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Differential Imaging Microscopy of Physically and Chemically Complex Surfaces Undergoing Atmospheric Corrosion

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Problem Statement

- Atmospheric corrosion, one of the primary failure mechanisms in aging microelectronic devices, is very difficult to assess without direct observation.
- Traditional electrochemical techniques can not be applied, and the ability of sensors or instrumented structures to capture the kinetics of the corrosion process on actual devices is limited.
- Critical need to develop techniques which that provide detailed kinetic information on atmospheric corrosion mechanisms, such that reliable predictive models can be assembled.

What is Differential Imaging?

- **Subtraction of an image taken at time *t* from an image taken at time *t+Δt***
  - Eliminate portions of the image which do not change
  - Resultant image nominally contains only those aspects which have changed
  - Can be applied to any imaging technique – not just optical

Background

- **Differential imaging technique for in-situ observation of atmospheric corrosion**
  - Similar to work of Huang, et. al (2006) for aqueous samples
  - System consists of imaging hardware/optics and an exposure chamber, along with data acquisition and image analysis software

- **Demonstration of system capabilities performed on noble metal plated copper specimens, simulating metallurgies commonly used in microelectronic connectors**

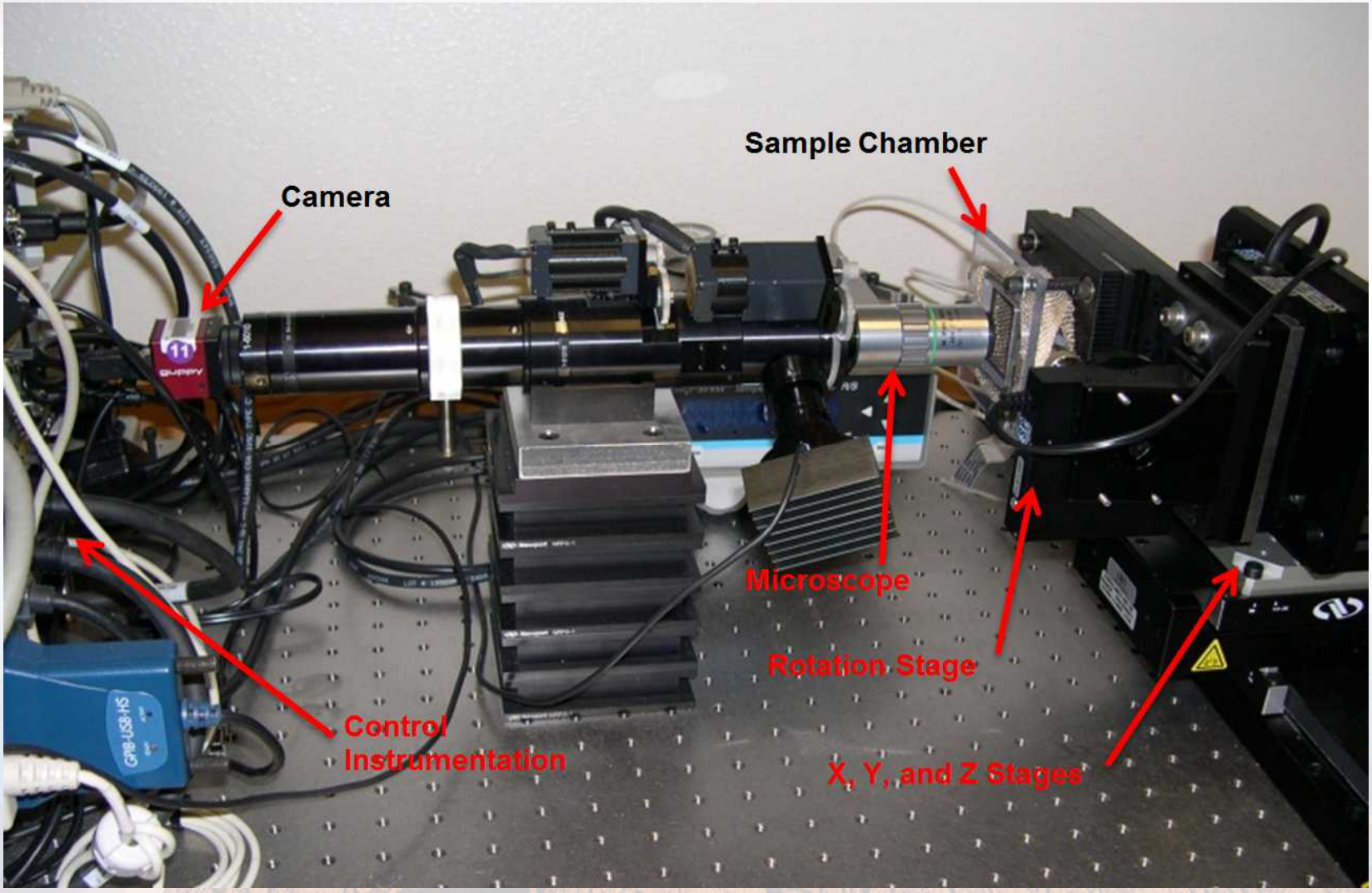
R.S. Huang, C.J. Lin, H.S. Isaacs, Electrochemical and Solid-State Letters, Vol. 9, no. 2 (2006) pp. B11-B14.

System Configuration

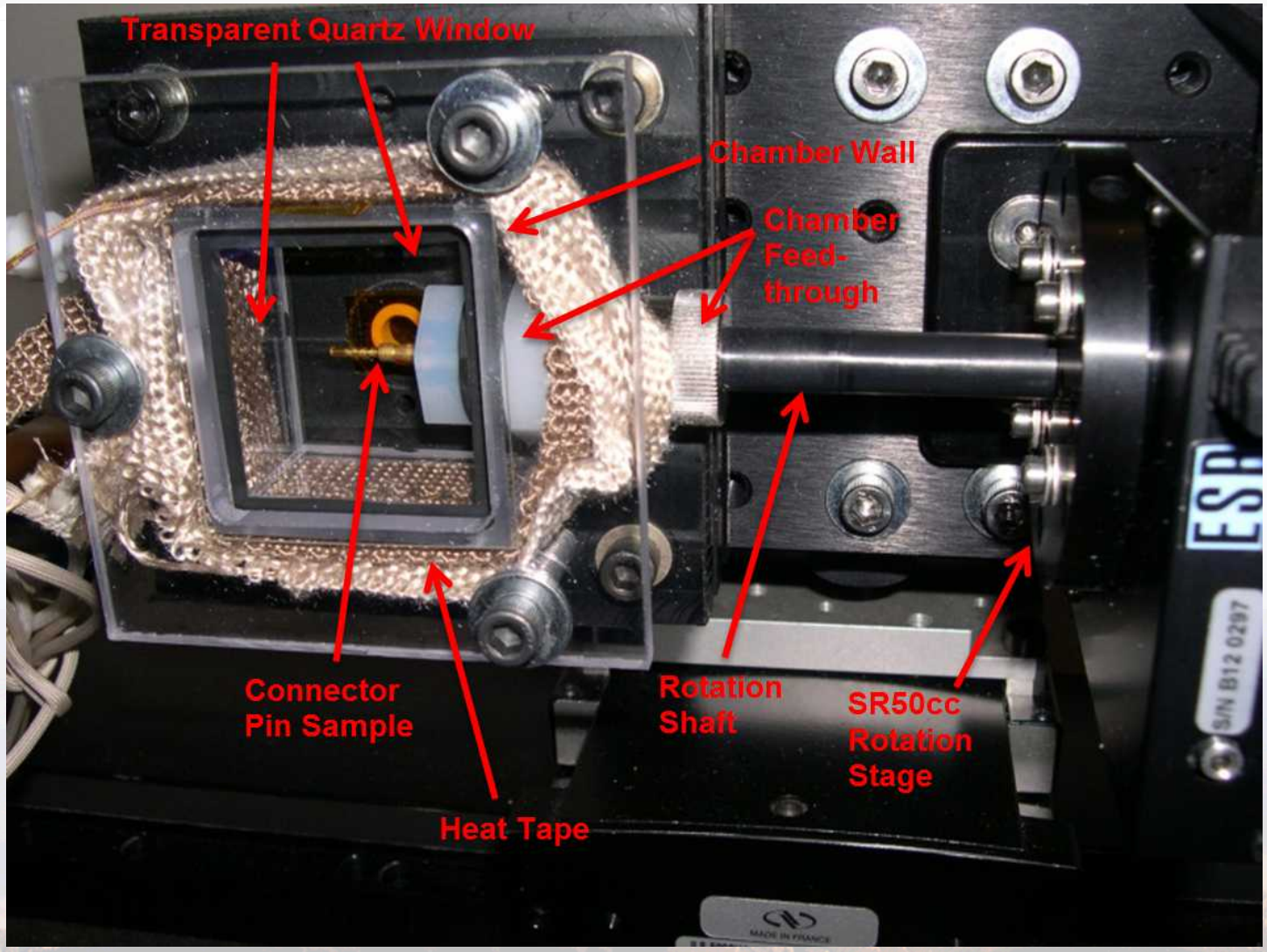
- **Goal: develop a system capable of monitoring a corroding surface in-situ**
  - Must be non-destructive
  - Need to be able to map a surface
  - Need to have flexibility in terms of the specimen geometry and environmental conditions

- **Basic system components**
  - Image acquisition system (resolution, repeatability, reliability)
  - Sample positioning/manipulation (repeatability, reliability)
  - Atmospheric control/exposure system
  - Control software to bring it all together and do image manipulation (subtraction, stitching, mapping)

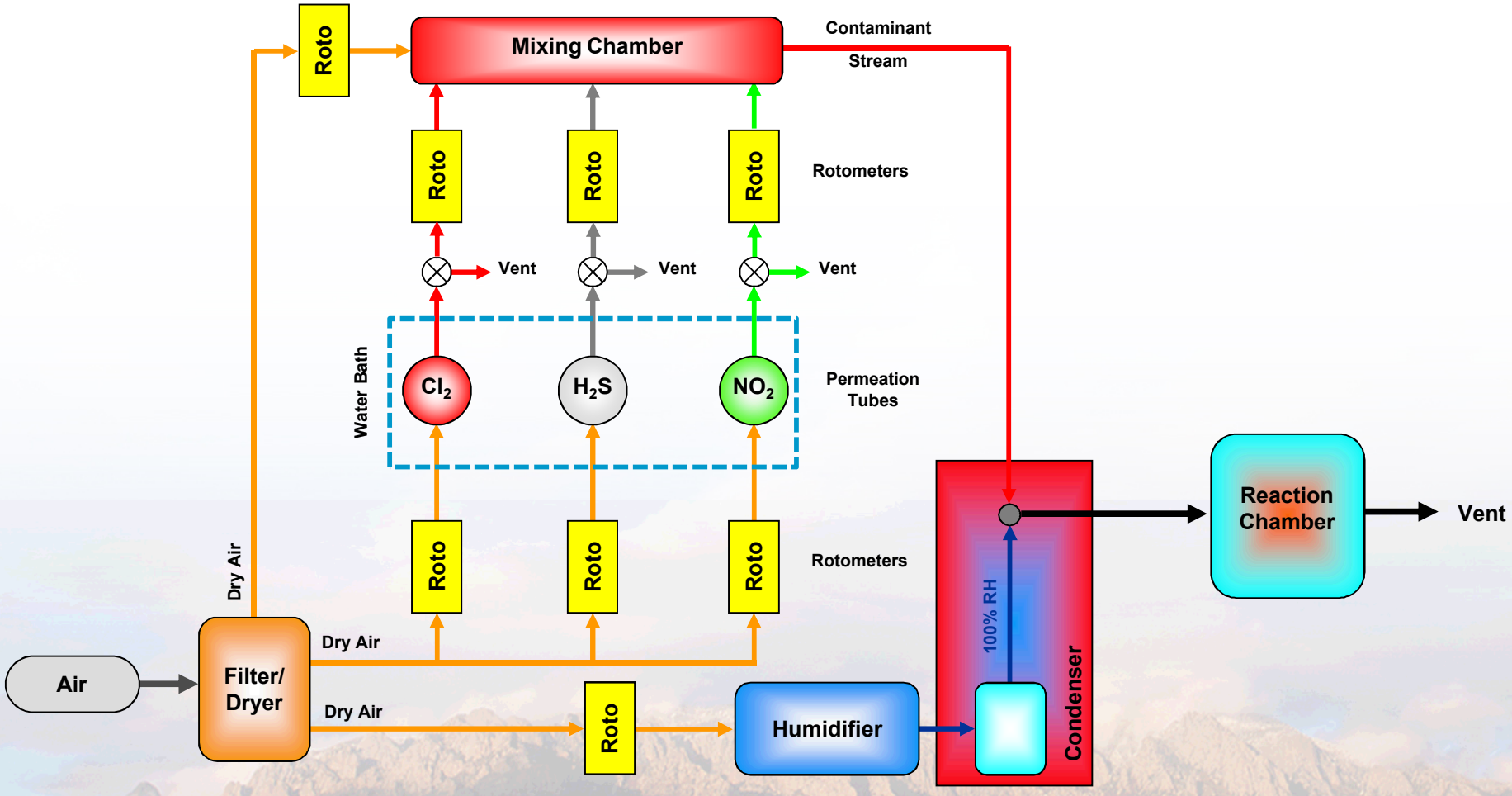
Overall Physical Configuration



The Exposure Chamber
(One Example)

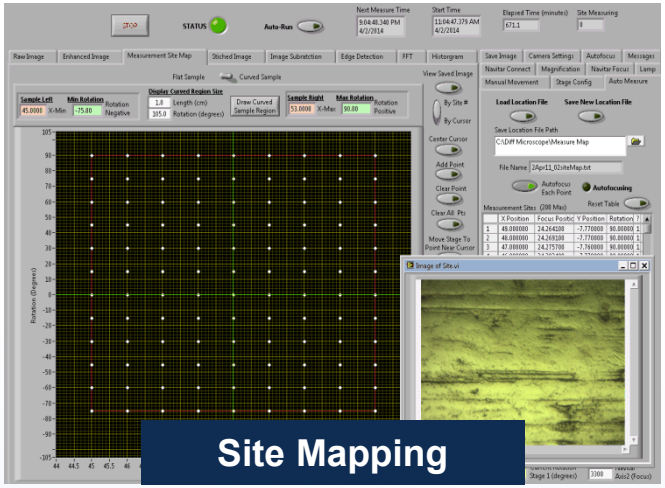


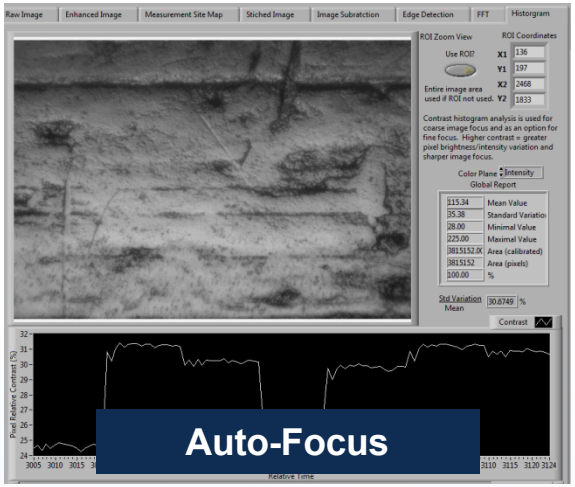
Environmental Control via a Traditional Mixed Flowing Gas System

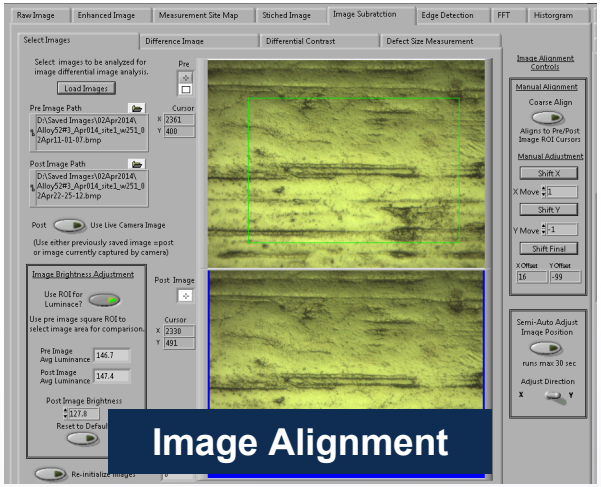


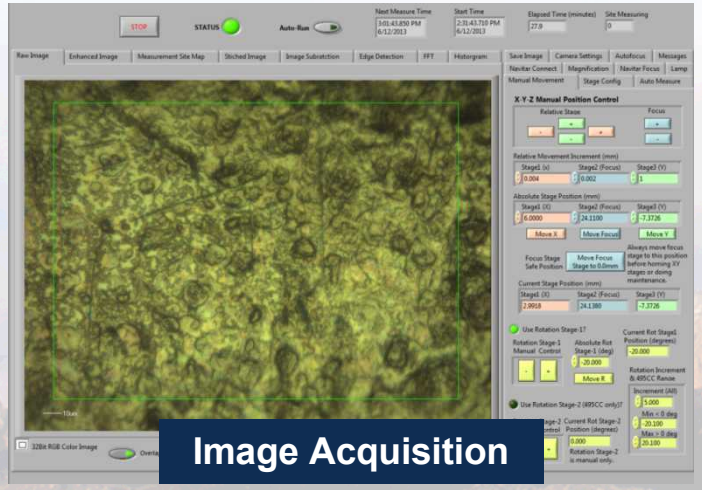
LabView Software for Automation

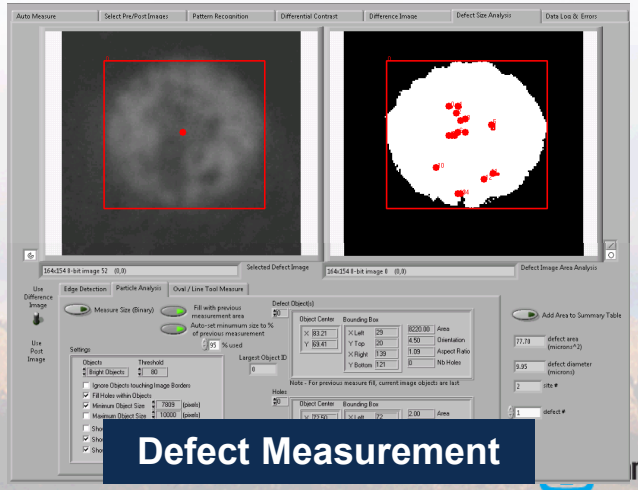
- LabView control software enable automated continuous image acquisition with the ability to resolve 1-2 micron features and provide repeatable, sub-micron, sample positional control.











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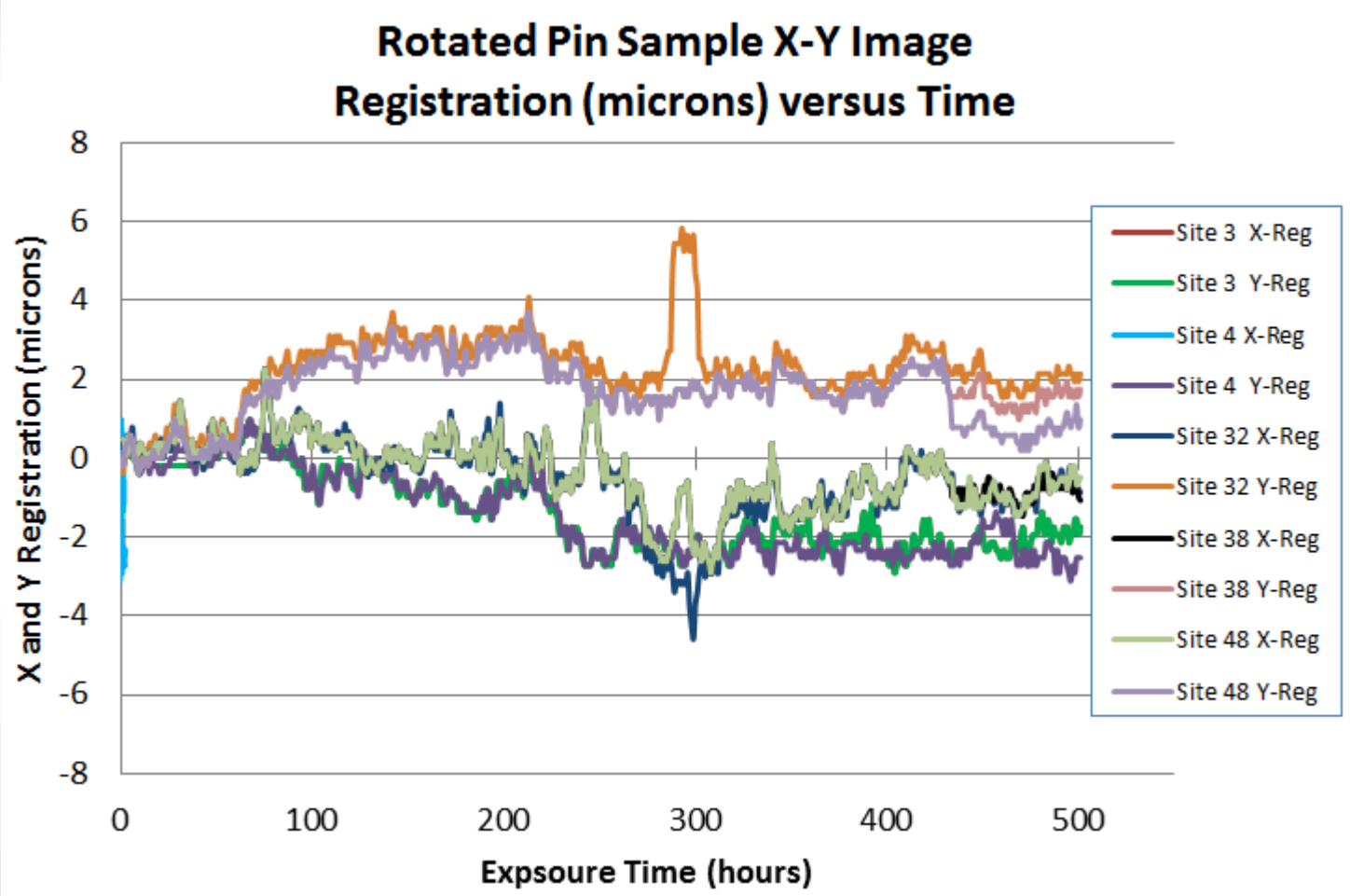
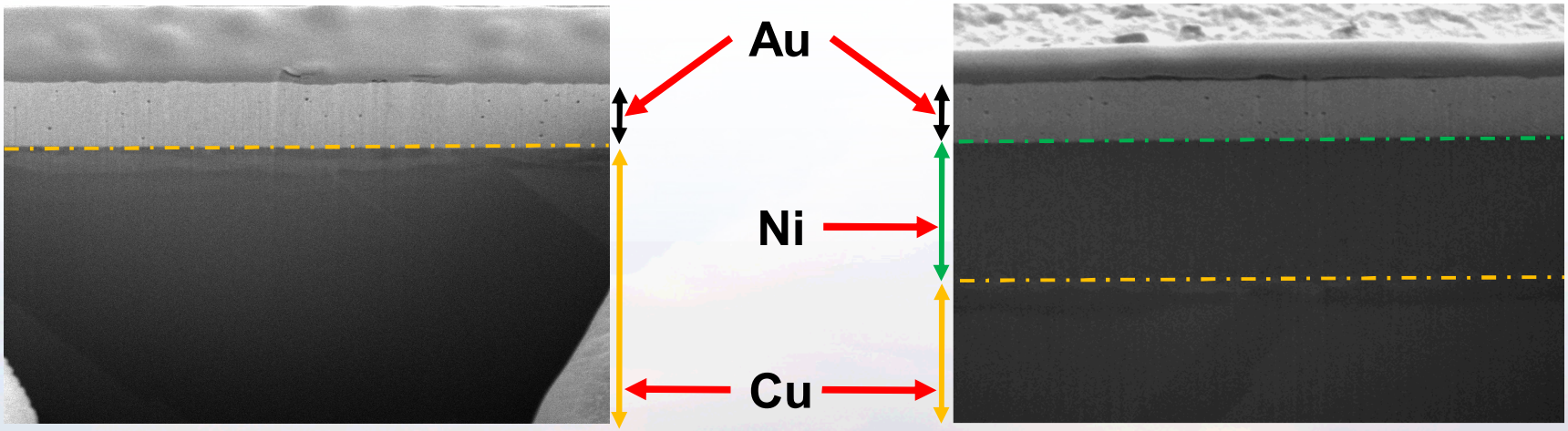
System Reliability and Robustness



- **Monitoring multiple sites over time requires high positional accuracy**
  - Accuracy of approximately +/- 5 microns for fully automated operation demonstrated for 500 hrs.
  - Approximately 1,000,000 motorized stages movements occurred during this time period without any faults or requiring any manual intervention

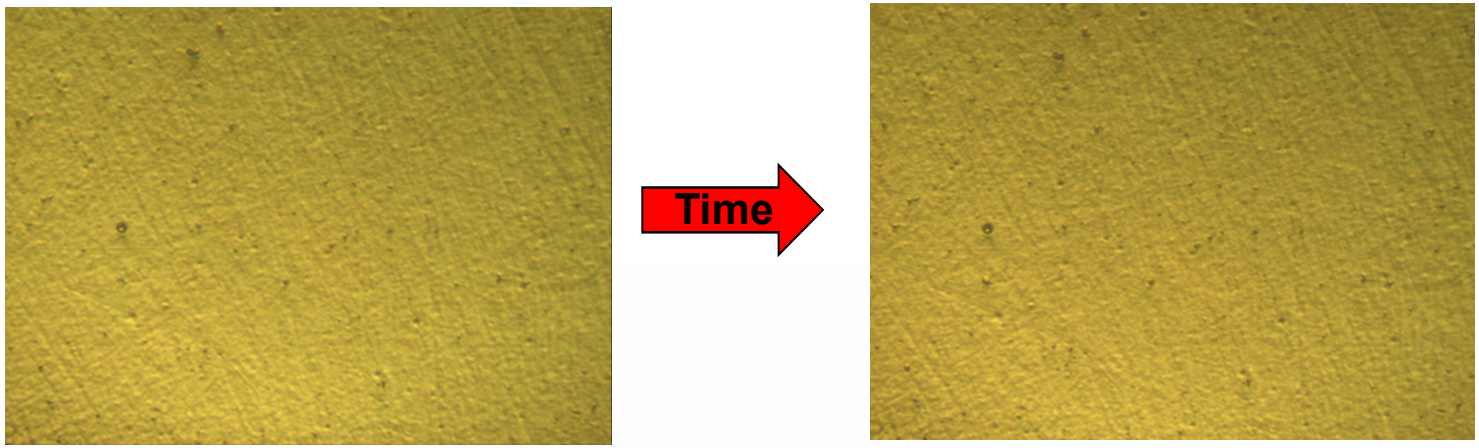
Application:
Atmospheric Corrosion of Connectors

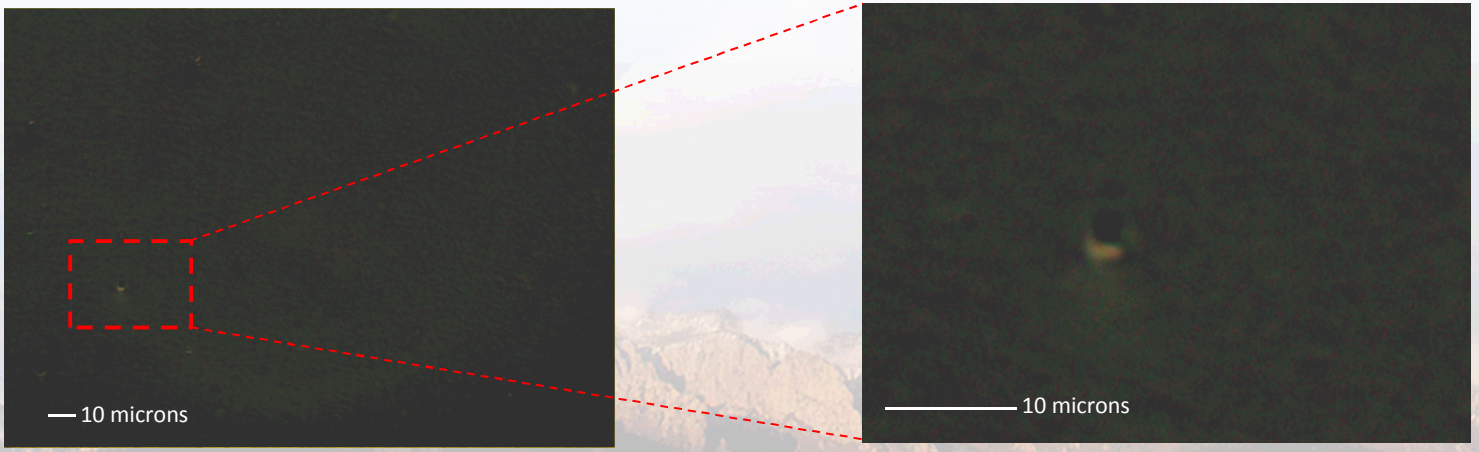
- **Oxygen free copper panels, mechanically lapped to a 15-20nm RMS finish**
- **Electroplated with one of two metallurgies**
  - 2.5 µm Au (ASTM Type I, Code C, class 2.5)
  - 2.5 µm Au over 5 µm Ni



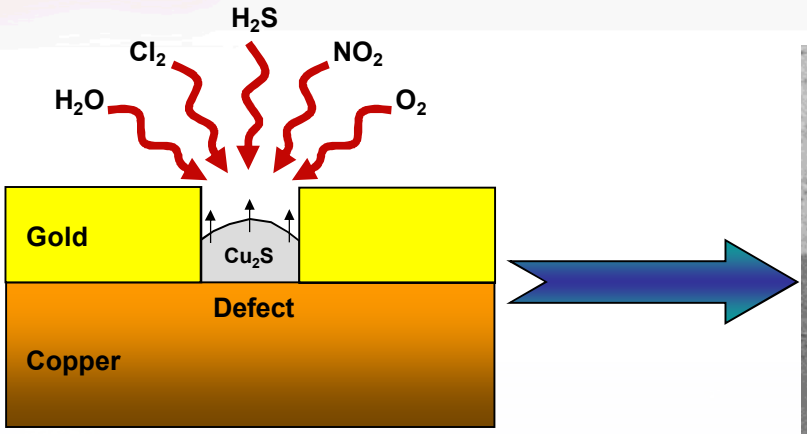
- **Models developed for the electrical performance of such contacts depend on accurate characterization of the corrosion process over time (corrosion site density, size distribution, etc.)**

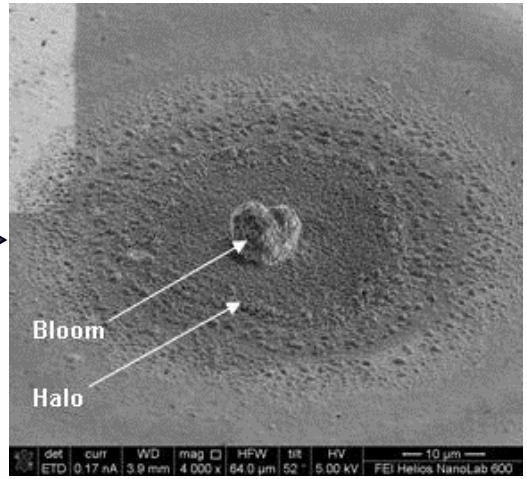
Data Acquisition and Image Analysis Software Resolving Individual Defects

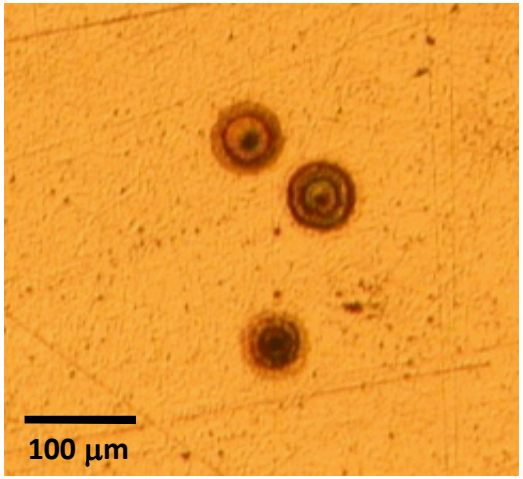




Corrosion Morphology

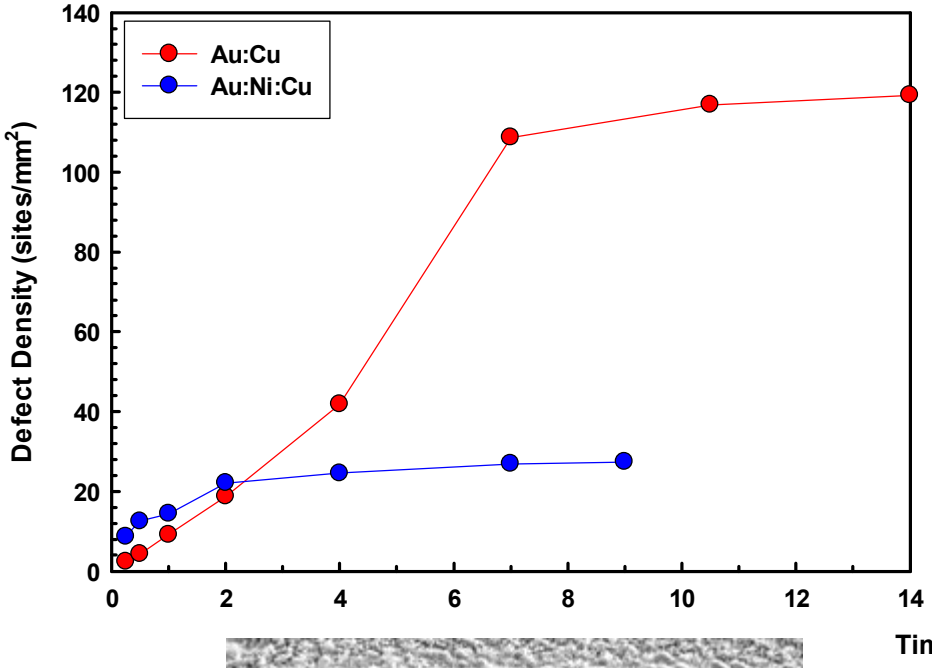


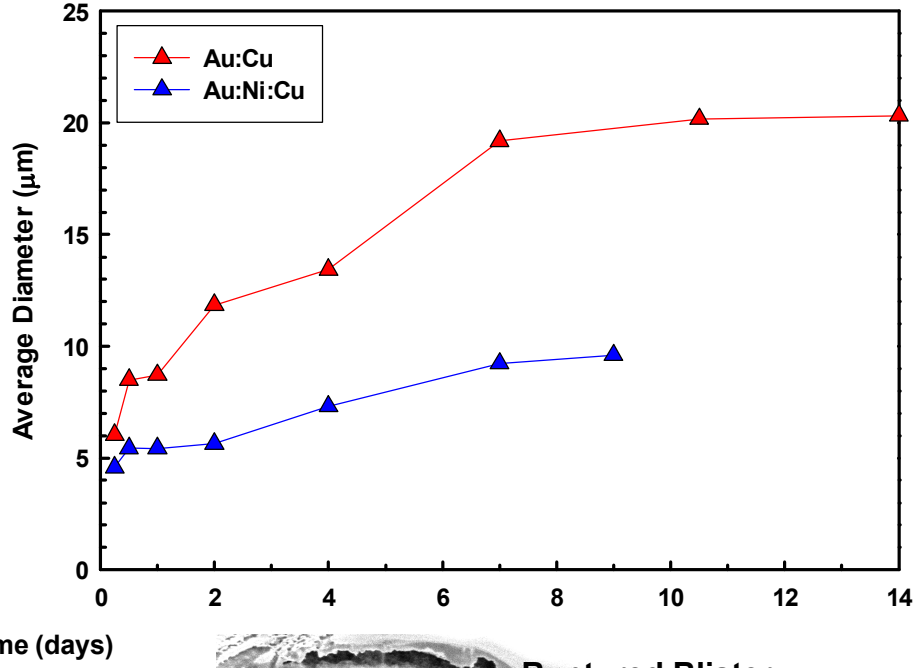


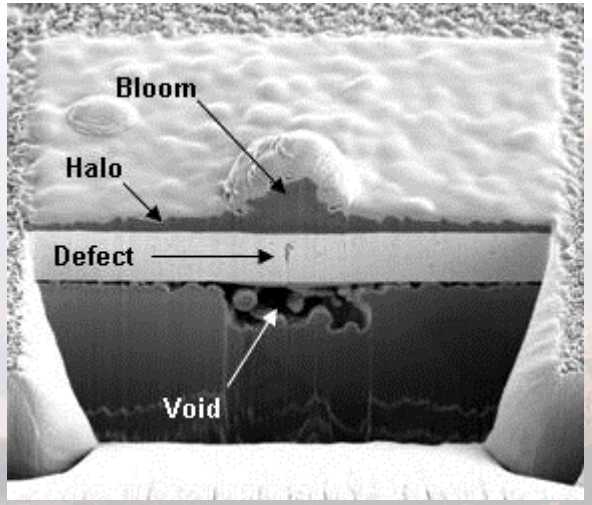


- **Existing models require precise knowledge of specific aspects of the corrosion process**
  - Number density and size distribution of corrosion sites
  - Growth behavior of individual corrosion sites
- **Prior investigations (at SNL and in the literature) provide very limited information on the aforementioned quantities.**

Image Analysis – Defect Density vs. Time
Au:Ni:Cu vs. Au:Cu Samples







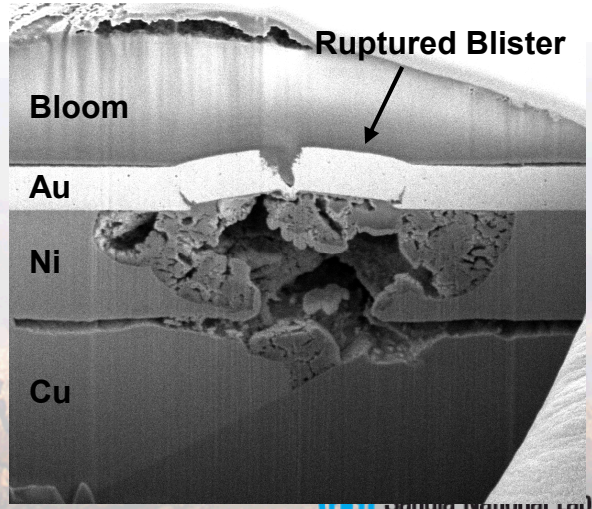
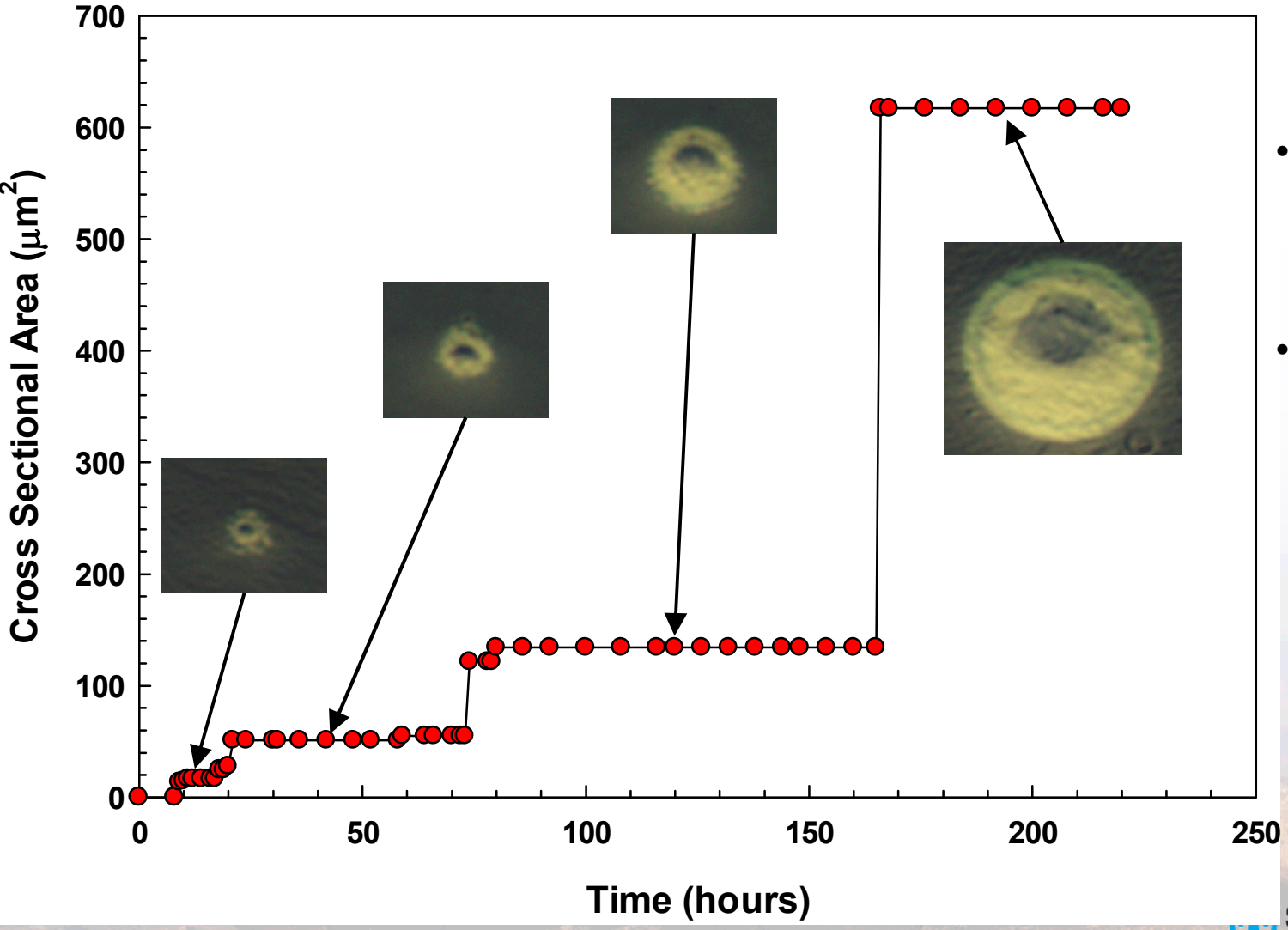


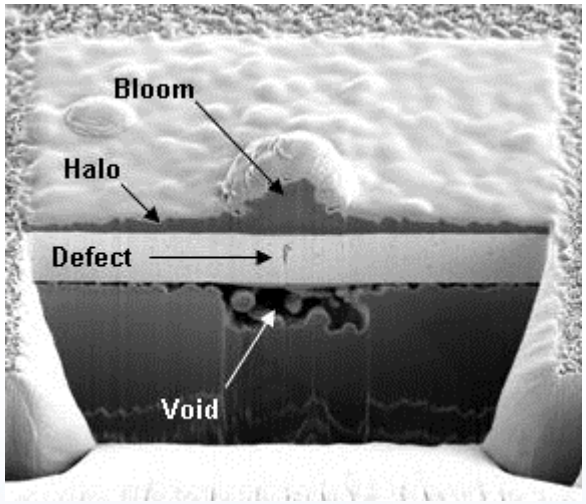
Image Analysis on a Planar Surface
Individual Defect Size vs. Time for Au:Ni:Cu Sample



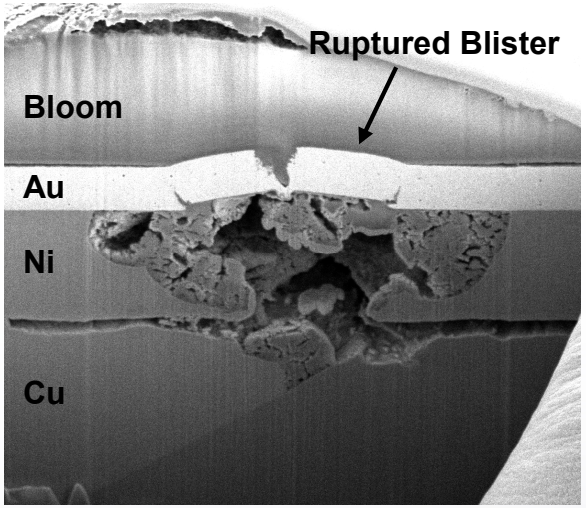
- Stepped growth process
- Large size correlated with breach of Ni layer and creep of copper sulfide

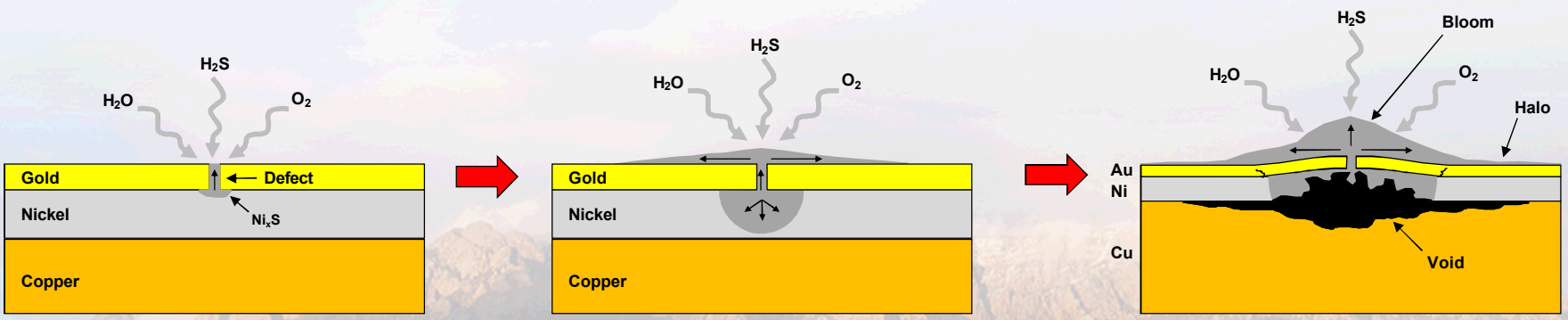
Why Is There A Multi-Stage Growth Process?

Au:Cu



Au:Ni:Cu





What Happens When the Cu is Removed:
Early stage growth is similar for Au:Ni plated Alloy 52

3 Weeks, Battelle Class 2 – **Pre-Exposure** = **Corrosion Sites**

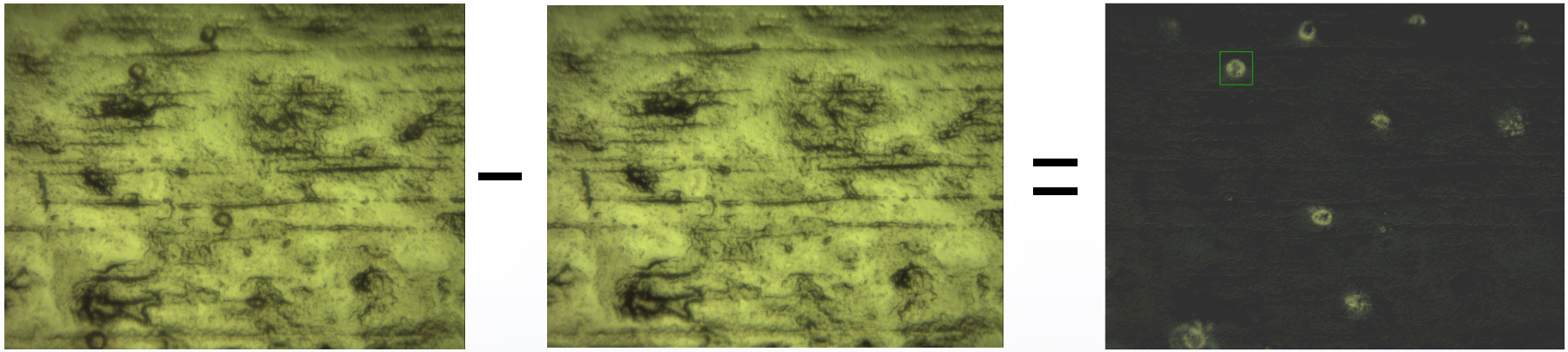
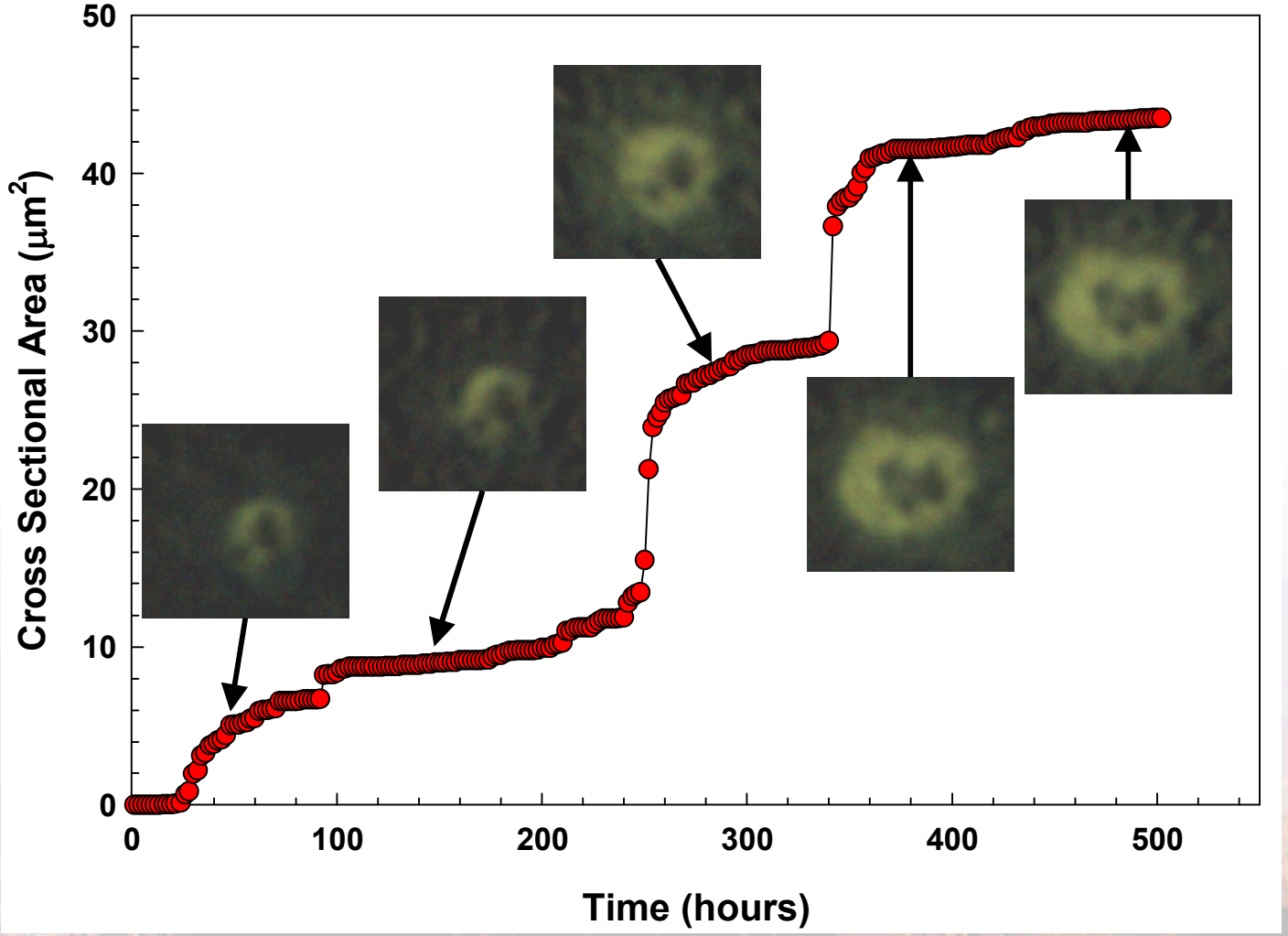


Image width = 250 µm

- Corrosion sites are blisters and do not develop into the traditional bloom and halo associated with plated copper, consistent with the theory that the initial stages of attack on gold plated samples with a Ni underlayer is the development of a nickel sulfide containing blister, which ruptures once corrosion has reached the underlying copper.

Image Analysis on a Curved Surface Individual Defect Size vs. Time for Au:Ni:Alloy 52 Sample



- Stepped growth process
- Smaller size (consistent with blister formation)

Summary/Conclusions

- **Differential imaging system has been developed that enables samples undergoing atmospheric corrosion to be imaged in real time**
  - System functionality demonstrated on noble metal plated copper as used in microelectronic connectors

- **System has provided considerable insight into the sulfidation process for noble metal plated copper**
  - Unbiased measure of corrosion site density and size distribution
  - Time dependent evaluation of corrosion site size
    - Multi-stage growth process revealed
    - While additional work is necessary to confirm, experiments to date agree with mechanism proposed (Enos, 2010) for site nucleation, growth, and passivation/stifling

D.G. Enos, Corrosion Vol. 66, no. 10 (2010), pp. 105003-1.