

Response to reviewer comments

Changes to be made by the Author(s) regarding the video:

1. Please increase the homogeneity between the video and the written manuscript. Ideally, all figures in the video would appear in the written manuscript and vice versa. The video and the written manuscript should be reflections of each other.

Response: [We believe that the video reflects the paper. We have a real video that shows the action and highlights the procedure in real time in more complete manner than shown in some figures.](#)

2. Furthermore, please revise the narration to be more homogenous with the written manuscript. Ideally, the narration is a word for word reading of the written protocol.

Response: [We originally had that done, but since then the Editor has requested additional changes to the manuscript; the movie was prepared by the ORNL production team, and our video expert has made numerous changes already. We cannot keep changing minor things, and our video expert has officially declined to address any further complicated edits. Our opinion is the following: we believe that any researchers who would be interested in and benefitted by this paper and the accompanying video are already expert microscopists, and only need help with the details \(protocol steps\) of the process, not e.g., how to load a sample into the electron microscope. Our protocol description is not \(and cannot be\) intended to train a novice microscopist to be able to complete a good in situ gas reaction experiment.](#)

3. Please ensure that the title of the submission is the same in the text and the manuscript.

Response: [We updated the title slide.](#)

4. Please ensure subheadings are the same in the text and the video.

Response: [We believe they match.](#)

5. Please ensure that there is a chapter title card: Representative results and it describes all the figures presented in the representative result section of the manuscript. The other figures can be shown wherever these are referenced in the text.

Response: [We added the "Representative results" card and the results are shown through the movie in real action.](#)

6. Please change the Summary card to Conclusion instead.

Response: [We kindly disagree with the comment, since this is a protocol, we believe that the title "Summary" is more suitable than "Conclusions".](#)

7. Please remove all the commercial terms from the video. e.g., protochips, atmosphere software, etc. Wherever possible please hide the commercial terms when showing the instrument.

Response: [Unfortunately, our ORNL production team will not be able to address this part.](#)

8. Please place the title card at the end of the video as well.

Response: [We placed the title card at the end of the video.](#)

9. • A lot are out of focus, at 3:44, 2:22, 2:48, 4:10, 5:32, 6:03, 8:33, 8:38, 9:50, 10:16

Response: [We kindly disagree with the Editor. This was done on purpose.](#)

10. • At 3:10 through 3:15 the video jumps between four different shots and figures too quickly for the viewer to grasp what is important

Response: [We believe it is just fine.](#)

11. • Voice over audio volume lowered significantly at 2:56

Response: We requested our production team to update it.
12. • When the speaker isn't talking, there is a clicking sound in the background. Please cut out the parts where the speaker isn't talking

Response: We believe the parts are needed for the coherency of the movie. Usually, it takes longer to show it.

Replay to Editor's comments:

1. In the video this is M-Bond, please change to cyanoacrylate glue as we cannot have commercial terms.

We requested that change to our ORNL team production.

2. In the video this is Preparation of Atmosphere holder. Please make it same in the video and the text. This part please ensure that the text and the narration is the same for this section and in the same order. Currently this does not match.

The small mismatch in wording is due to additional changes to the text of the manuscript that were requested by Editor, after the video was made. We cannot keep changing and updating the video → it is too time-consuming, and our ORNL production team has declined to keep doing it. We believe that the video is just fine as it is.

I did re-record the audio but our ORNL production team said they will not be able to work on this.

If we receive the raw file of the movie from ORNL production team, I can provide audio files to JoVE team to update the audio.

3. In the video please do not make a separate subheading “start the experiment” to make it homogenous with the text.

The “start the experiment card” was removed.

4. At 12:33 please insert a chapter title card in the video stating Representative results

We requested to add “Representative results” title card

5. Please ensure subsections headings are the same in the text and the video and follow the same order to make the manuscript text homogenous to the video.

We updated the order; I moved FIB section after Mask section.

6. As we are a methods journal, please ensure that the Discussion explicitly cover the following in detail in 3-6 paragraphs with citations:

- a) Critical steps within the protocol
- b) Any modifications and troubleshooting of the technique
- c) Any limitations of the technique
- d) The significance with respect to existing methods
- e) Any future applications of the technique

This was addressed as shown below:

“In the present work, an approach to perform *in situ* STEM reactions with and without water vapor is demonstrated. The critical step within the protocol is E-chip preparation and maintaining its integrity during the loading procedure. The limitation of the technique is (a) the specimen size and its geometry to fit the nominal 5- μm gap between paired (MEMS)-based silicon microchip devices as well as (b) a total pressure used in the experiments with water vapor since the highest total pressure depends on the quantity of water vapor. The significance of this method with respect to existing methods is that we can perform *operando* experiments, i.e., we analyze the specimens under real conditions, enabled by an RGA system that confirms/monitors experimental conditions. Additionally, there are opportunities for future applications of the technique to diverse materials systems that may require different methods and procedures for sample deposition on E-chip heaters.”