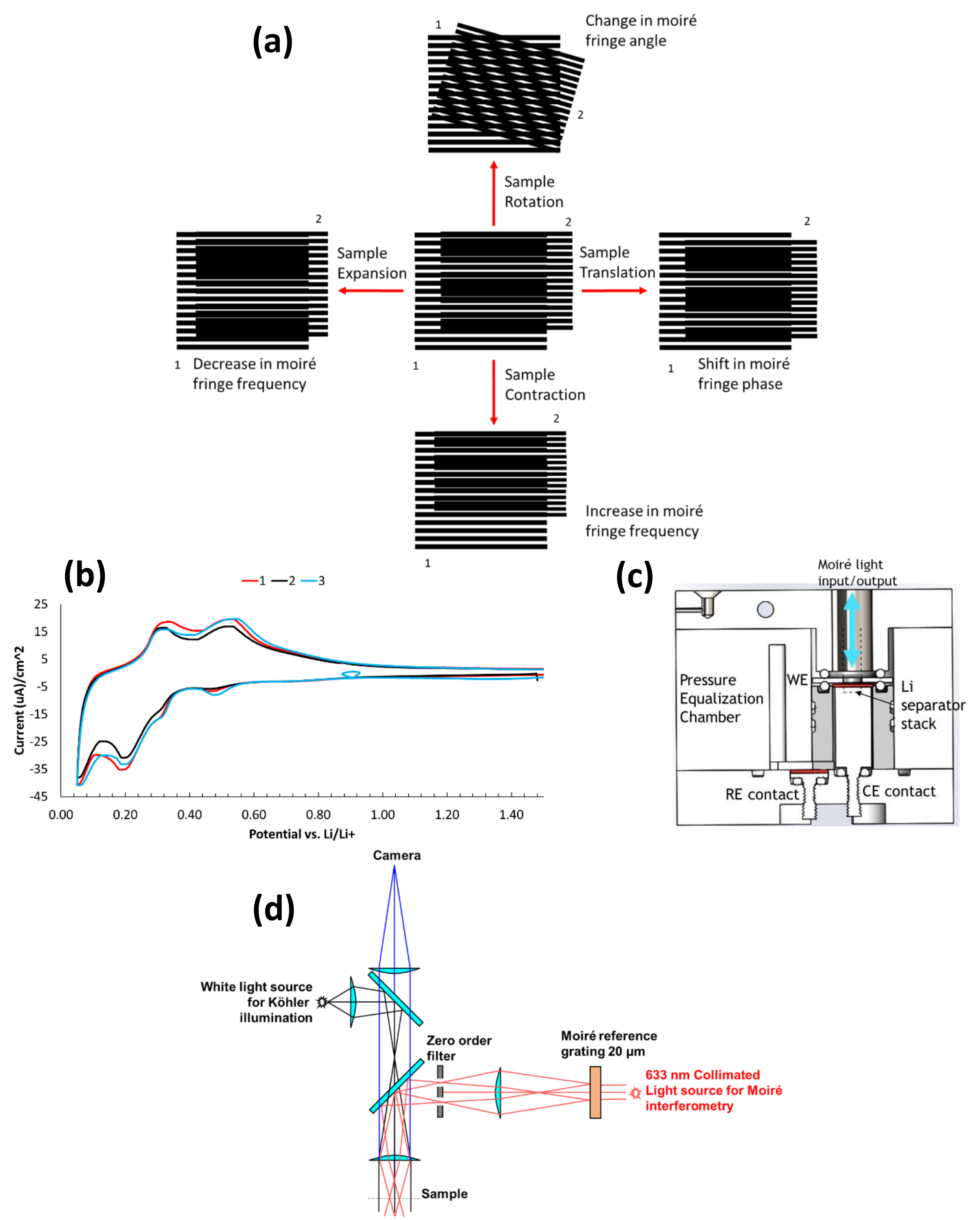

Figure 1: (a) How moiré works with the reference grating labeled 1 and the sample grating labeled 2, (b) CV of 3 replicates of a successful 50 nm Si, 500 nm Cu foil sample, (c) diagram of house-made *in situ* cell, and (d) diagram of house-made moiré interferometer

determined that this was due to sensitivity to tip and tilt of the sample. With perfect alignment, the change in tilt caused a large change in perceived strain behavior making a systematic tilt correct difficult (**Figure 2c**). An auto collimator was built and used to make the sample perpendicular to the objective. However, this became infeasible with the foils *in situ*. The thin foils were used to enhance strain resolution required because of the confinement of the Si from the Cu, but they also made it nearly impossible to maintain a sufficiently flat surface. Because of these limitations, it was determined that moiré interferometry would be very difficult to use as an *in situ* method for measuring strain at the resolution needed for this project.

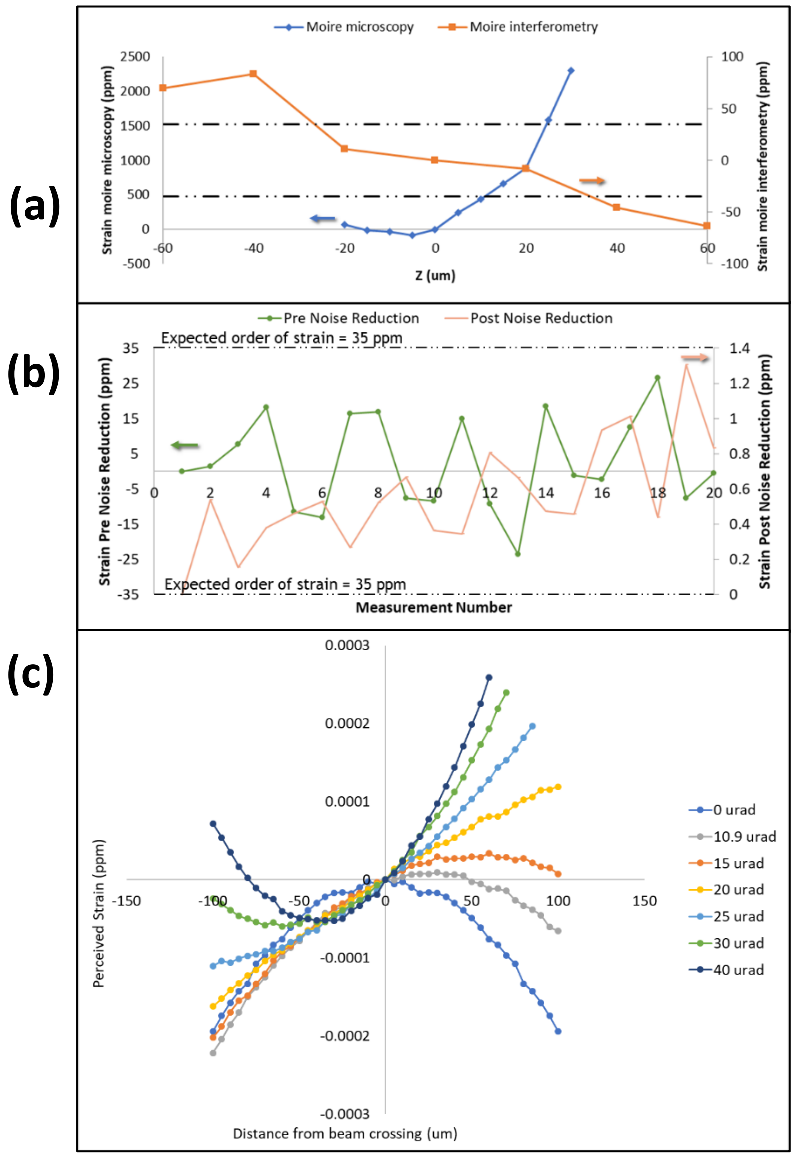

Figure 2: (a) Improvements on noise using interferometry, (b) improvement on z-insensitivity using interferometry, and (c) Gaussian beam simulations of the effect of sample tilt. Similar trends were observed experimentally, but with a larger magnitude.

Decision Outcome: No-Go

The project had a go/no-go decision in quarter three to decide if moiré could probe calendar life in the silicon SEI. The first step to achieving this goal is the ability to measure strain, which could then have been correlated with SEI mechanical failure and the resulting impact on calendar life. Because robust strain measurements in the SEI were unlikely, a no-go decision was made on this technique.

Acknowledgements

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References

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2. D. Post, B. Han and P. Ifju, *High Sensitivity Moire: Experimental Analysis for Mechanics and Materials*, Springer, Verlag New York (1994).