A Combined *In-situ* and Electron Tomography Holder for (S)TEM.

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Introduction

Recent advances in *in-situ* and tomographic methods using (scanning) transmission electron microscopy ((S)TEM) provide the unique possibility of obtaining 3-dimensional (3D) information and *in-situ* studies of gas-solid chemical reactions on the nanometer scale [1, 2]. In combination with the well-known analytical techniques, electron energy-loss spectroscopy (EELS) and energy dispersive X-ray spectroscopy (EDXS) we can obtain 3D, dynamic, analytical, and structural information down to the atomic level.

Instrumentation

The JEOL JEM-2500SE STEM/TEM at UC Davis (CHMS) is equipped with a Schottky field-emission source operated at 200 kV, a post column Gatan imaging filter (863 GIF Tridiem) for EELS and a Thermo System Six for EDXS.

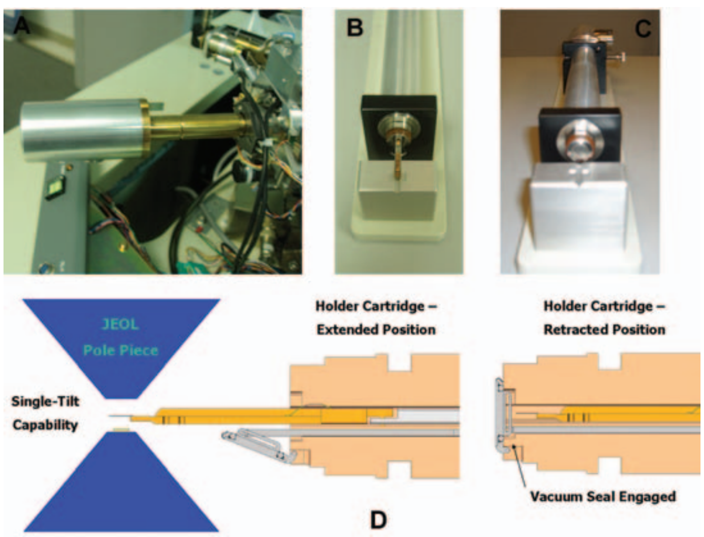

Figure 1: Vacuum Transfer Tomography Holder (Phase I); Vacuum transfer between reaction chamber and the microscope. (A) Inside the (S)TEM, (B) in extended, (C) in retracted position, and (D) the schematic drawing.

Discussion and conclusion

The first phase of the "*in-situ*"/tomography holder is a vacuum transfer holder with an external multi-gas reaction chamber (cf. Fig. 1 + 2). The specimen can be heated over a laser heated specimen cradle (< 500°C; cf. Fig. 3). The cradle design improves the temperature distribution within the specimen and the dual ceramic shielding maximizes the specimen temperature and minimizes the heat transfer to the microscope.

The second project phase will have the reaction chamber fully integrated into the (S)TEM for true *in-situ* investigations (cf. Fig. 4). This approach includes placing a reaction chamber within the TEM pole piece gap. The reaction chamber implies a mechanism for gas handling (Fig. 4B). The CO_2 laser enters the column through the access port opposite the goniometer (Fig. 4C).

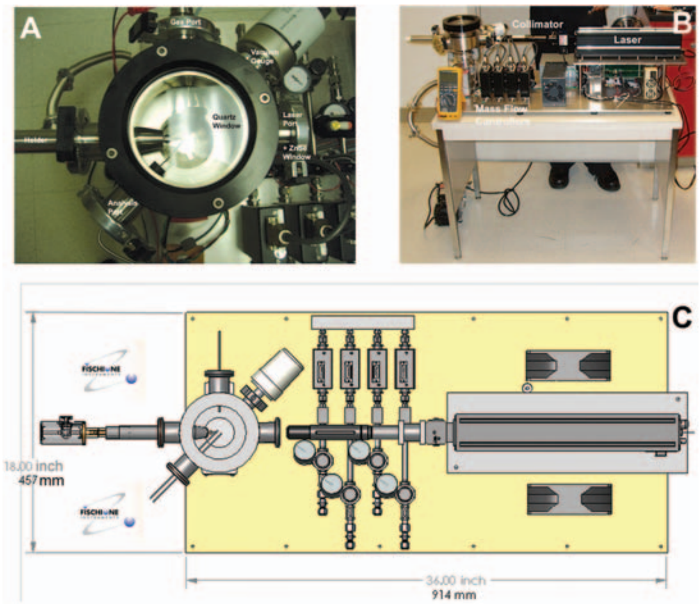

Figure 2: (A) External reaction chamber, (B) image and, (C) schematic drawing of the reaction assembly (Phase I).

With the Fischione instruments, Model 2030, Phase II vacuum transfer *in-situ*/electron tomography holder we can then take the full advantage of doing combined tomography and *in-situ* studies.

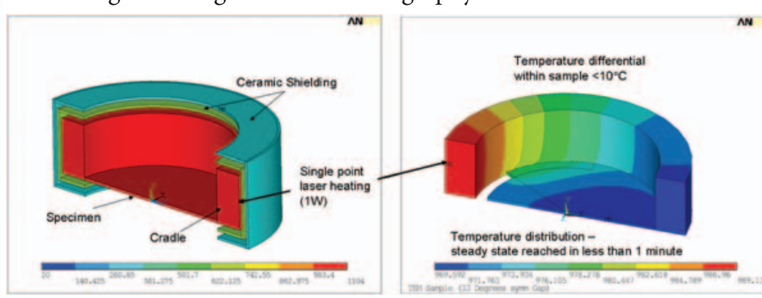

Figure 3: (A) Schematic drawing of the specimen cradle and (B) the temperature distribution.

Specifications

In-Situ/Tomography Holder (Fischione instruments, Model 2030), Phase I.

Vacuum Transfer Tomography Holder

-Compatibility with the JEOL 2500 goniometer and external reaction chamber

-Vacuum transfer between the reaction chamber and microscope

-Room-temperature, single-tilt tomography holder

-Specimen size: 3mm diameter, wide range of thicknesses

-Resolution: 0.34nm

-Drift: <1.5nm/min

-Primary tilt: up to +/- 70°

-Field of view: up to 1.5mm at 70°

Multi-gas Reaction Chamber

-4 separate individually controlled mass flow controllers

-Vacuum system capable of <5e-7 torr base vacuum level

-LabVIEW executable vacuum, gas, and heating controls

Laser Heated Specimen Cradle

-Sample heating up to 500°C

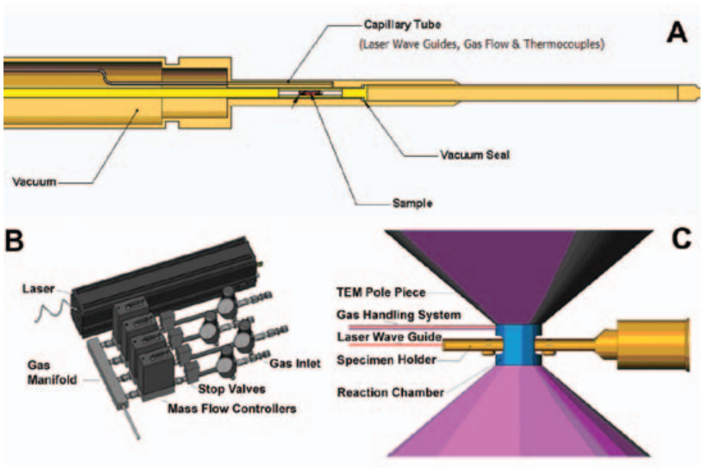

Figure 4: In-Situ/Tomography Holder, Phase II. (A) Specimen in retracted position, (B) mass flow controllers and stop valves and, (C) the integration with the (S)TEM.

In-Situ/Tomography Holder (Fischione instruments, Model 2030), Phase II.

A fully integrated Vacuum Transfer, *In-situ* Reaction Tomography Holder with all capabilities from Phase I.

Acknowledgements

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References

[1] I. Arslan, T.J.V. Yates, N.D. Browning and P.A Midgley, Science 309 (2005) 2195-2198.
[2] P.L. Gai, Microsc. Microanal. 8 (2002) 21-28.