

Development of simple image processing for in-situ TEM toward live processing

Dr. Junbeom Park¹, Hongyu Sun², Janghyun Jo³, Eva Jodat¹, André Karl¹, Shibabrata Basak¹, Rüdiger-A. Eichel^{1,4}

¹Institute of Energy and Climate Research - Fundamental Electrochemistry (IEK-9), Forschungszentrum Jülich GmbH, Jülich, Germany, ²DENSsolutions B.V., Delft, The Netherlands, ³Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (ER-C), Forschungszentrum Jülich GmbH, Jülich, Germany, ⁴Institute of Physical Chemistry, RWTH Aachen University, Aachen, Germany

Background incl. aims

In-situ transmission electron microscopy (in-situ TEM) has become an essential tool for studying dynamic phenomena at the nanoscale [1]. Observing nanoscale phenomena with in-situ TEM allows us to understand the mechanism in more detail, which leads to improving material properties or increasing the process efficiency. However, most of the information is available only after a long time of analysis, which ends up as a passive approach. To actively investigate the phenomena, more information should be extracted during the in-situ experiment to manage the stimuli in real time.

Live processing during the in-situ TEM experiment can allow to overcome this limitation by actively controlling the stimulus conditions according to the observed features, thus allowing dynamic adjustments during the experiments. Achieving live processing requires two critical components: 1) establishing real-time communication channels between the TEM, the in-situ stimuli, and the processing server, and 2) developing fast and efficient image processing algorithms capable of handling the high throughput of data generated during in-situ TEM experiments. Until now, artificial intelligence (AI) has not been able to help with live processing due to the large amount of computing power and time required. While the establishment of real-time communication channels requires cooperation with TEM manufacturers, this paper focused on the development of image processing algorithms suitable for live processing.

In this work, we present the development of simple but effective image processing techniques suitable for live processing in in-situ TEM. Three cases of segmentation from electrochemical in-situ TEM experiments were shown as: (Case 1) plating and stripping of Zinc (Zn), (Case 2) dendritic growth of Zn, and (Case 3) particle-like plating and stripping of Copper (Cu) [2, 3]. Through these case studies, we demonstrate the ability of simple image processing to understand electrochemical phenomena and its potential for applications.

Methods

a) Performing in-situ electrochemical TEM experiments

In-situ Liquid TEM holder (Stream, DENSsolution) assembled with 3-electrode Micro-Electro-Mechanical System (MEMS) chips was used for experiments. In case 1, Pt electrode MEMS chip and 0.1 M ZnSO₄ solution (flowing) were used and cyclic voltammetry (CV, -1.5 V ~ +0.8 V for 0.01 V/s scan rate) was applied. In case 2, Pt electrode MEMS chip and 0.1 M ZnSO₄ solution (non-flowing) were used and chronopotentiometry (CP, 5 μ A for 10 seconds) was applied. In case 3, TiN_x electrode MEMS chip and 0.02M CuSO₄/0.01M KH₂PO₄ solution (flowing) were used and chronoamperometry (CA) with 4 cycles (one cycle: +1.5 V for 15 seconds and -1.5 V for 15 seconds) was applied. Case 1 and 2 were monitored at STEM mode and case 3 was monitored at TEM mode.

b) Developing simple image processing methods dedicated to in-situ TEM

All the image processing methods are developed as python codes. To reduce the noise level, a gaussian filter was applied as pre-processing. Key strategies are 1) subtraction of the reference image (pure electrode) from target image (during experiment) to extract the information changes and 2) thresholding the subtracted image to binary-segmenting as feature and background. Especially case 2, the subtraction was performed between images with 1 second difference, in order to emphasize the fast growth of dendrite within 1 second. After the segmentations, the deposition information such as deposited area was numerically extracted and plotted.

Results

In case 1 (Figure 1a), Zn plating and stripping phenomena were recorded with CV stimuli. On the recorded STEM images, the plated Zn is well visible, but the brightness of the plated Zn looks similar to the rectangular Pt electrode on the right side. After the simple image processing as described in methods, the plated Zn is clearly visible without Pt electrode and the area of plated Zn can be measured easily. After measuring the area throughout the whole CV process, the plated Zn area can be synchronized with the CV stimulus data, which leads to a quantitative understanding of the phenomena.

In case 2 (Figure 1b), Zn dendritic growth was recorded with CP stimuli. From the video, a dendritic growth took only a few seconds, so the information related to the growth rate is more important than the amount of growth. By subtracting the 1-second difference between frames, the grown dendrite is visualized in 1 second, which can directly show not only how fast the dendritic growth is, but also which direction is preferred. In this case, Zn dendrite growth occurred to the left where the counter electrode is located.

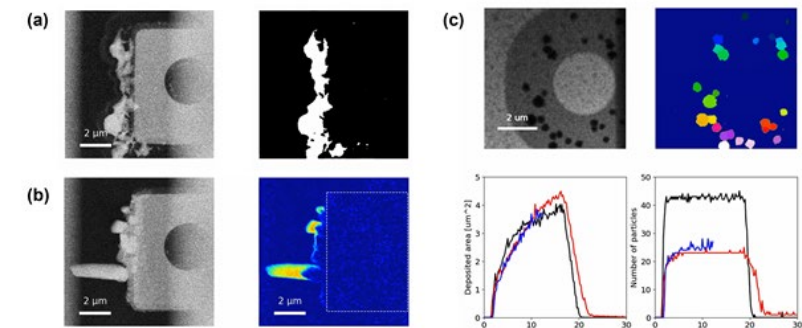
In case 3 (Figure 1c), Cu particle-like plating and stripping was recorded with cyclic CA stimuli. On the recorded TEM images, plated Cu particles are well visible on the top of the electrode (round shape with hole), but small dots are

also visible at the outside of electrode. Large Cu particles on the top of the electrode are formed based on electrochemical deposition and small Cu particles outside of electrode are electron beam induced deposition. To focus on electrochemical deposition, the only electrode area was selected to further particle segmentation. After the image processing, each individual Cu particle was segmented and could be tracked during the plating and stripping process. The area and number of particles were plotted with a time scale to study the plating and stripping phenomena.

Conclusions
In-situ TEM has been enabled to observe countless phenomena in nanoscale, but quantitative information was very limited due to huge amount of dataset which cannot be handled by human. This paper showed that the bunch of quantitative information can be extracted from recorded in-situ TEM video by simple image processing. We believe that simple image processing will allow to actively investigate the phenomena at nanoscale by correlating the observed feature and applied stimuli in real time.

Figure 1. Examples of image processing from in-situ TEM dataset. (a) STEM image and segmented image of metal deposit. (b) STEM image and reconstructed image of metal dendrite growth within 1 second. (c) STEM image, segmented image of particle shape deposits and graphs about deposit area and number of particles vs. time. [2, 3]

Graphic:



Keywords:

in-situ TEM, Live, Image processing

Reference:

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