J McGuire Technologies

Applications: Equipment for Characterizing and Processing Materials for Energy Storage Devices

Metrology for Ex-Situ & In-Situ - Plasma Diagnostics / Thin Film Characterization / Nanoparticle Deposition / AFM / MOCVD / Plasma Cleans for Electron Instruments

k-Space Associates: Thin-Film Characterization & Imaging Technologies
http://www.k-space.com
ibss Group, Inc: Downstream Asher rids hydrocarbon contamination
http://www.ibssgroup.com
Nanosurf AFM: AFM - NSOM - RAMAN - TERS
http://www.nanosurf.com
Valence Process Equipment: MOCVD for LEDs and Advanced Processing
http://www.valenceprocess.com
Impedans, Ltd: Plasma Diagnostic Solutions for R&D and Production
http://www.impedans.com

- > 20 Years Capital Equipment and Component Sales to R&D, Government Labs, Semiconductor Fabs, Compound Semi Companies

- Independent sales representative / jp.mcg@sbcglobal.net / Central Valley, CA 95219 / PH: 209.478.1672

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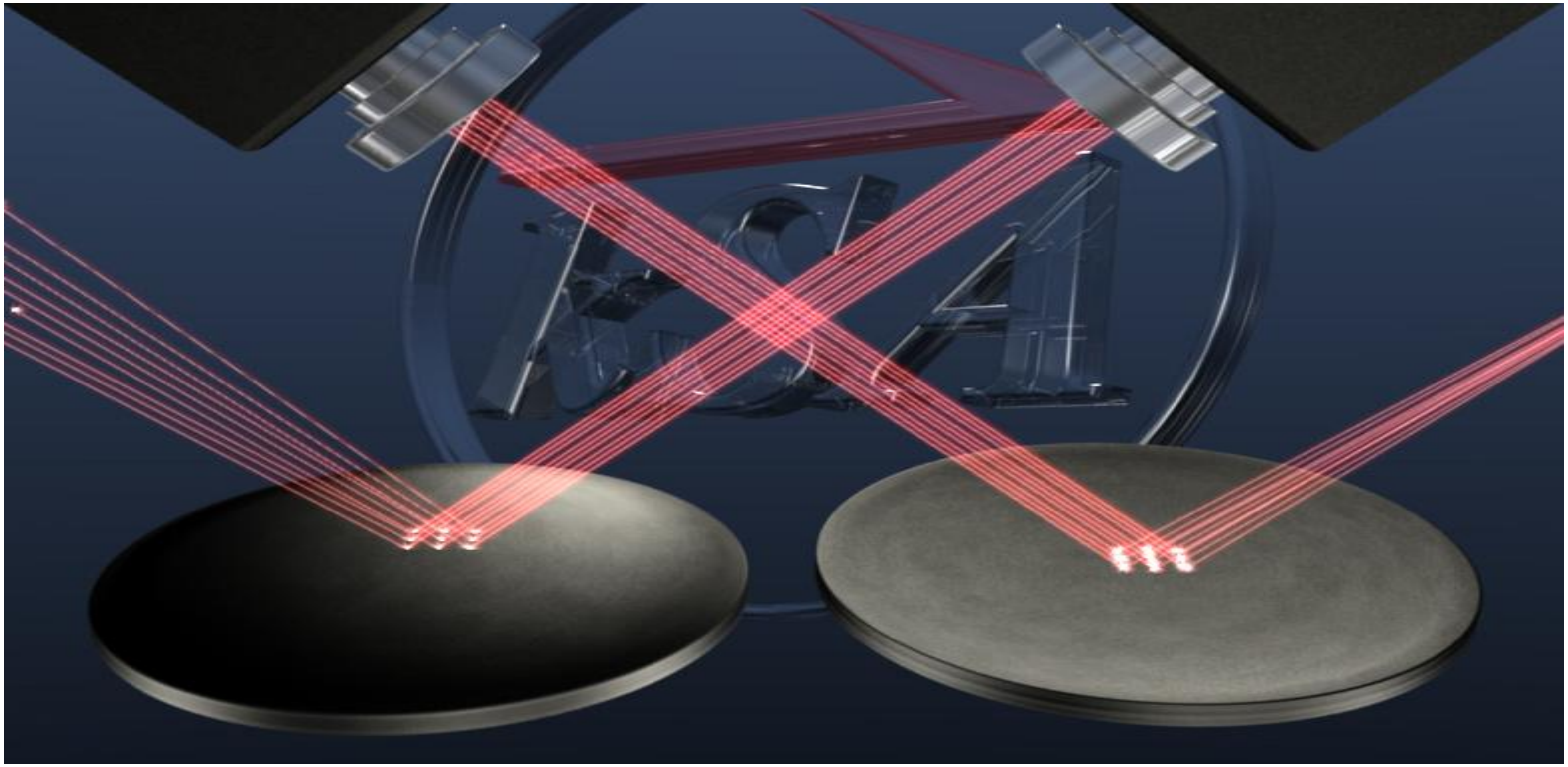

The (MOS) multi-beam optical sensor technique employs an array of parallel laser beams, and measures the relative change in the spacing between them (measures curvature change in 2 orthogonal directions) / application for high energy density lithium-ion batteries.

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MOS Method: Show quantitative connection between stress measurements and observed mechanical damage. For a silicon thin-film electrode, it is straightforward to take realtime measurement of stress-evolution, and those are used to calculate mechanical dissipation in the electrode during lithiation / delithiation, and compare to polarization losses.

- Experiment: A Si wafer (coated with the Cu and Si thin films) was assembled into a home-made electrochemical cell. Electrochemical measurements were conducted in an environmental chamber. Stress in the silicon thin-film was measured by monitoring the substrate curvature change during electrochemical lithiation and delithiation. Substrate curvature was monitored with a MOS wafer curvature system.

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Prior testing relied on cantilever (single beam) deflection method to observe stress evolution during lithiation / de-lithiation – does not account for refractive indices nor incident angles of all media through which the laser beam travels / vibration sensitive and reduces S/N ratio.

MOS testing records the vertical & horizontal displacements of the (multiple) laser spots as a function of time during all electrochemical experiments on Si (i.e. amorphous sputtered film) – acquisition rate = 1 Hz / no vibration sensitivity, very accurate.

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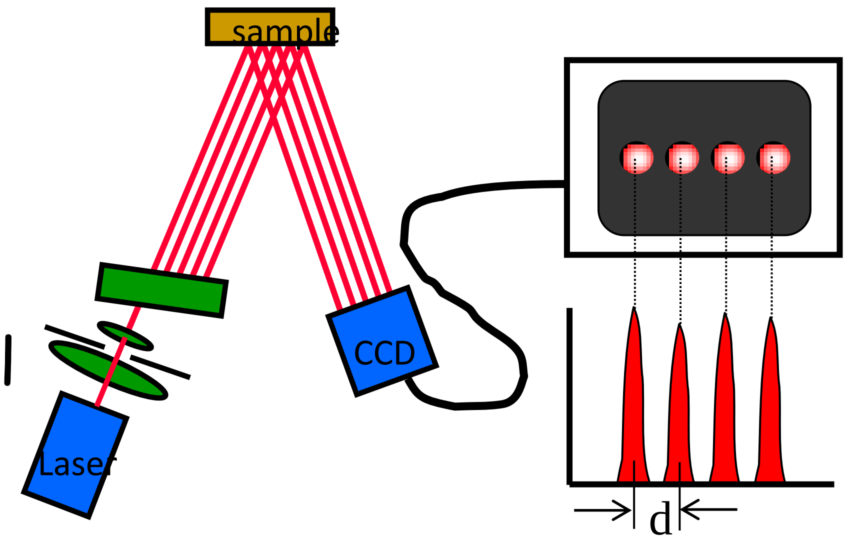
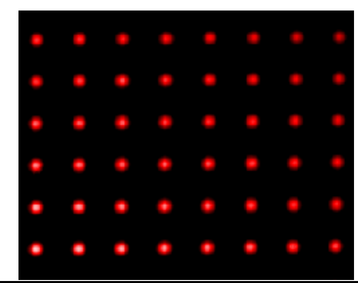
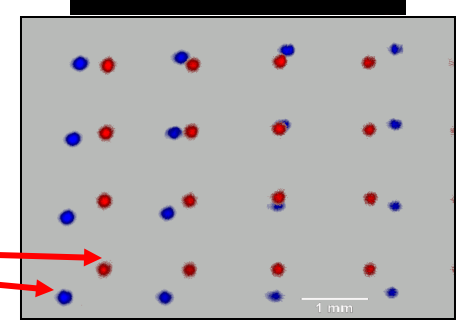
In-Situ Stress/Bow Monitoring

- Basic Principle:
  - Samples under stress 'deform' or change curvature
- Patented Multi-beam Optical Sensor (MOS)
  - How it works:
    - Parallel laser beams reflects from the sample
    - Beam position is measured with a CCD camera
    - Frame grabber digitizes the image, MOS software calculates the beam spacing
  - Change in beam spacings determine curvature, 2D stress profile







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MOS Method: Show quantitative connection between stress measurements and observed mechanical damage. For a silicon thin-film electrode, it is straightforward to take realtime measurement of stress-evolution, and those are used to calculate mechanical dissipation in the electrode during lithiation / delithiation, and compare to polarization losses.

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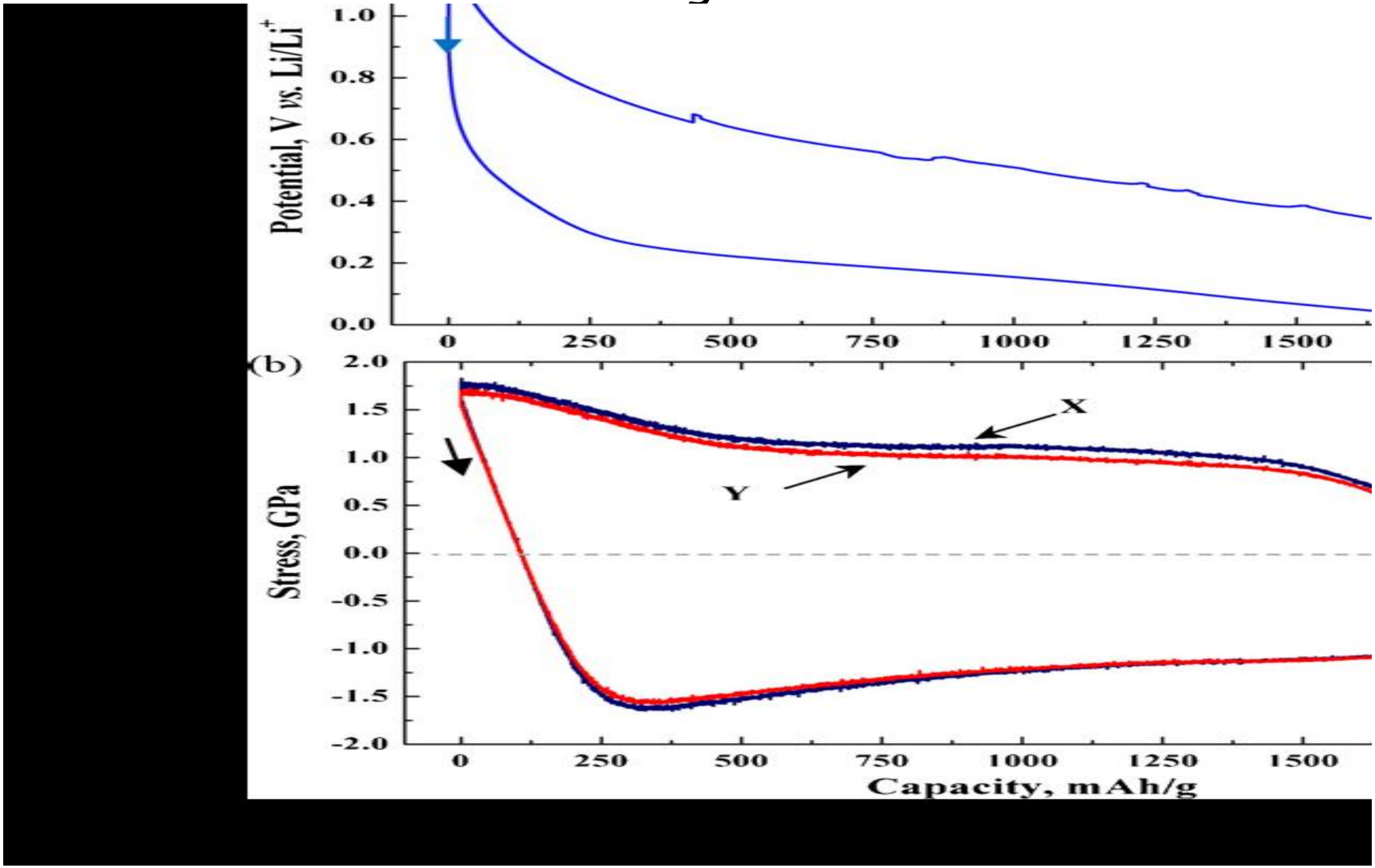
Testing Results



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Upon lithiation, the substrate prevents the in-plane expansion of the film, which results in compressive stress in the film, and it increases linearly with time (or capacity). If we assume that the strain induced by Li in Si is proportional to its concentration, then the linear increase in the compressive stress indicates elastic response.

Upon delithiation, the unloading is initially elastic; the stress reverses elastically until it becomes ca. 1 GPa in tension, where the film begins to flow in tension in order to accommodate the reduction in volume. The flow stress increases to about 1.75 GPa when the upper potential limit of 1.2V is reached. The stress response is similar in compression and tension, for any state of charge, and the flow stress in compression and tension are approximately the same. Thus, the film undergoes repeated compressive and tensile-plastic flow during successive lithiation–delithiation processes, respectively.

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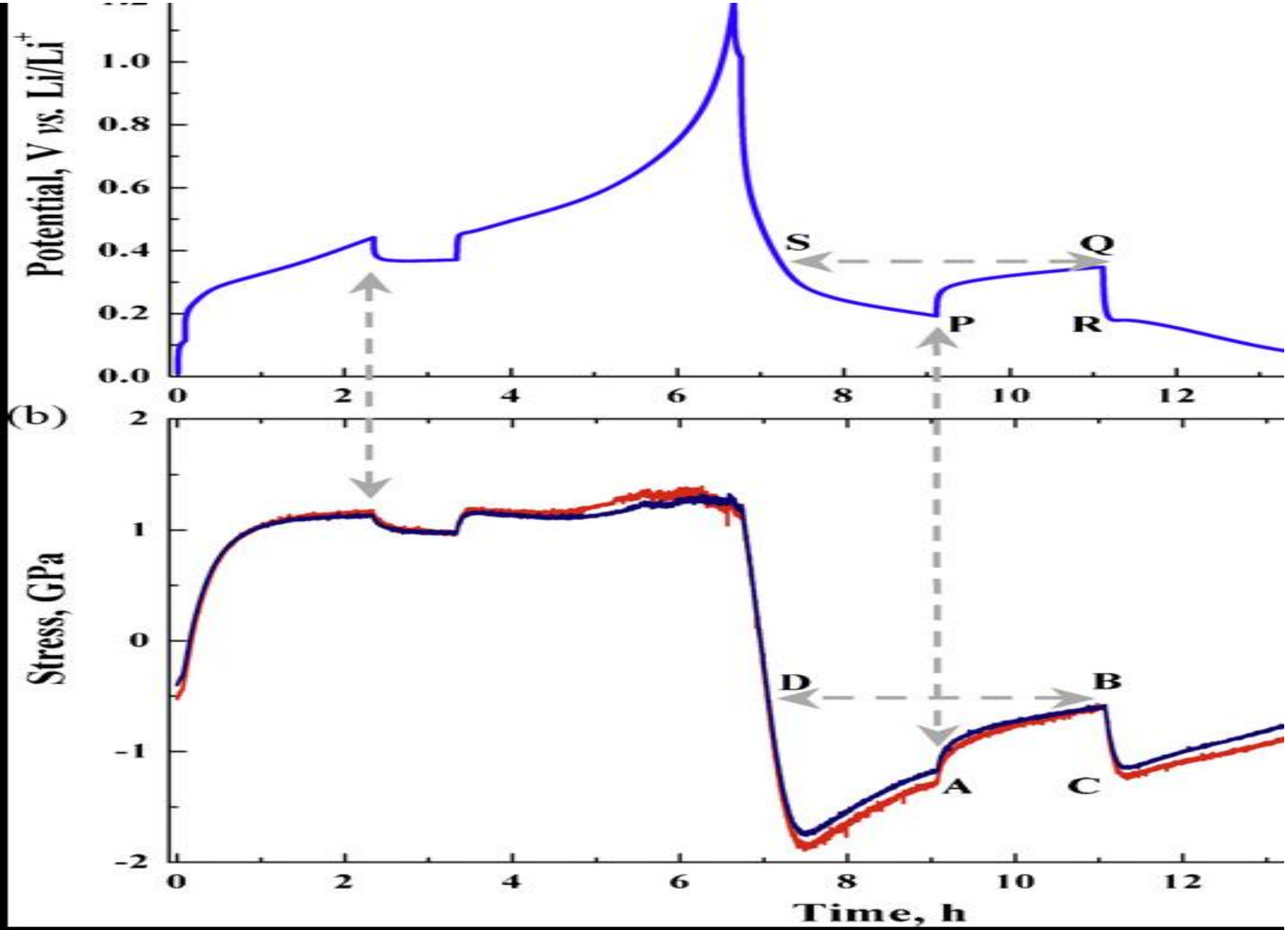

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MOS testing showing the connection between actual / repeatable stress measurements and observed mechanical damage for high energy density lithium-ion batteries due to Silicon's high charge capacity and low density. MOS ensures Si-based anodes / cathodes are fully tested.

- Testing has demonstrated the use of the multi-beam optical sensor technique to measure stress evolution in a silicon thin-film electrode during lithiation and delithiation; and during the open-circuit relaxation upon current interruption during lithiation or delithiation. Stress evolution upon electrochemical cycling reveals that the tensile-plastic flow, dissipating mechanical energy. The stress evolution data enables estimation of the mechanical dissipation, which was found to be comparable to the polarization losses elsewhere in the cell. This observation also suggests that stress contributes significantly to the chemical potential of lithiated silicon and hence the electrode potential.

ibss Corporation

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The **GV10x Downstream Asher** is intended to be used on any analytical high vacuum tool where hydrocarbon or carbon contamination prevents satisfactory quantitative analysis. The GV10x Downstream Asher oxidizes hydrocarbon [HC] contamination in these analytical chambers. The Downstream (DS) Process removes gas phase HC contamination quickly and ashes surface HC contamination more slowly.

- Analytical systems that examine samples of various material types with electron and ion beams at high magnification, CDSEM, SEMs, FIBs, TEMs, must be HC free to prevent hydrocarbon contamination from obscuring sample details and preventing other spurious effects. Other applications include XPS, Synchrotron mirror deposit cleaning, etc.

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- Energetic ions and electrons produced within the GV10x Source by an inductively coupled RF field are neutralized, also called recombination, approximately 4 to 6 inches from the Source exhaust. Beyond this point, in the downstream, there are no energetic species. Downstream plasma electrons and ions lose energy quickly and NO, NO2 and N2O molecules slowly. The long-lived oxygen radicals oxidize, also called ashing, hydrocarbons into CO, CO2 and H2O and these residual molecules are pumped free of the vacuum chamber. Unlike cold trapping, hydrocarbons are permanently removed from the chamber, not merely captured on a cold surface.
- Oxygen radicals are the active species produced from ambient air or oxygen gas. These O radicals are transported by the vacuum chamber pressure differential. Pumping system differential depends on two parameters pumping speed and system conductance. The volume between the Source and pumping port positions will be cleaned as the O radicals are transported downstream through the chamber. Elevated surface temperatures volatize bound HC contamination, so baking before or during the DS Process is suggested for heavily contaminated surfaces.

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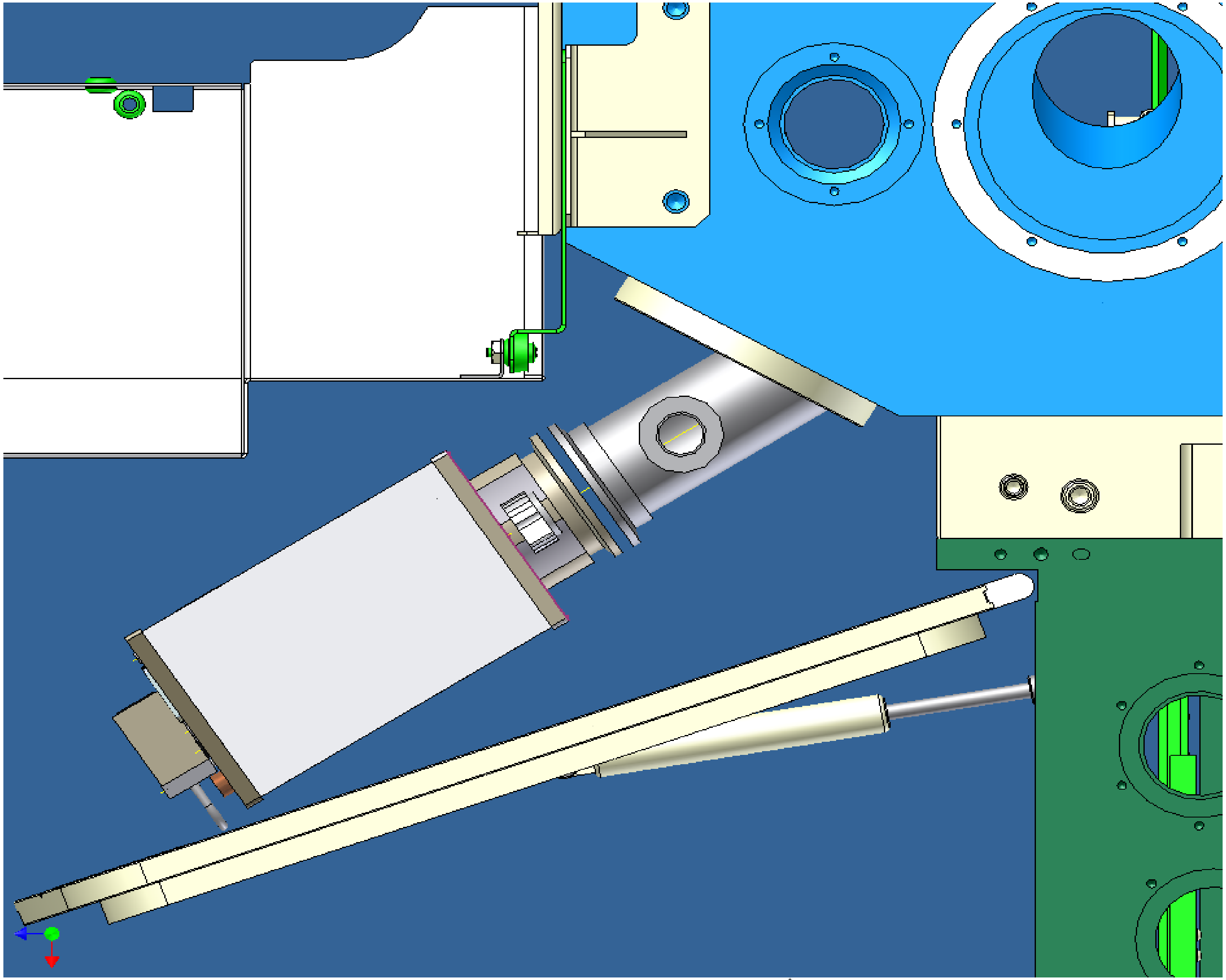

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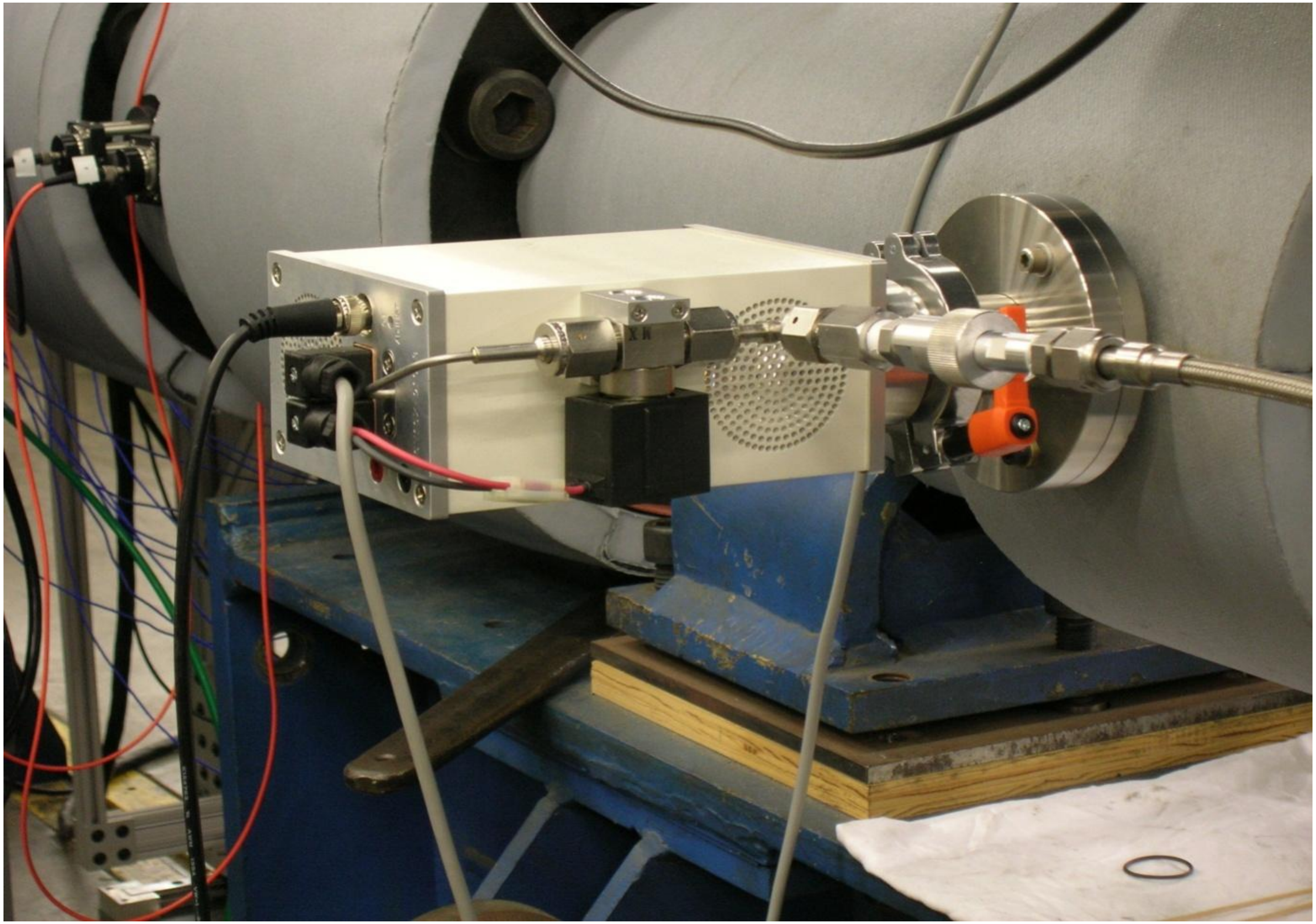

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On behalf of kSA Associates and ibss Corp. – Thank You for your Time – do advise if you would like an info-follow-up