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Detecting Temperature-Induced Strain Changes using *In Situ* Transmission Kikuchi Diffraction

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While transmission Kikuchi Diffraction (TKD) is traditionally used to map crystal orientations, TKD's sensitivity to changes in crystal structure allows it to map thermally induced strain as well [1, 2]. However, indirect measures such as misorientation are not sensitive enough to detect changes at the parts-per-thousand level. Here, we introduce a new TKD-based method for mapping strain that exploits its ability to determine lattice parameters with picometer precision. In a scanning electron microscope (SEM) we raster the electron beam over an electrically-contacted graphite flake, recording a TKD pattern at every point. TKD maps of the graphite under bias (hot) show changed lattice parameters relative to the no-bias (cold) condition, which we attribute to changes in the temperature of the sample.

A thin, exfoliated graphite flake is transferred to three gold electrodes on an electron-transparent silicon-nitride window (Fig. 1a). We collect TKD patterns with an Oxford Symmetry EBSD camera on a Helios G4 PFIB UXe SEM. Here a full TKD data set consists of 128×119 patterns (the region shown in Fig. 1b). We alternately heat, cool, and heat the graphite flake during the course of a single scan, biasing it with 70 mW for 150 s, 0 mW for 100 s, and then 70 mW again for 50 s until the scan is finished (Fig. 2a). Oxford Instruments' AZtecHKL and AZtecICE software extract the mean angular deviation (MAD) from the TKD patterns. Further processing of the MAD values in Mathematica [3] gives the ratio of graphite's out-of-plane lattice parameter *c* to its in-plane lattice parameter *a*. Mapping the *c/a* ratio as a function of the beam position (Fig. 2b) reveals a signal that is correlated with the changes in bias.

Materials with noncubic crystal structure (e.g. graphite) typically have different coefficients of thermal expansion (CTE) along different axes [4]. Thus, varying the temperature varies the ratio of the two lattice parameters. We therefore attribute the changes of the *c/a* ratio to the temperature shifts from hot to cold and back to hot. Averaging each row of the graphite in Fig. 2b, we observe changes in the *c/a* ratio (0.37%) correlated with the application of the bias (Fig. 2c). The increase observed at high temperature relative to room temperature agrees qualitatively with our expectation based on graphite's CTE: the out-of-plane (*c*-axis) CTE is greater than the in-plane (*a*-axis) CTE [4]. By assuming the graphite flake is at room temperature under 0 mW bias and integrating the nonlinear CTE as a function of temperature [5, 6], the temperature change is quantified to be 130 ± 14 K.

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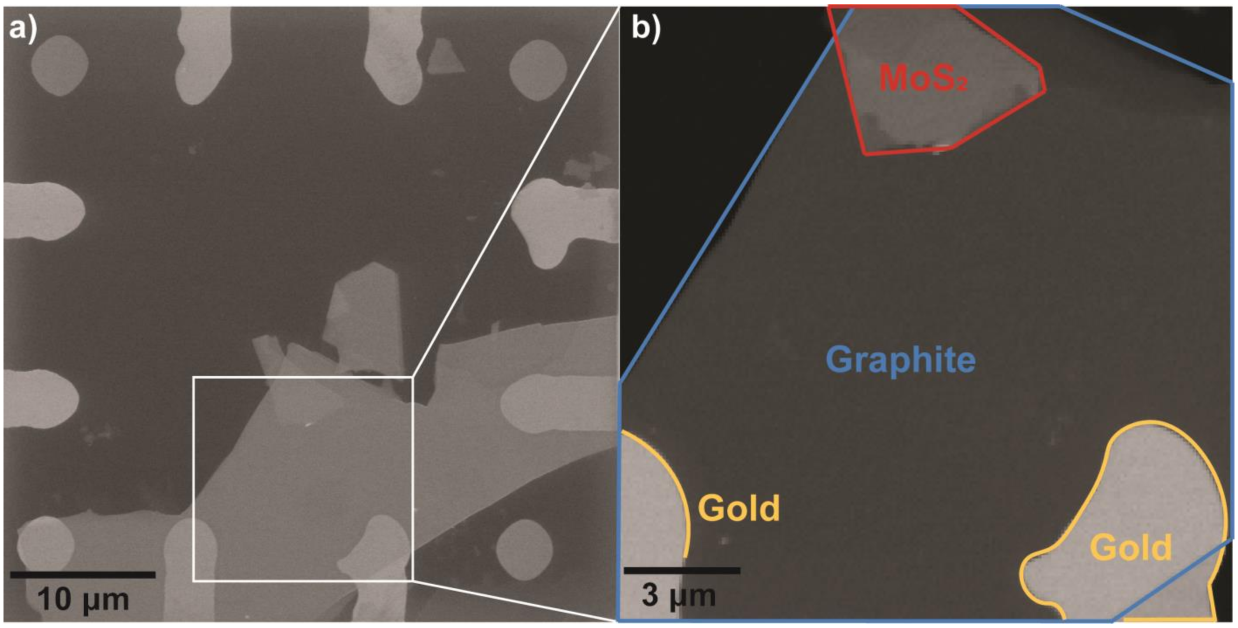

Figure 1. a) A low-mag SEM image of a graphite flake making electrical contact to three gold electrodes on a Si_3N_4, electron transparent window. b) The region where the TKD patterns are collected. The small second flake seen in (b) is MoS_2 and this area is masked during the analysis.

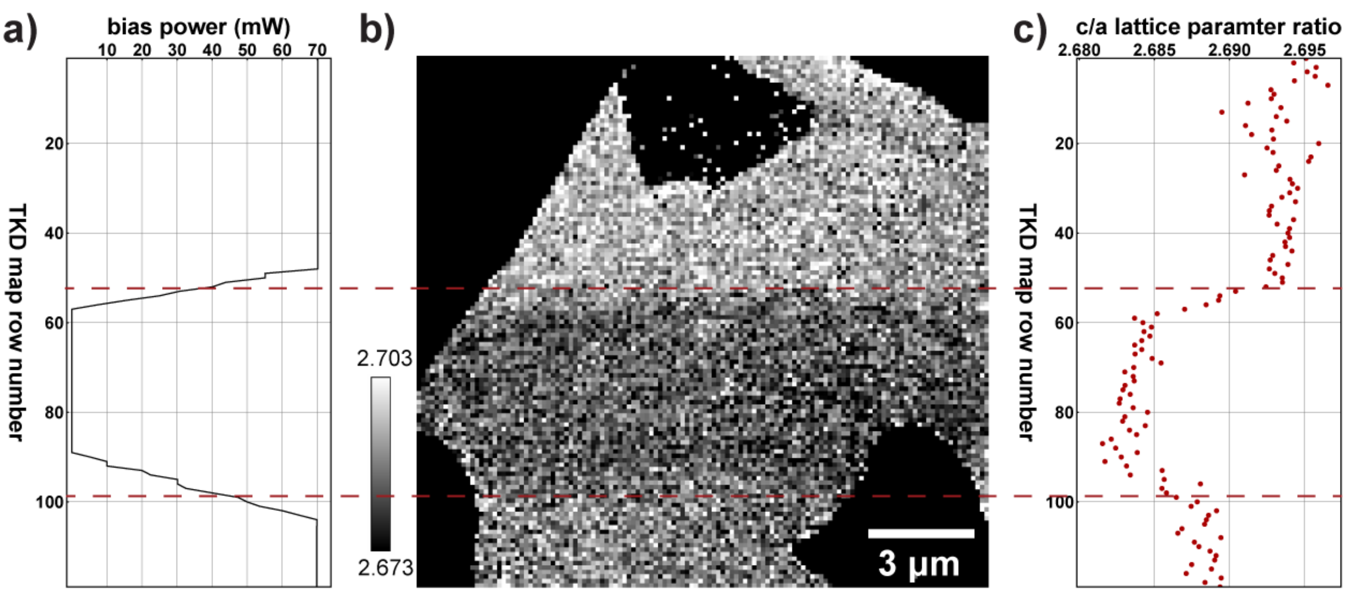

Figure 2. a) Power applied to the graphite flake at each row in the imaged region (Fig. 1b). b) A map of the *c/a* lattice parameter ratio of the graphite. c) The row average of the *c/a* ratio.

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