doi:10.1017/S1431927622003713
Microsc. Microanal. 28 (Suppl 1), 2022
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Capturing Reaction Kinetics of Atomically Thin Device Materials by High-throughput in-operando SEM

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We systematically explore nucleation, crystal growth and aging and failure mechanisms for thin films at the atomic monolayer limit. Particularly for the large family of layered 2D materials, many exciting properties and device concepts have been reported, yet the understanding of fundamental mechanisms that can underpin scalable process technology is lagging far behind. We adapt in-operando metrology to explore in detail such mechanisms for atomically thin films, including graphene, hBN and WS_2 [1,2,3]. Here, we focus on the use of SEM motivated by the ability to bridge between relevant size scales from nm to mm and compatibility with a large range of substrates including non-planar device architectures. Combining a heater stage with localized gas injection allows us to access a wide range of reactive atmospheres with the in-lens SEM detector. We adapt hyperdimensional reaction assays to collect big data sets that open pathways to full statistical analysis approaches and much refined models. In this talk we focus on localized thermal oxidation of monolayer WS_2 (Figure 1) for temperatures between 450-680°C and mbar pressure range. The corrosion reaction manifests itself via the nucleation and expansion of etch pits and we simultaneously track the detailed kinetics across ~10^3 pits per individual reaction sample. We develop image processing algorithms to recognize and segment across ~10^3 images and extract individual, time-dependent reaction rates and related distributions at the nanoscale. We can reveal and start to understand many interconnected dependencies including crystallographic effects and transitions between different reaction regimes. [4]

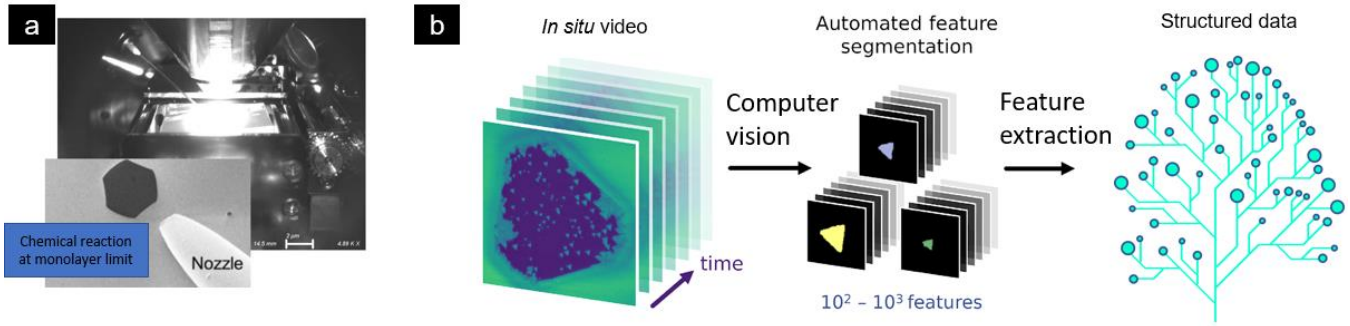

Figure 1. In operando characterization of corrosion kinetics in monolayer WS_2. (a) SEM and sample set-up. (b) Automated image processing, data tree formation and modelling of reaction kinetics.

References:

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[4] We thank Dr L. Han from Carl Zeiss Ltd. for support in adaptation of the instrumentation.

https://doi.org/10.1017/S1431927622003713 Published online by Cambridge University Press