Optical reflectance-based software for automated characterization of nanomaterials: outcomes of the NSF Nanohub's NanoMFG node

Vu Nguyen[1], Wan Li[2], Hannah Gramling[1], Yunsu Park[1], Ke Xu[2], and Hayden Taylor[1,*]

Department of Mechanical Engineering[1] and Department of Chemistry[2]
University of California, Berkeley, CA 94720.

*E-mail: hkt@berkeley.edu

We introduce two software tools to aid in the characterization of nanoscale-thickness materials, and in the development of nanomanufacturing processes based on them. The first tool, *2dreflect*, automatically determines from optical reflectance images the spatial distribution of the number of atomic layers of a 2D material—or heterostructures of multiple materials—on a flat substrate. Scaling up the manufacturing of devices made from atomically-thin 2D materials demands high-throughput, non-destructive characterization techniques to provide insights into the materials' quality and areal yield. Thin-film optical interference, an inherent effect in the observation of transparent layered structures [1], provides a promising pathway to studying 2D materials since it can resolve sub-nanometer thickness differences and can be quickly recorded with a simple optical setup. Although thin-film interference has been widely used to determine the number of atomic layers in experimental samples via white light optical microscopy [2], it requires careful calibration and human judgement when interpreting images. What is needed for more robust detection is an image-processing algorithm to analyze reflected light intensities and determine material composition automatically.

We have built such an analysis software tool, called *2dreflect*, and used it successfully to characterize monolayers, homogenous multilayers, and heterostructures of MoS_2 and WS_2. The tool is based on simulation of how a sample's surface reflectance—relative to that of a bare adjacent substrate—depends on illumination wavelength and layer thickness composition. The well-known transfer matrix formulation is used, and when given one or more reflection images obtained at known wavelengths, the tool tags regions of the image from a specified set of possible layer combinations. We show that *2dreflect* distinguishes reliably between MoS_2 samples ranging from 1 to 4 molecular layers in thickness (Fig. 1). We can also resolve the small differences between bilayer samples that have been constructed from two monolayers by sequential mechanical exfoliation without angular alignment, and bilayers exfoliated directly from a mined crystal, whose layers are expected to be aligned and therefore more closely spaced. We further observe that after thermal annealing, the optical signature of artificially constructed bilayers approaches that of natural bilayers. We have used *2dreflect* to analyze mechanically exfoliated and chemical vapor-deposited material. The software is implemented with a graphical interface and is scheduled to be released on nanohub.org [3] in summer 2019.

The second tool, *nanovisc*, uses color video of a nanoimprint lithography process to determine the effective viscosity of the resist. Using our previously described 'nanocaliper' apparatus [4], a patterned, semi-transparent stamp is pressed into a spun-on film of UV-curing resist by a constant backside pneumatic pressure (Fig. 2a). Reduction of the resist's residual layer thickness (RLT) over time is tracked by imaging the intensity minima and maxima of red and blue LED light that undergoes thin-film interference in the resist and is partially reflected through the stamp (Fig. 2b). The extracted RLT–time relationship is interpreted with squeeze-film theory to estimate resist viscosity, potentially accelerating the screening of resist formulations, pre-bake processes, and UV exposure recipes.

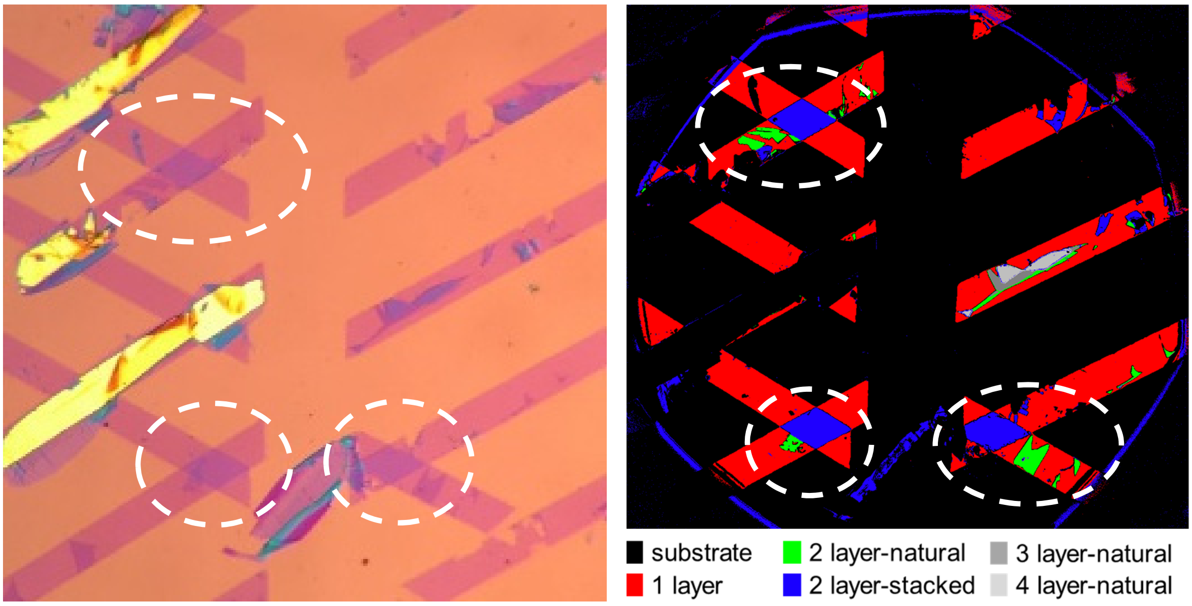
References

[1] *e.g.* W. Li *et al.*, *Nano Lett* **16**, 5027, 2016.

[2] *e.g.* Nolen *et al.*, *ACS Nano* **5** (2), 914–922, 2011.

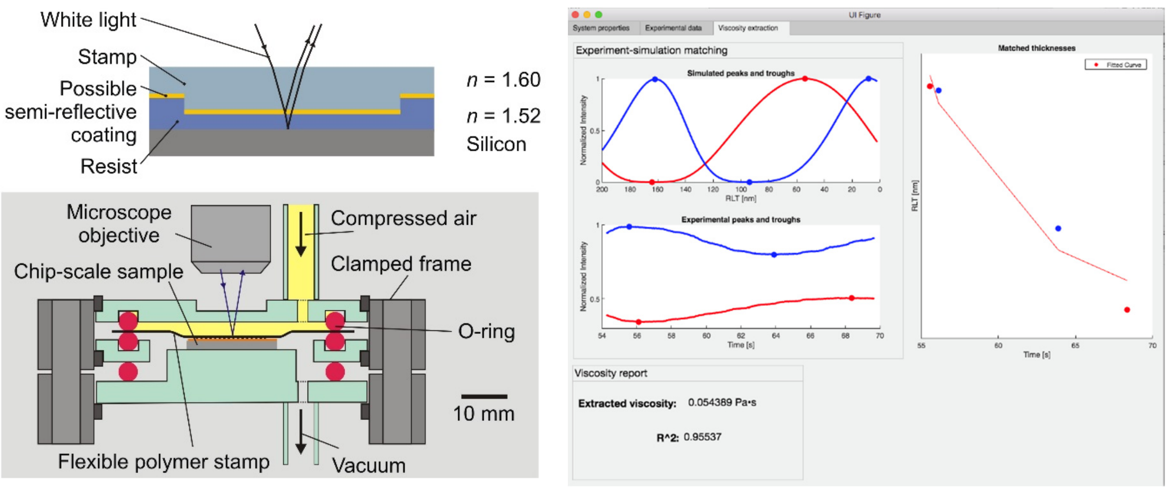
[3] *2dreflect* and *nanovisc* are among the software tools being developed at U.C. Berkeley as part of the NanoMFG node, a five-year partnership with the University of Illinois at Urbana–Champaign, funded via the National Science Foundation's Nanohub.

[4] H. Taylor and N. Dhakal, *Proc. NNT* 2013; H. Gramling and H. Taylor, *Proc. MNE* 2016.



(a) **(b)**

Figure 1. Operation of the *2dreflect* tool. (a) Unprocessed white-light optical microscopy image of two sets of mechanically exfoliated MoS_2 patterns, many of them monolayer, that were sequentially deposited onto an SiO_2/Si substrate Rectangular features are about 40 μm wide. Overlap regions of interest are circled with dashed white lines. (b) Pseudocolor image after analysis by *2dreflect*, where each color corresponds to a particular layer composition according to the legend. Results have been validated with atomic force microscopy and photoluminescence imaging.



(a) **(b)**

Figure 2. Operation of the *nanovisc* tool. (a) Nanocaliper setup capturing temporal variation of reflected illumination as RLT reduces [4]; (b) User interface of *nanovisc* tool showing how peaks and troughs of red and blue reflected intensities are identified and mapped unambiguously to particular RLT values that are registered in time. The time course of RLT is then interpreted with a squeeze-film flow model to evaluate effective viscosity and assess whether resist behavior deviates significantly from Newtonian.