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Differential Imaging Microscopy of Physically and Chemically Complex Surfaces Relevant to Materials Surveillance

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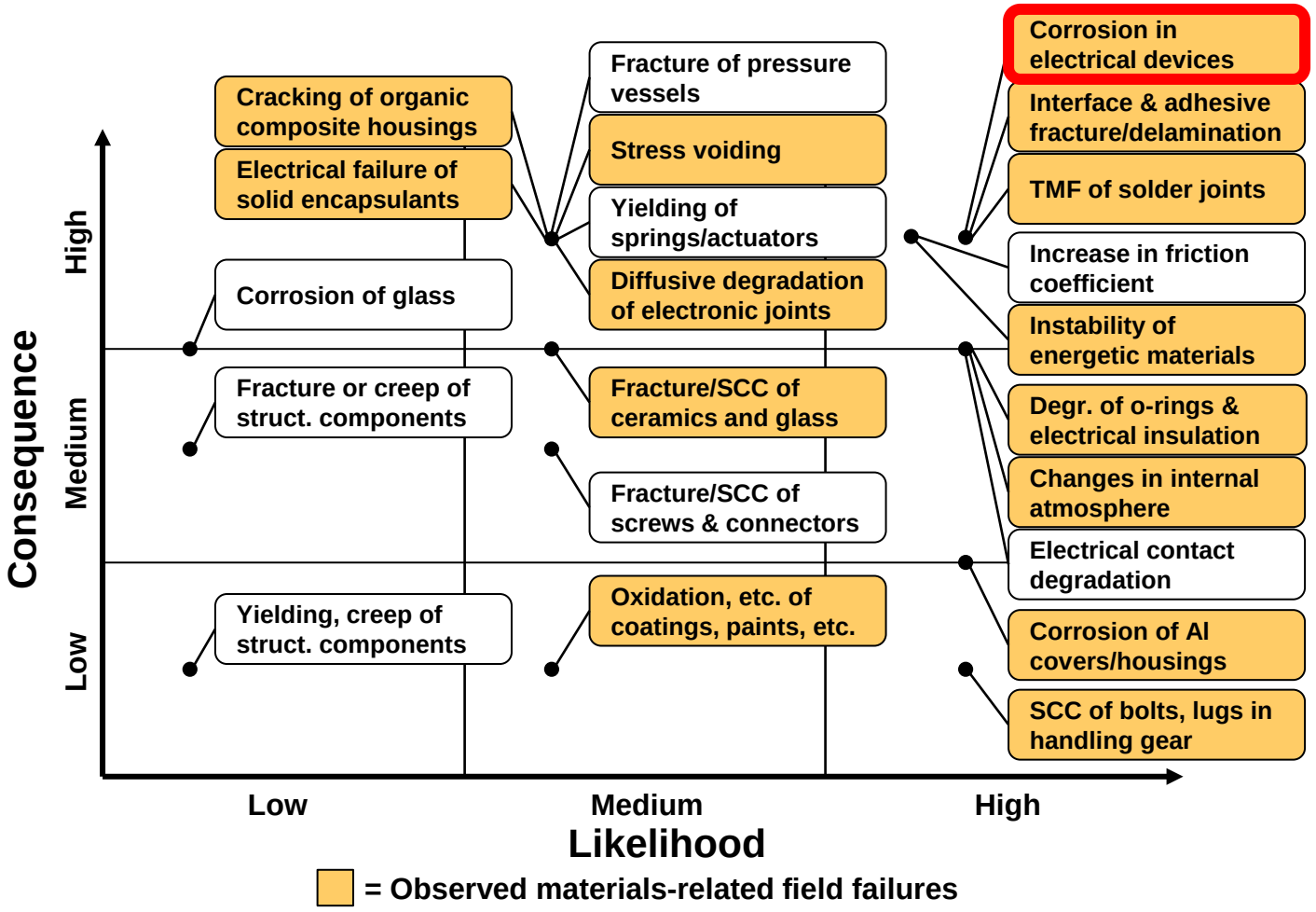
Atmospheric Corrosion of Electrical Devices – High Consequence, but Difficult to Interrogate

Materials-degradation modes possibly relevant to high-consequence/high-reliability military systems.

From Braithwaite, et al., SAND2003-0359



- Atmospheric corrosion processes can not be assessed via electrochemical techniques
- An optical technique is needed to overcome this issue

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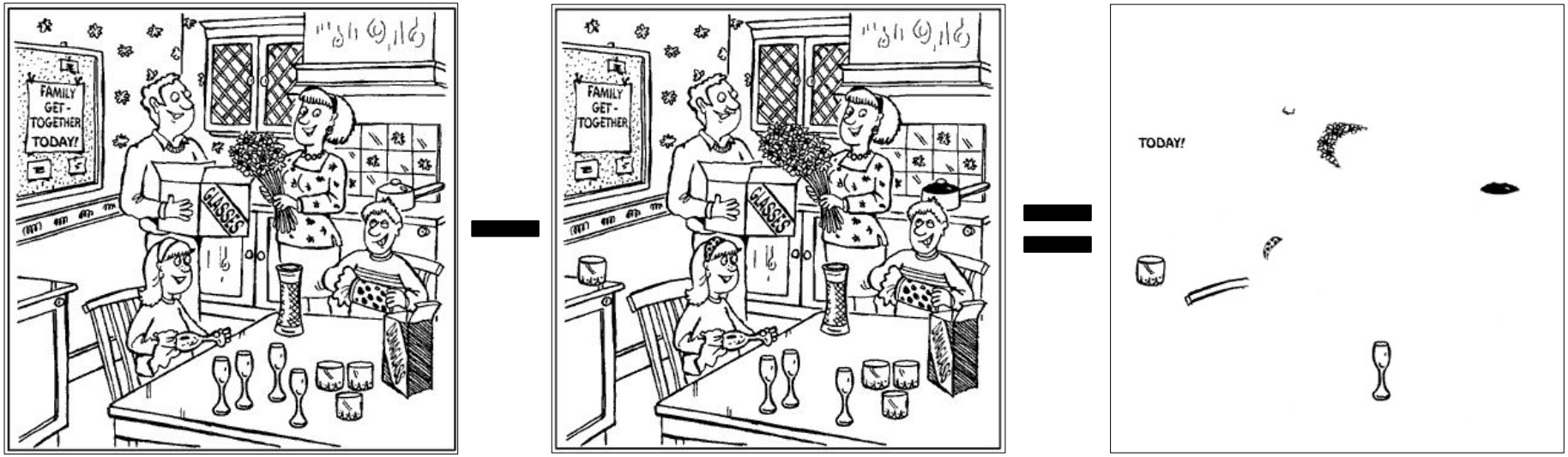
What is Differential Imaging?

- **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**

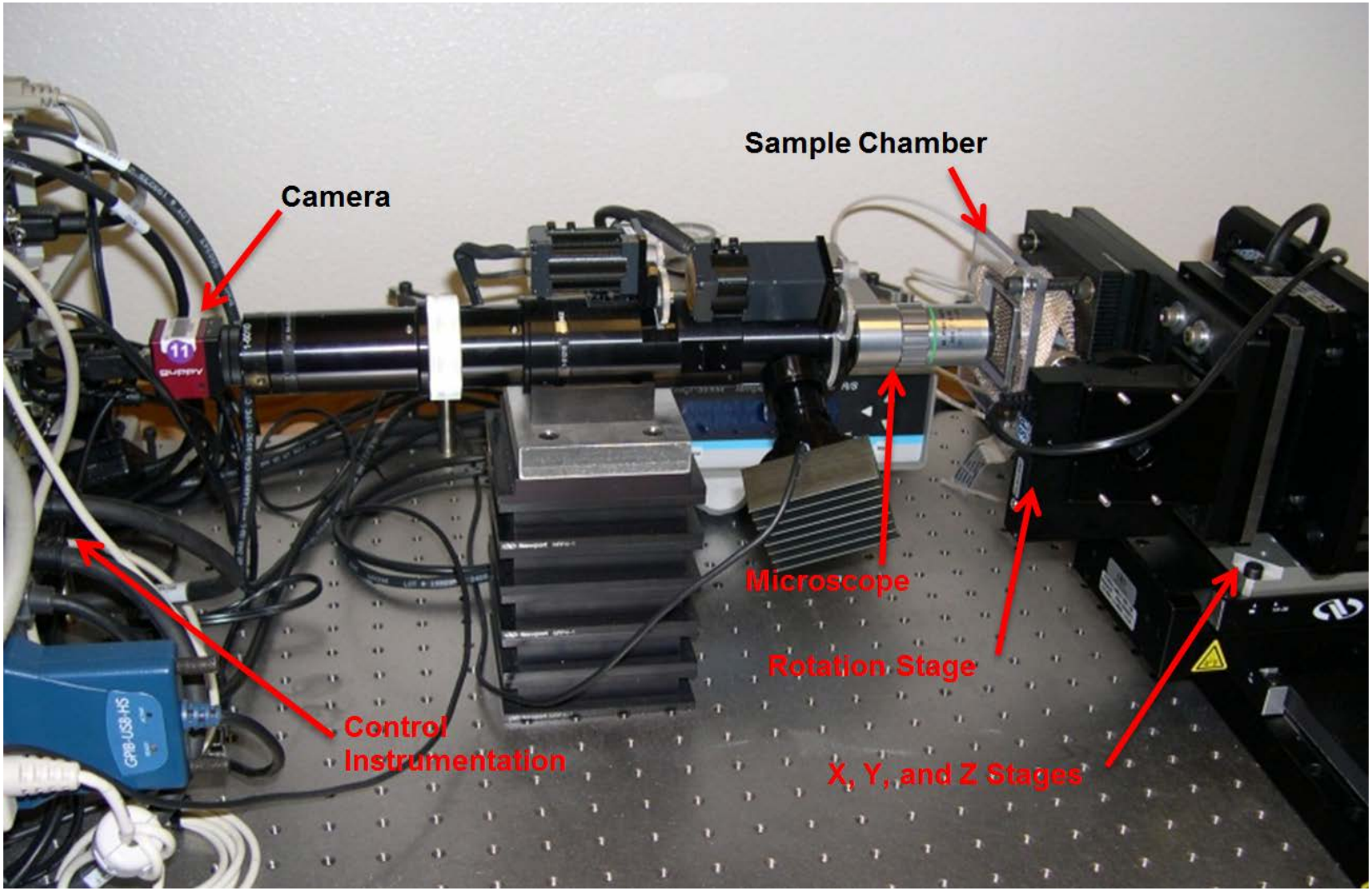
R.S. Huang, C.J. Lin, H.S. Isaacs, Electrochemical and Solid-State Letters, Vol. 9, no. 2 (2006) pp. B11-B14.

System Configuration

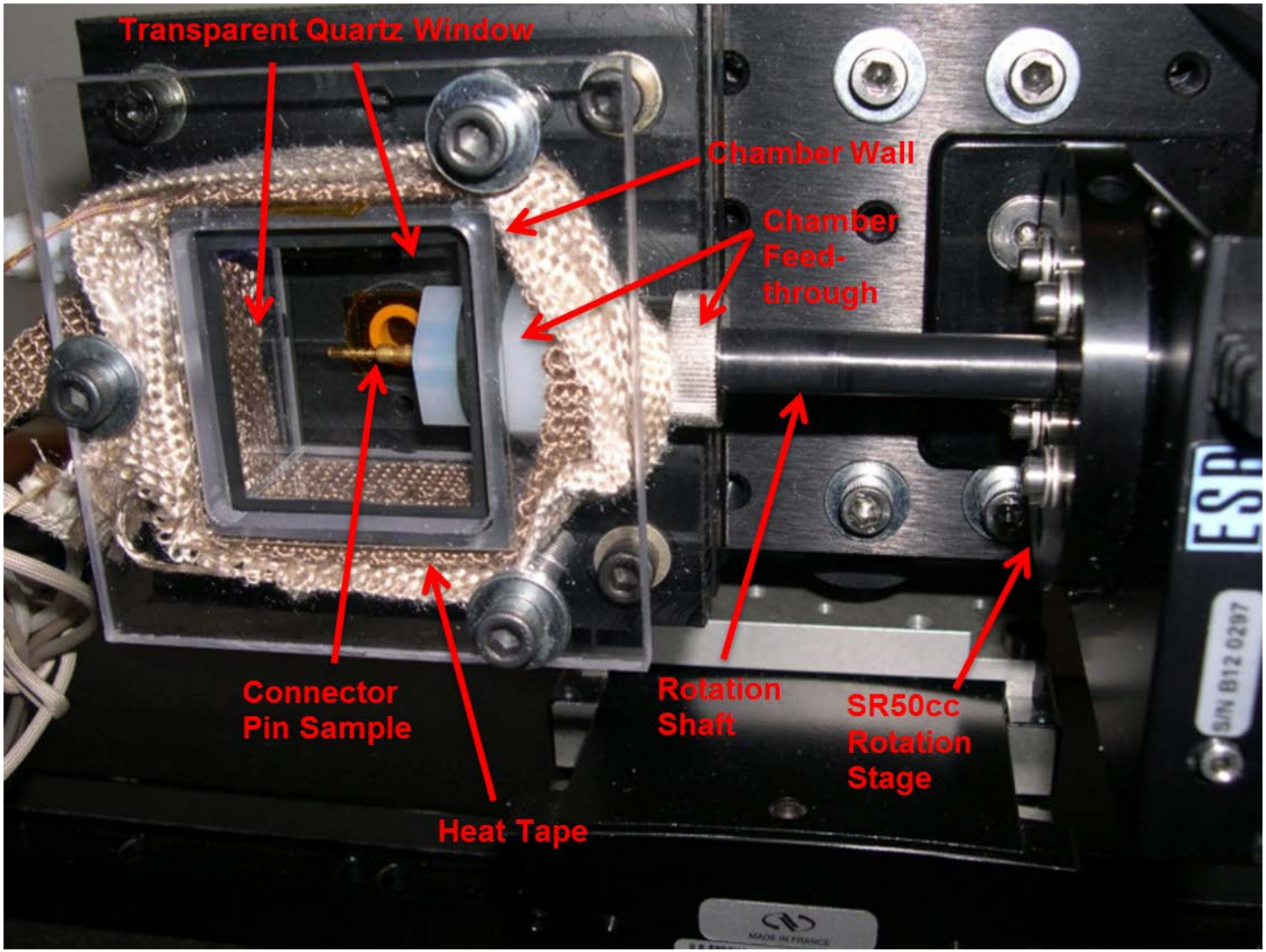
- **Goal: develop a system capable of monitoring a corroding surface in-situ**
  - Must be non-destructive
  - Need to be able to map a surface
  - Need to have flexibility in terms of the specimen geometry and environmental conditions

- **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)

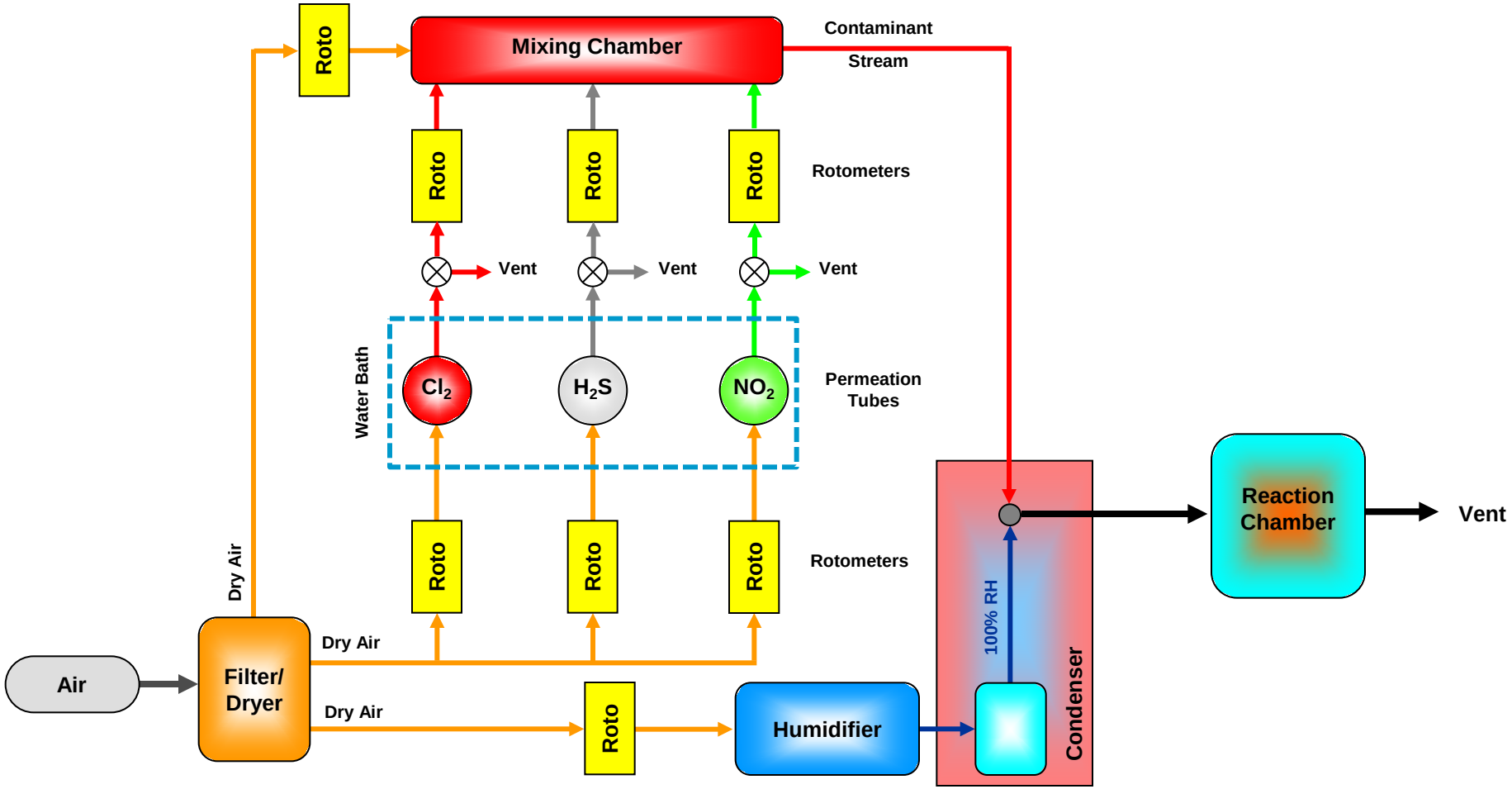
Overall System Configuration



The Exposure Chamber
(One Example)



Environmental Control via a Traditional Mixed Flowing Gas System



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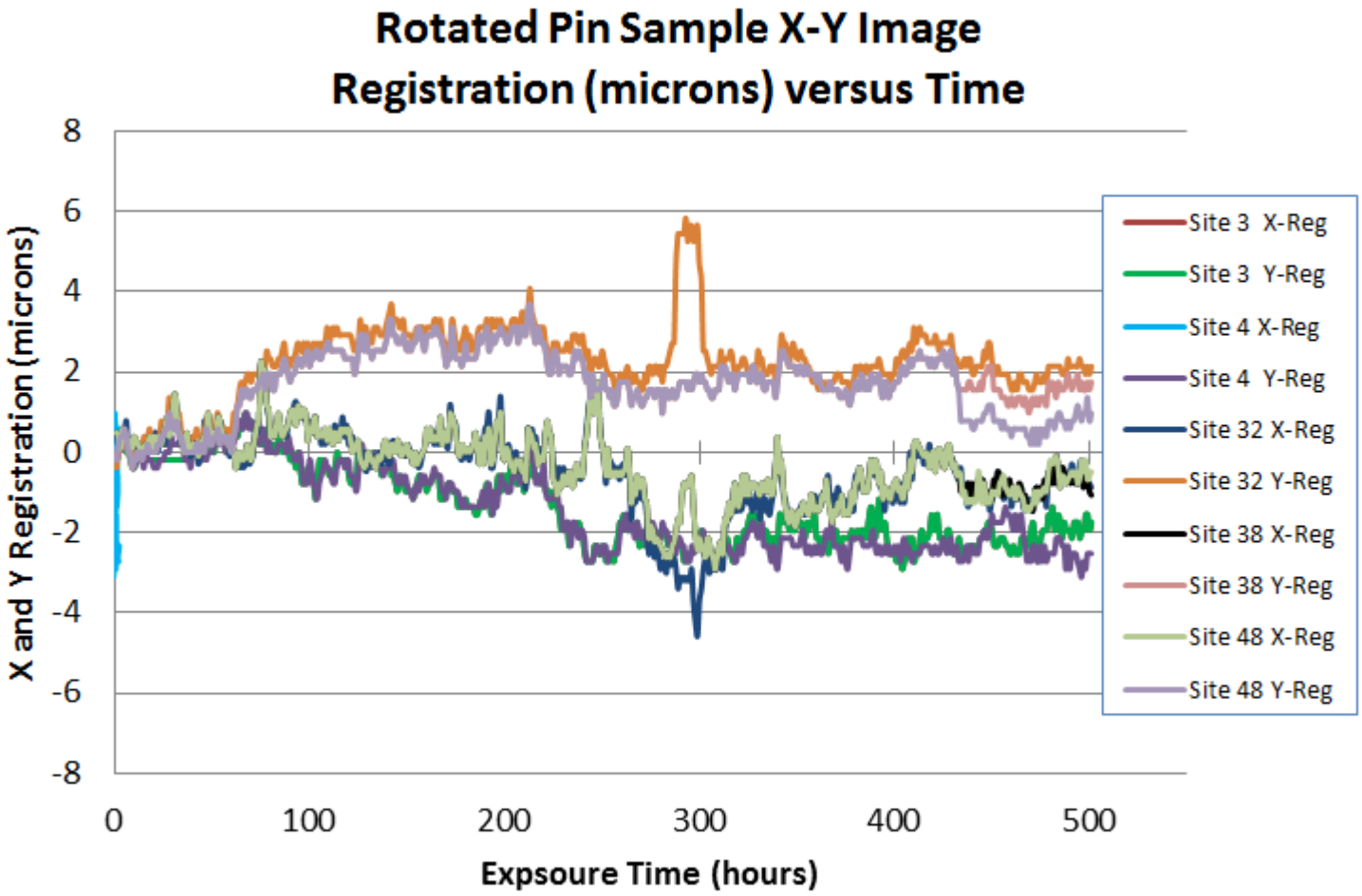
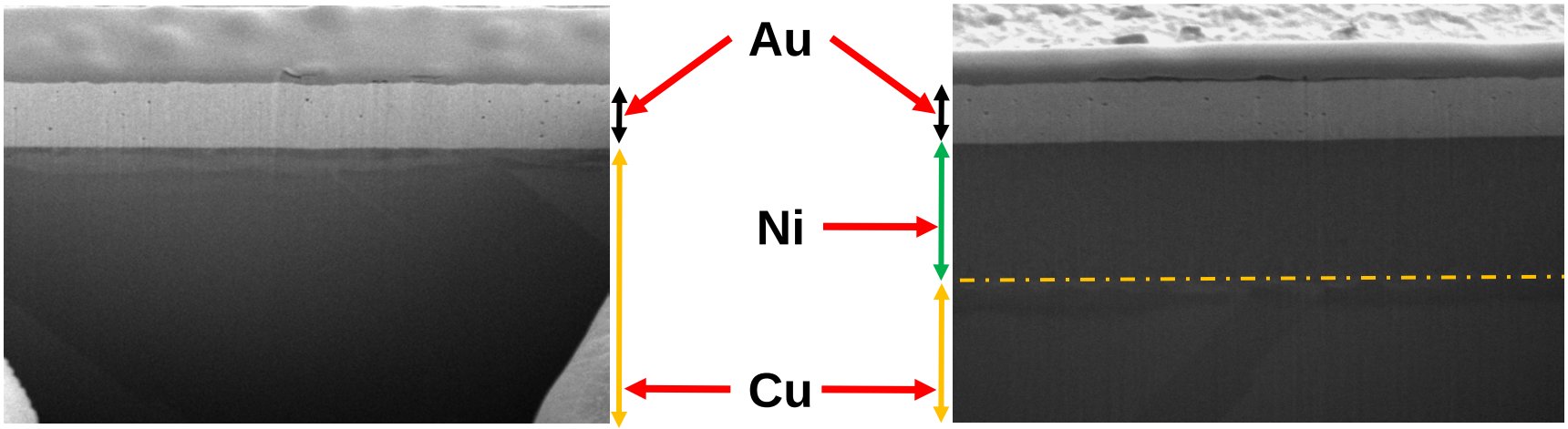
System Reliability and Robustness



- **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

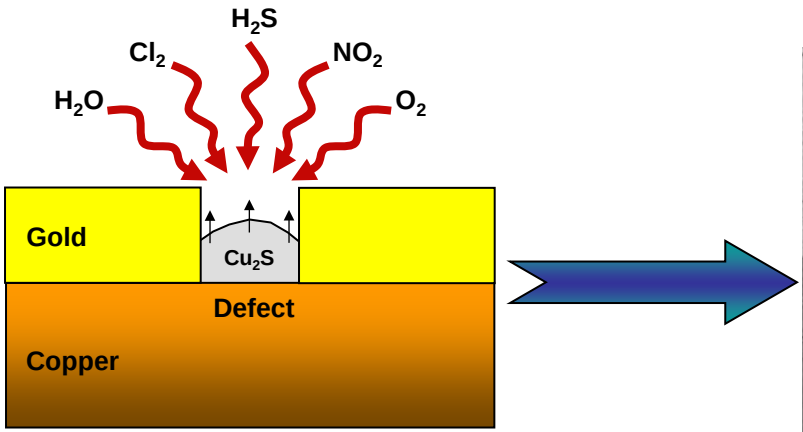
Application to Surveillance Relevant Materials: Atmospheric Corrosion of Connectors

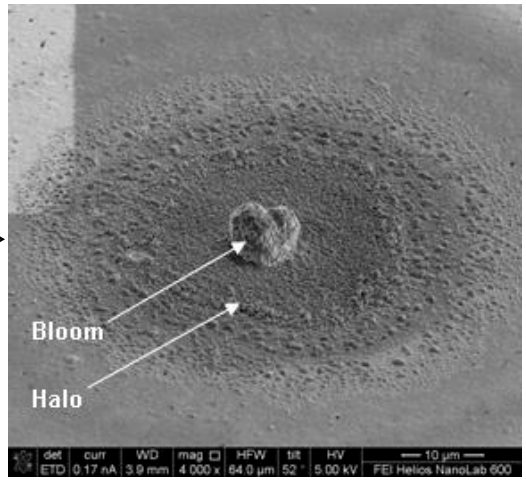
- **Material used for microelectronic connectors throughout the stockpile**
- **Oxygen free copper panels, mechanically lapped to a 15-20nm RMS finish**
- **Electroplated with one of two metallurgies**
  - 2.5 µm Au (ASTM Type I, Code C, class 2.5)
  - 2.5 µm Au over 5 µm Ni

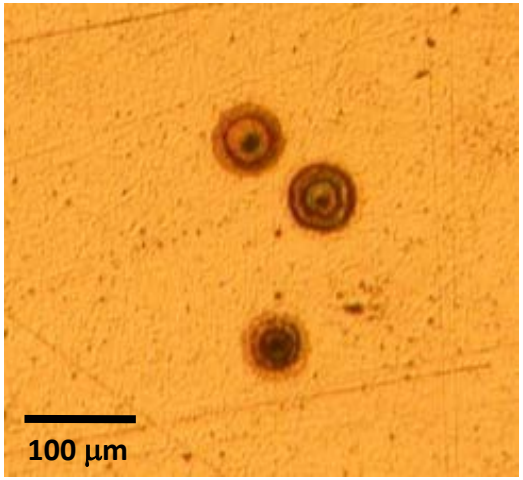


- **Models developed for such contacts (e.g., SAND08-5737) depend on accurate measure of corrosion process over time (corrosion site density, size distribution, etc.)**

Corrosion Morphology

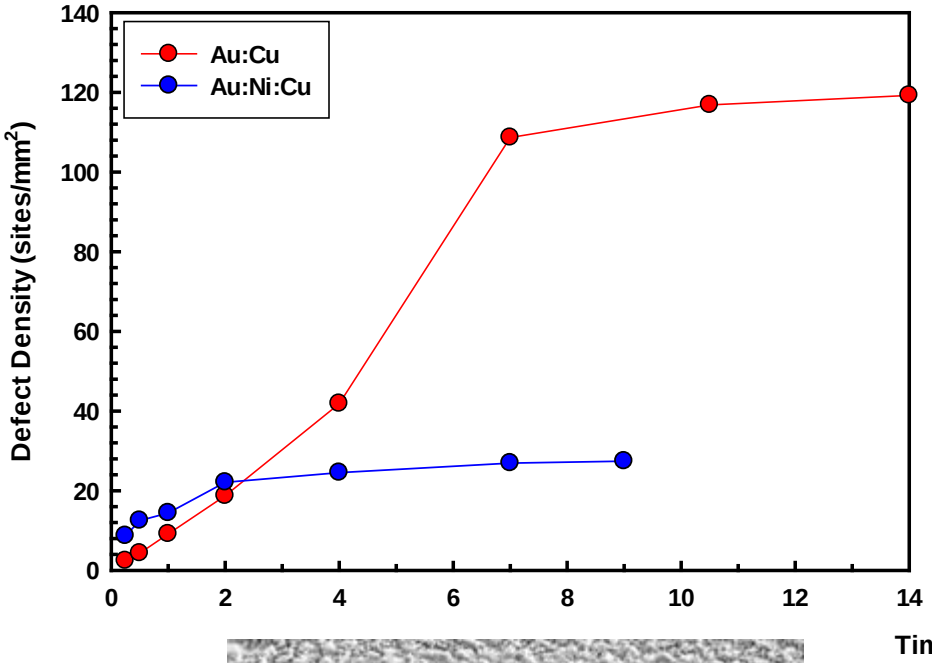


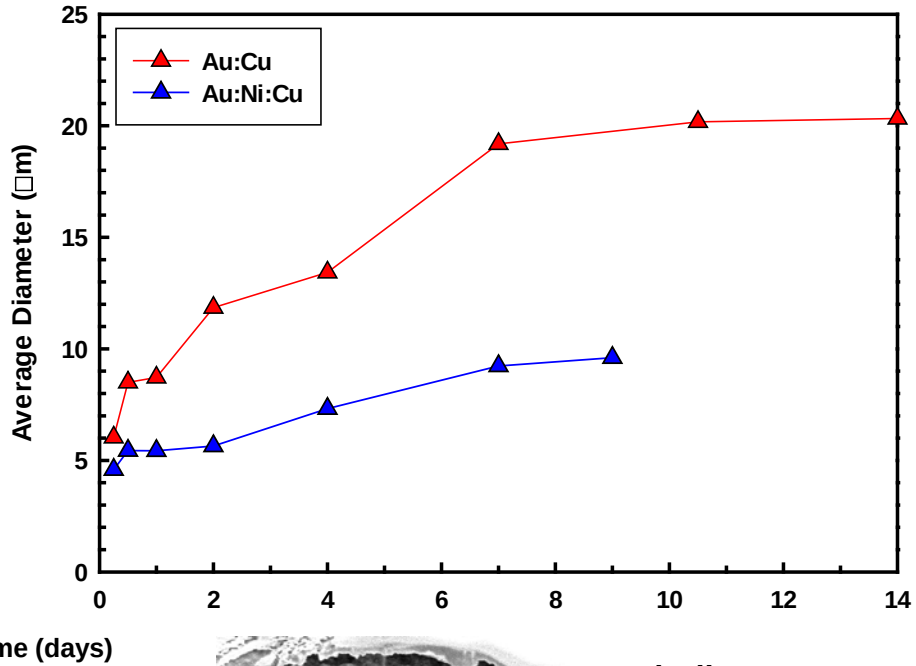


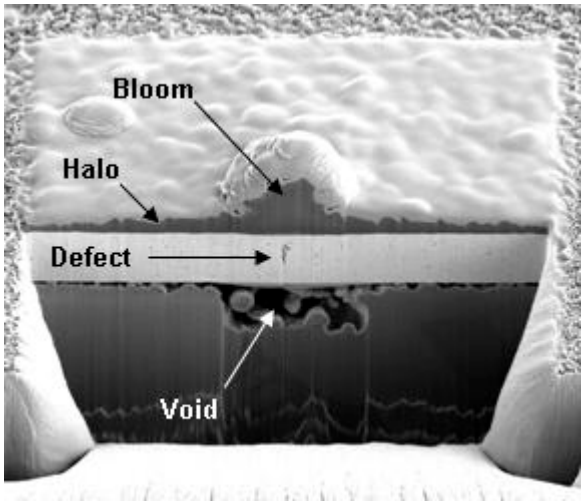


- **Existing models require precise knowledge of specific aspects of the corrosion process**
  - Number density and size distribution of corrosion sites
  - Growth behavior of individual corrosion sites
- **Prior investigations (at SNL and in the literature) provide very limited information on the aforementioned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

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







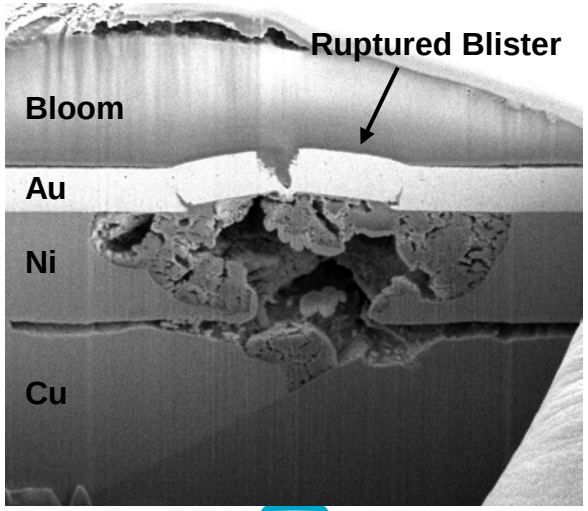


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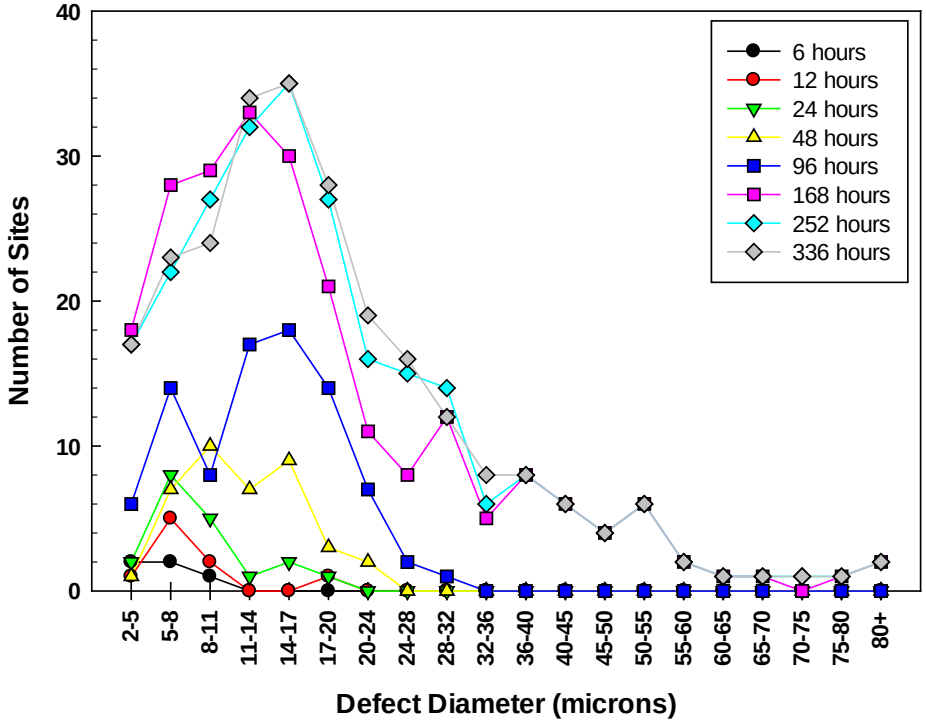
Image Analysis
Defect Size Distribution vs. Time - Au:Cu vs. Au:Ni:Cu



Au:Cu

Au:Ni:Cu

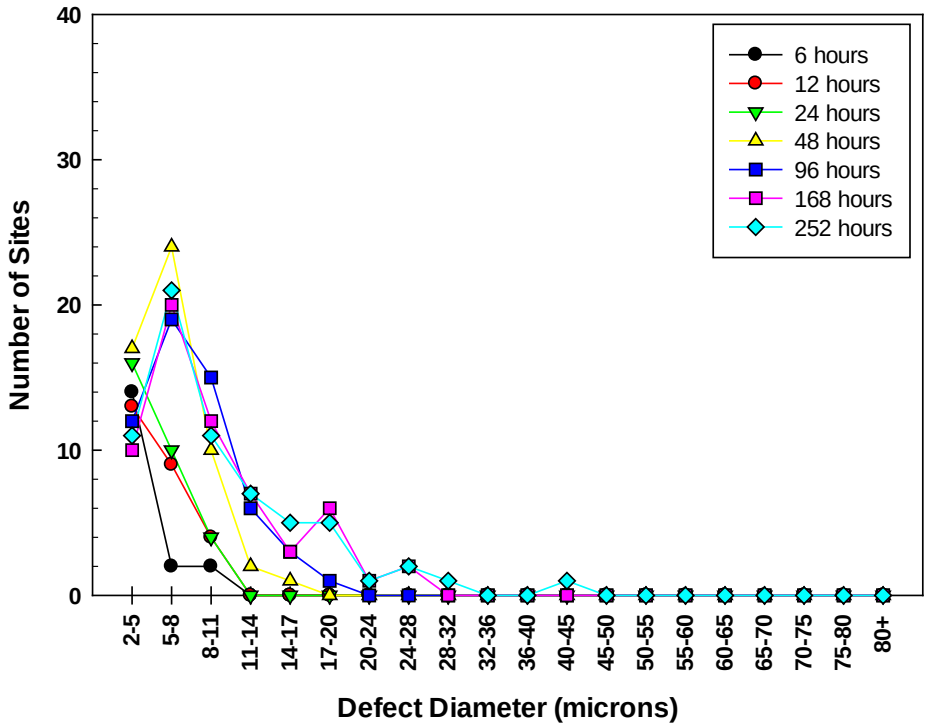


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Image Analysis on a Planar Surface
Individual Defect Size vs. Time for Au:Ni:Cu Sample

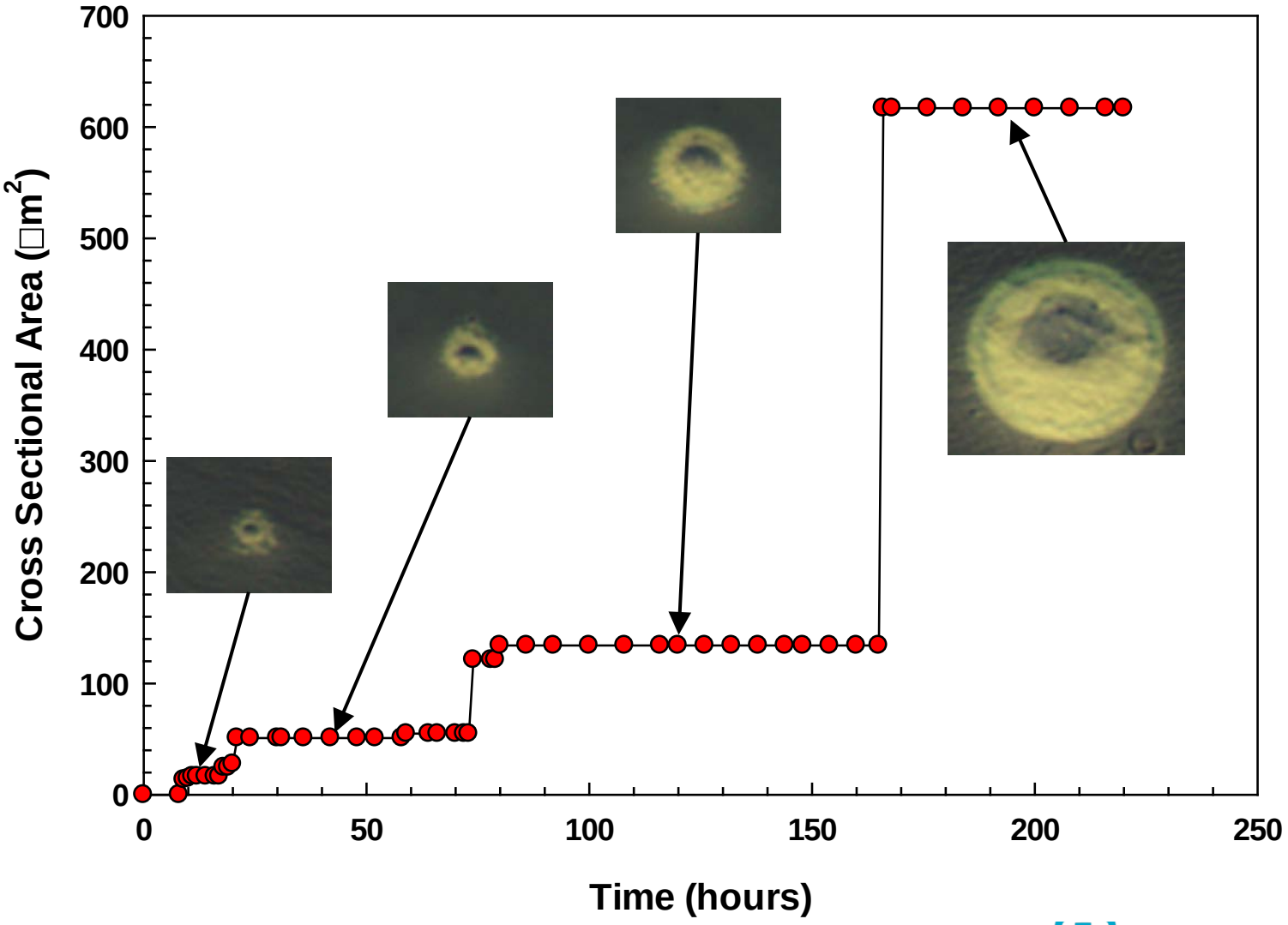
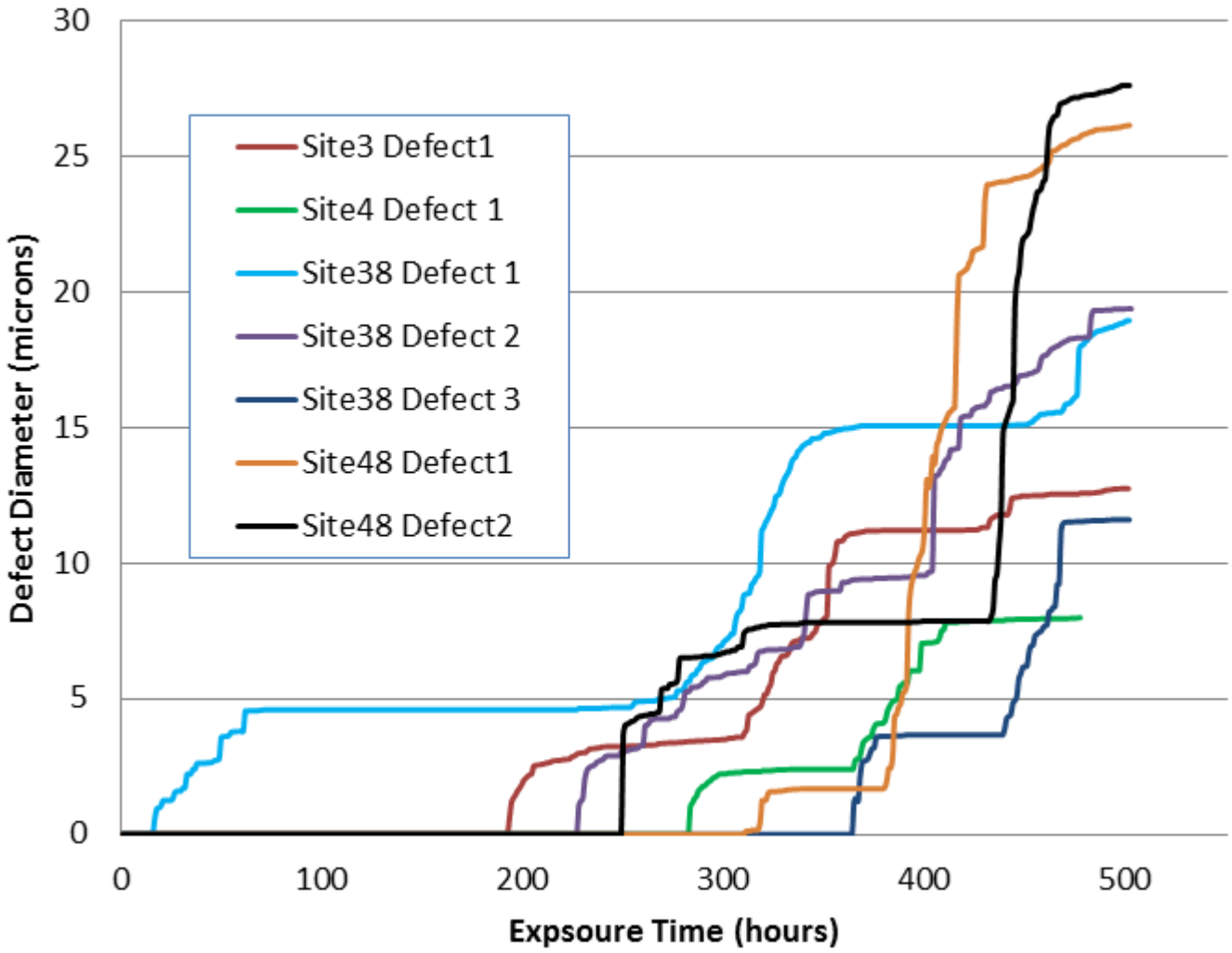


Image Analysis on a Connector Pin
Individual Defect Size vs. Time Connector pin



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.

Future Work

- **Development of system project complete after FY13**
  - Documentation
  - Software refinement (continuous)
  - Reliability demonstration

- **Now a viable analysis technique**
  - Allows for customization
  - Provides a non-destructive analysis of surface over time
  - Currently focused on connector degradation (noble metal plated copper and Alloy 52)
  - Combine with techniques such as contact resistance measurement to establish link between degradation and performance