From Dental Composites to Degraded MLI:

Selected short stories from the world of a Materials Engineer

B. Reed

Materials Engineering Branch

March 4, 2008

Problem Statement:

Tooth Structure Overview

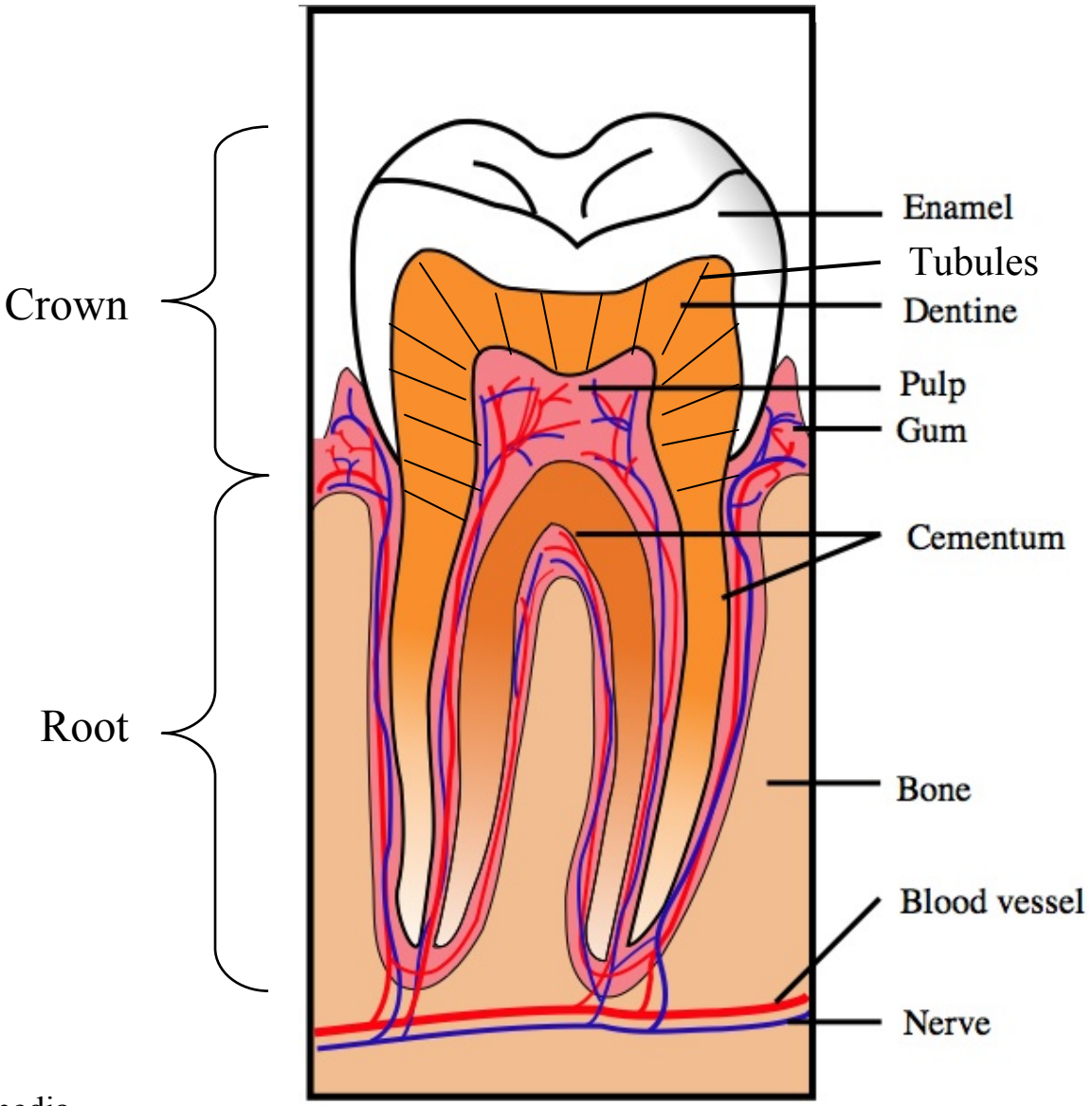
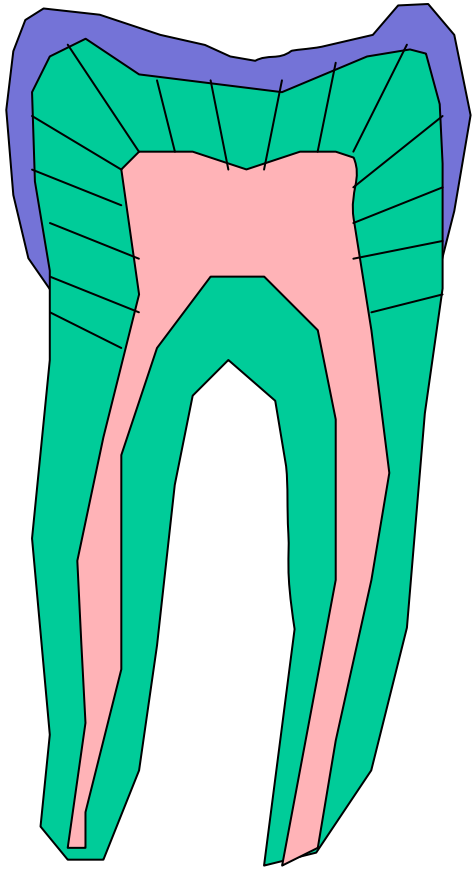


Image lifted from Wikipedia

Chemical structure of tooth components

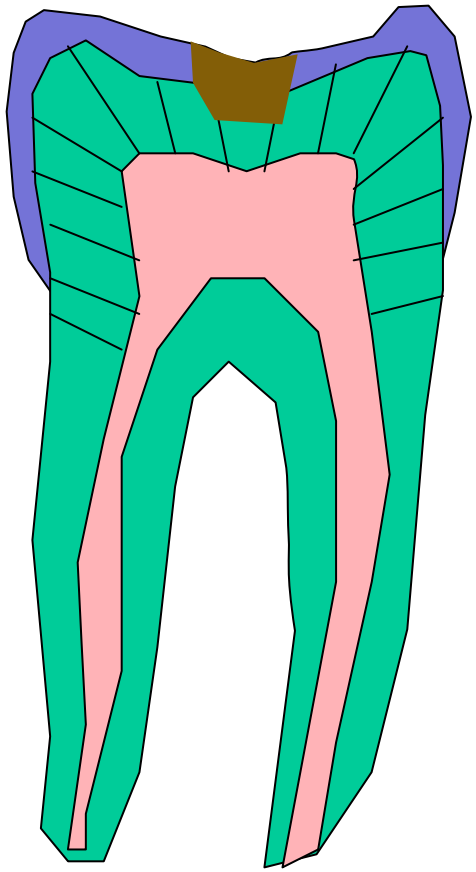


- Enamel: hard outer coating
  - 96 wt% hydroxyapatite
    - tetracalcium phosphate $Ca_4O(PO_4)_2$
    - dicalcium phosphate $Ca_2P_2O_7$
  - 2 wt% protiens
  - 2 wt% H_2O
- Dentin: softer inner material
  - 70 wt% hydroxyapatite
  - 20 wt% collagen
  - 10 wt% H_2O

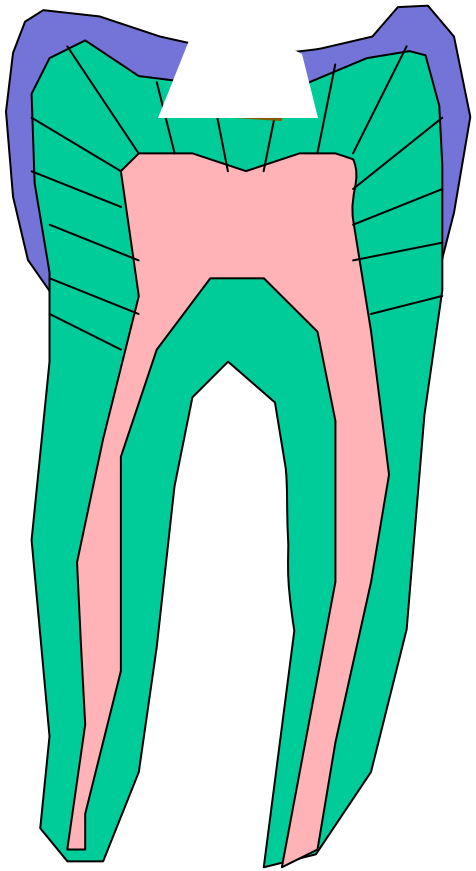
Mechanism of Dental Caries

1. Plaque bacteria ingest sugar (starch) that passes through the mouth.
2. The waste byproduct of the plaque is acidic (low pH).
3. The reduced pH increases the solubility of the hydroxyapatite causing loss of material.
4. Once the enamel has been removed the softer dentin gets attacked exposing the openings of dentin tubules.
5. Fluid then moves back and forth in the tubules due to rapid temperature changes or air passing over.
6. The moving fluid stimulates the nerves in the pulp and is registered as pain.

Carious Lesion



Cavity Prep



Filling Materials

Currently used (years in use)

- Au foil (2000)
- Silver amalgam (160)
  - Ag & Sn powder
  - liquid Hg
- Composite (45)
  - organic matrix
  - inorganic filler

Previously used

- Bone
- Ivory
- Sea Shell
- Ceramic
- Stone
- Pb

Advantages/Disadvantages of Composite Fillings with Adhesive Bonding

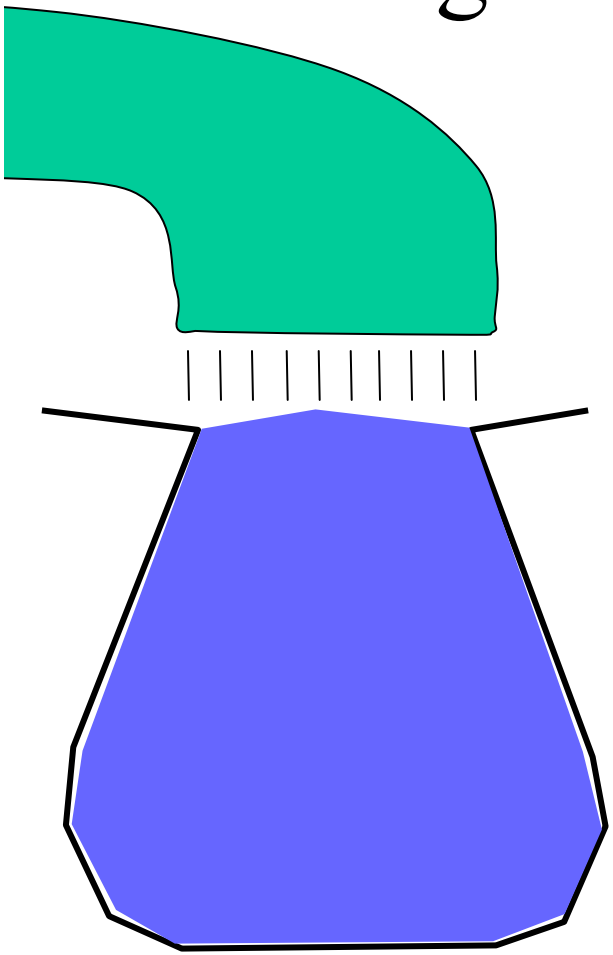
Pro

- Natural tooth color
- Semi translucent
- Better distribution of functional forces
- Reinforce weakened teeth

Con

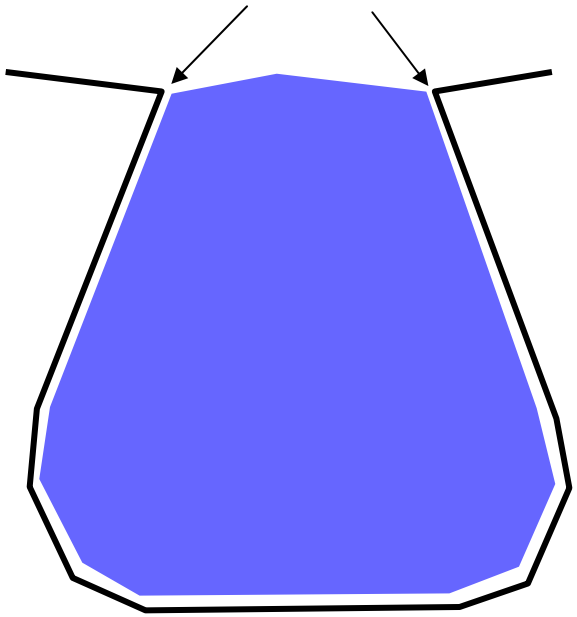
- Lower service life
  - secondary caries caused by polymerization shrinkage
- Lower mechanical properties

Curing of a dental composite



cavity prep stuffed with
unpolymerized composite

small gap for food &
plaque infiltration



cured (polymerized)
filling

Steps to Improve Bond Strength

- Surface Prep
- Like bonds to like
  - when this is impractical, use primer

- Oral environment challenges
  - patient is alive
  - hot/cold beverages
  - food/pharmaceutical staining
  - mastication - three body wear
  - aqueous/saliva

Theories of Adhesion

1. Mechanical
   - micromechanical interlocking
2. Adsorption - chemical bonds
   - Primary - ionic and covalent
   - Secondary - hydrogen, van der Waals

Acid Etching

Enamel

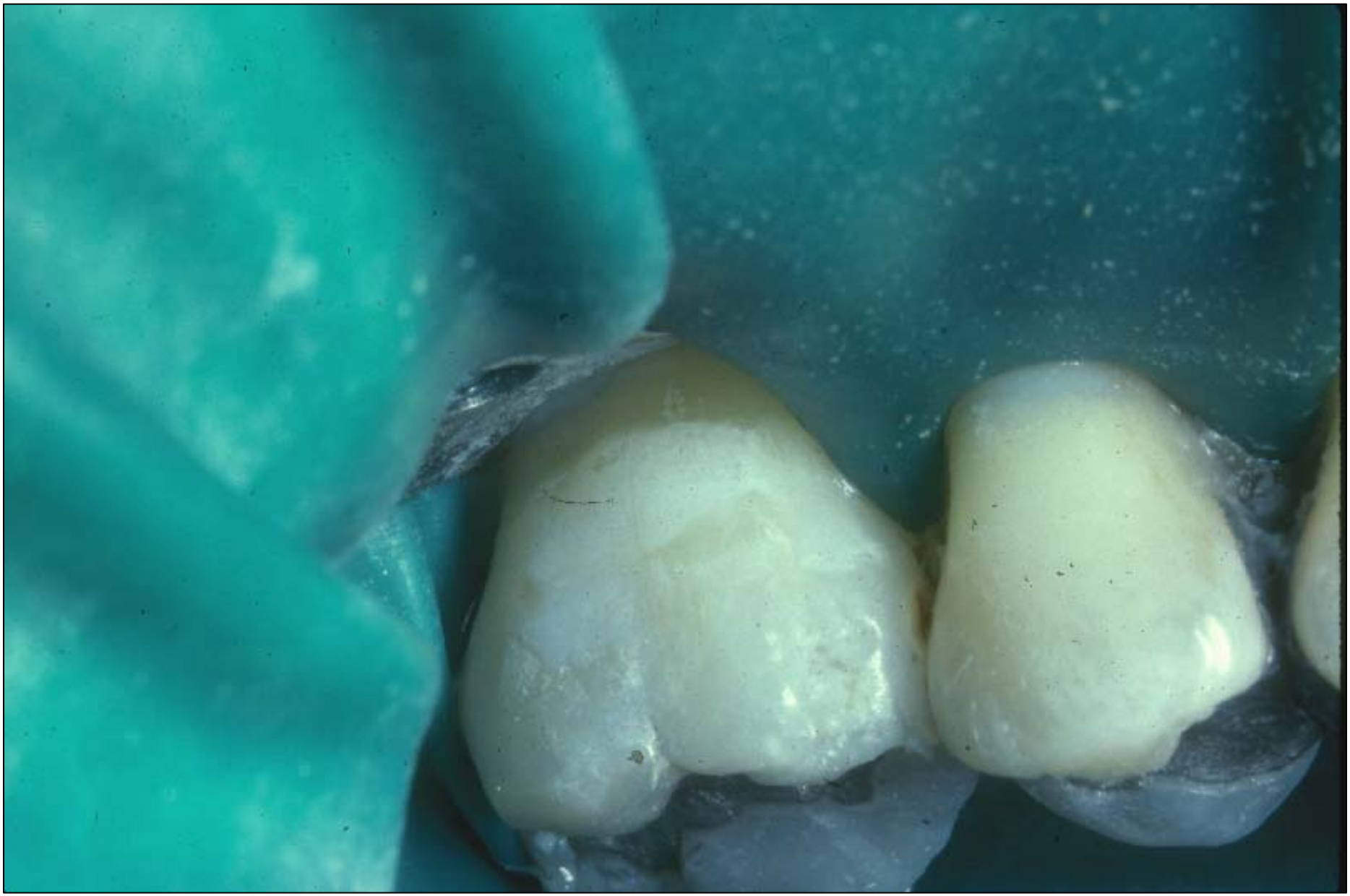
- Selective Demineralization
- Increases surface area
- Increases life of composite
- Decreases marginal staining
- Decreases secondary caries
- Decreases post-operative sensitivity
- Bond strengths » 22 - 30 MPa
- Creates high surface-energy
  - Permits efficient wetting by hydrophobic resin
  - Tag formation in microporosities

Dentin

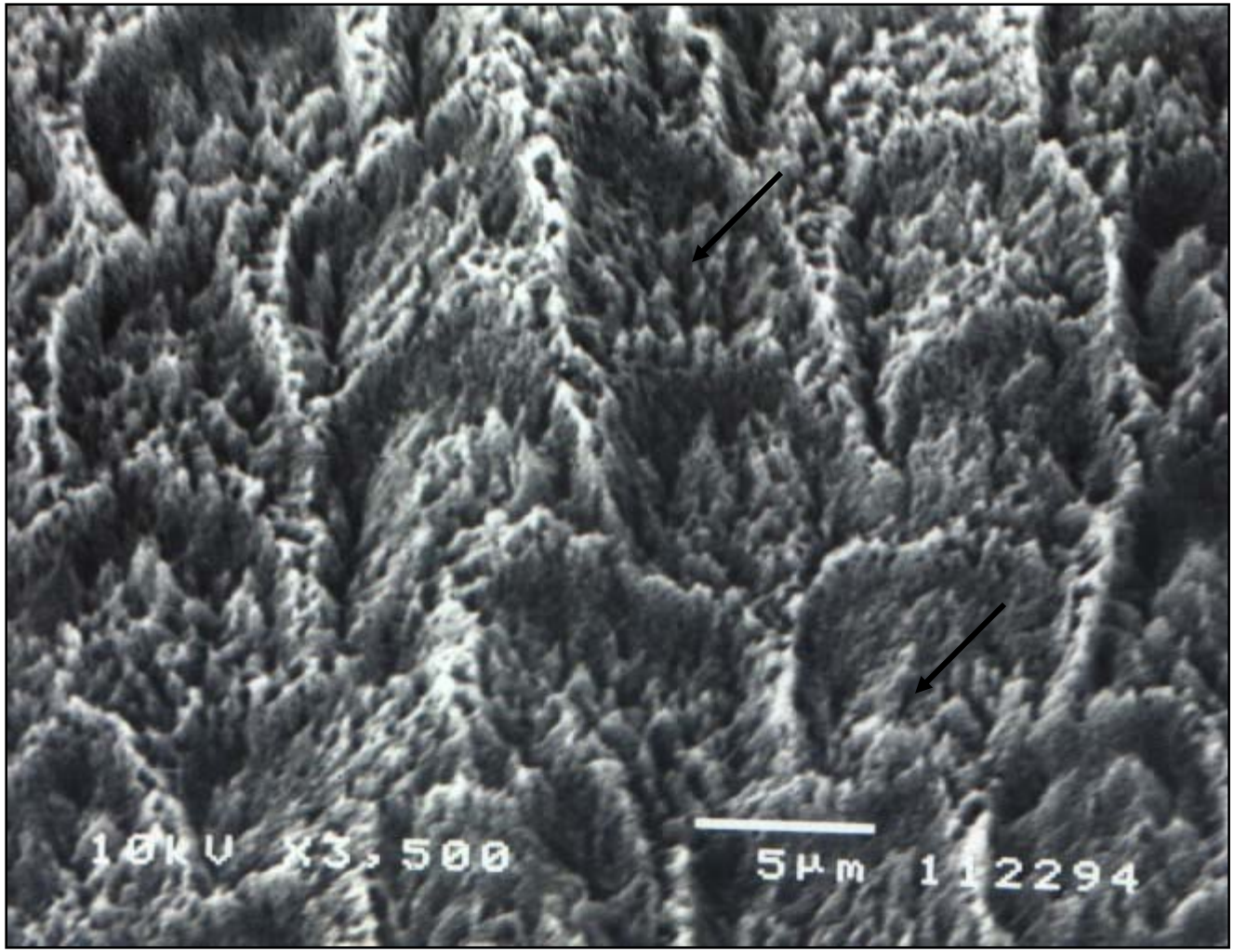
- Demineralizes dentin surface
- Opens dentinal tubules
- Exposes collagen
- Conditions dentin for better wetting of the primer

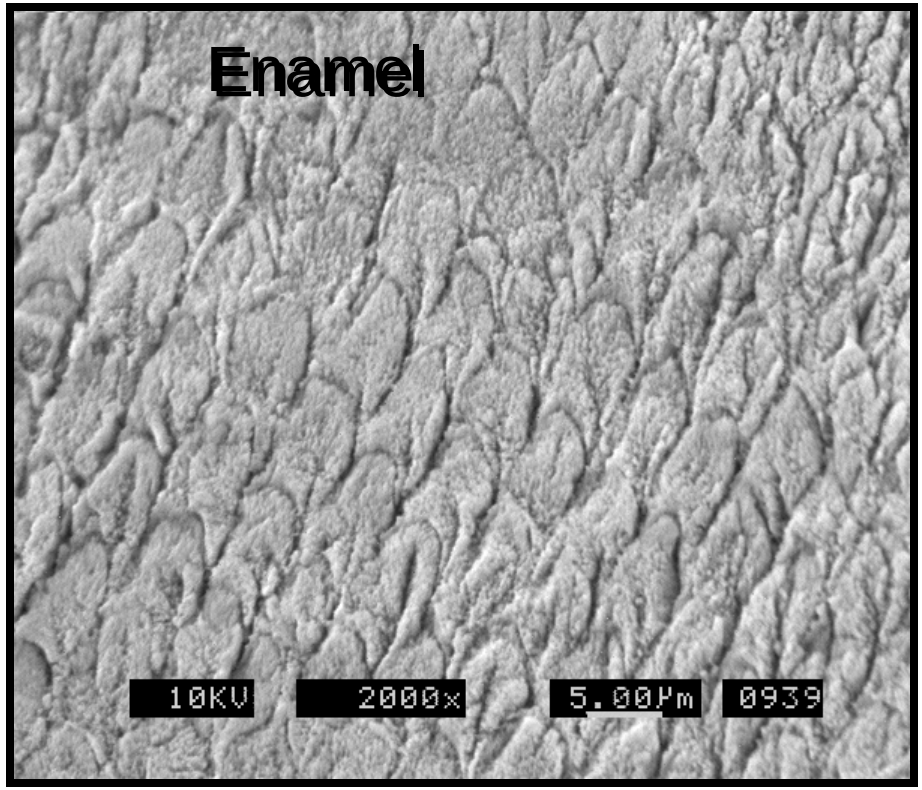
Depth of demineralization depends on:

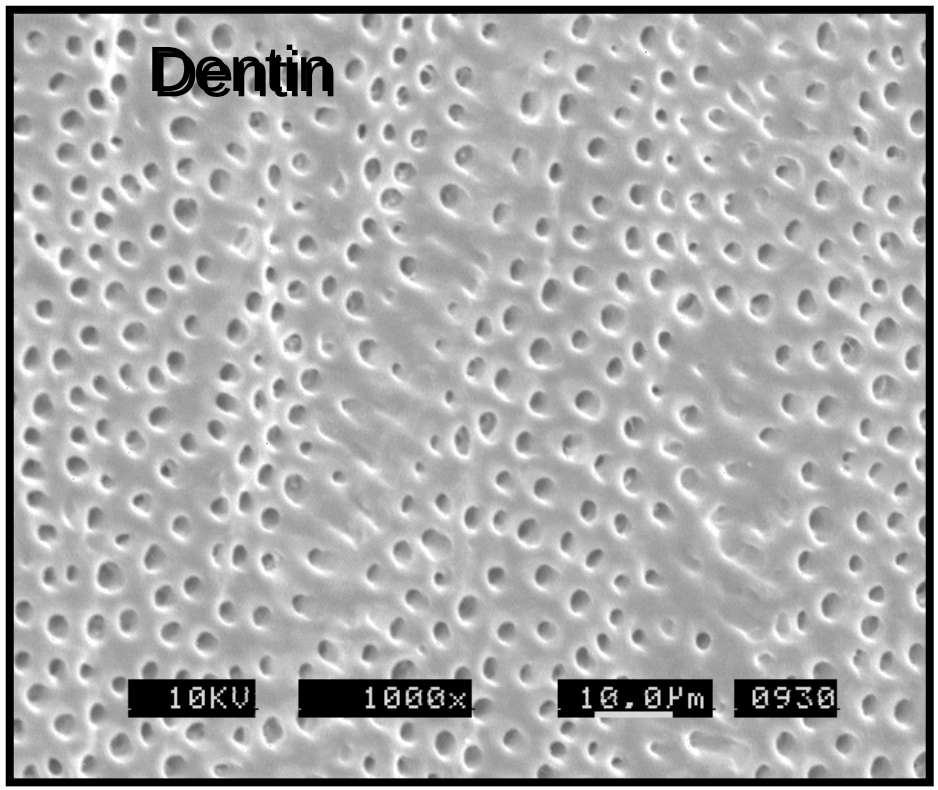
- Kind of acid
- Application time
- Acid concentration and pH
- Other components
- Moisture prevents the collapse of collagen.



Etched Enamel



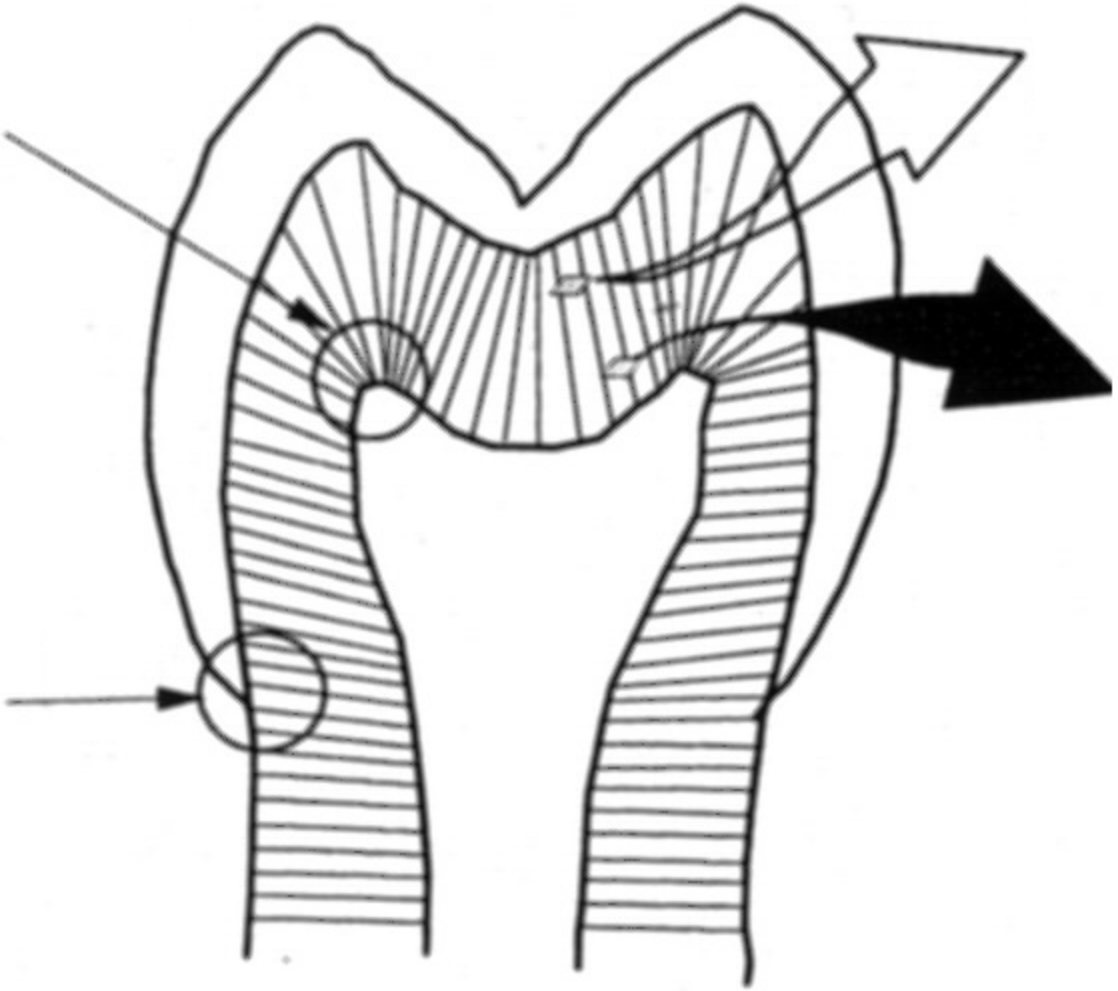

Enamel
10KV
2000x
5.00µm
0939


Dentin
10KV
1000x
10.0µm
0930

Dentin Tubule Volume

Most dense tubules

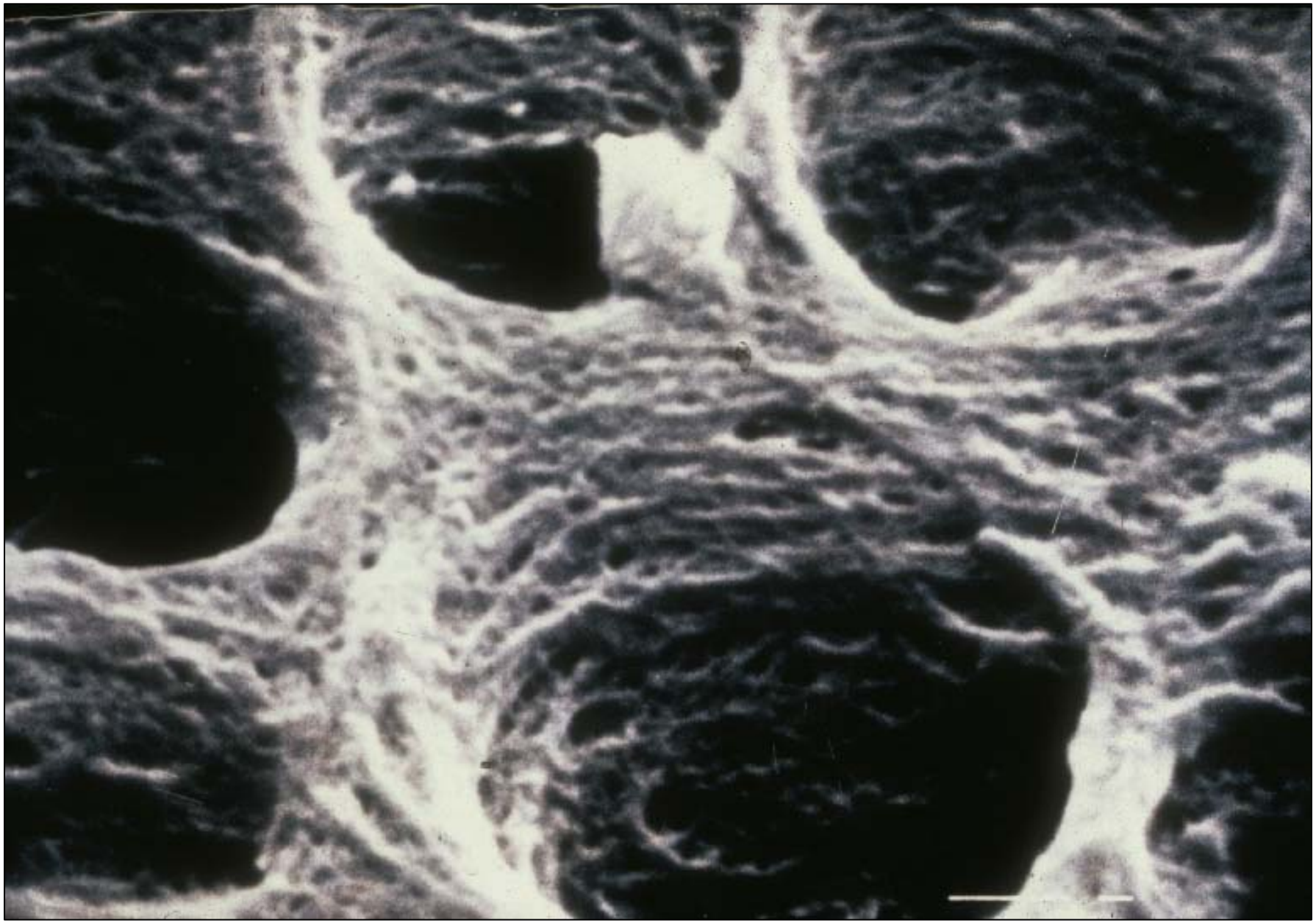
Least dense tubules

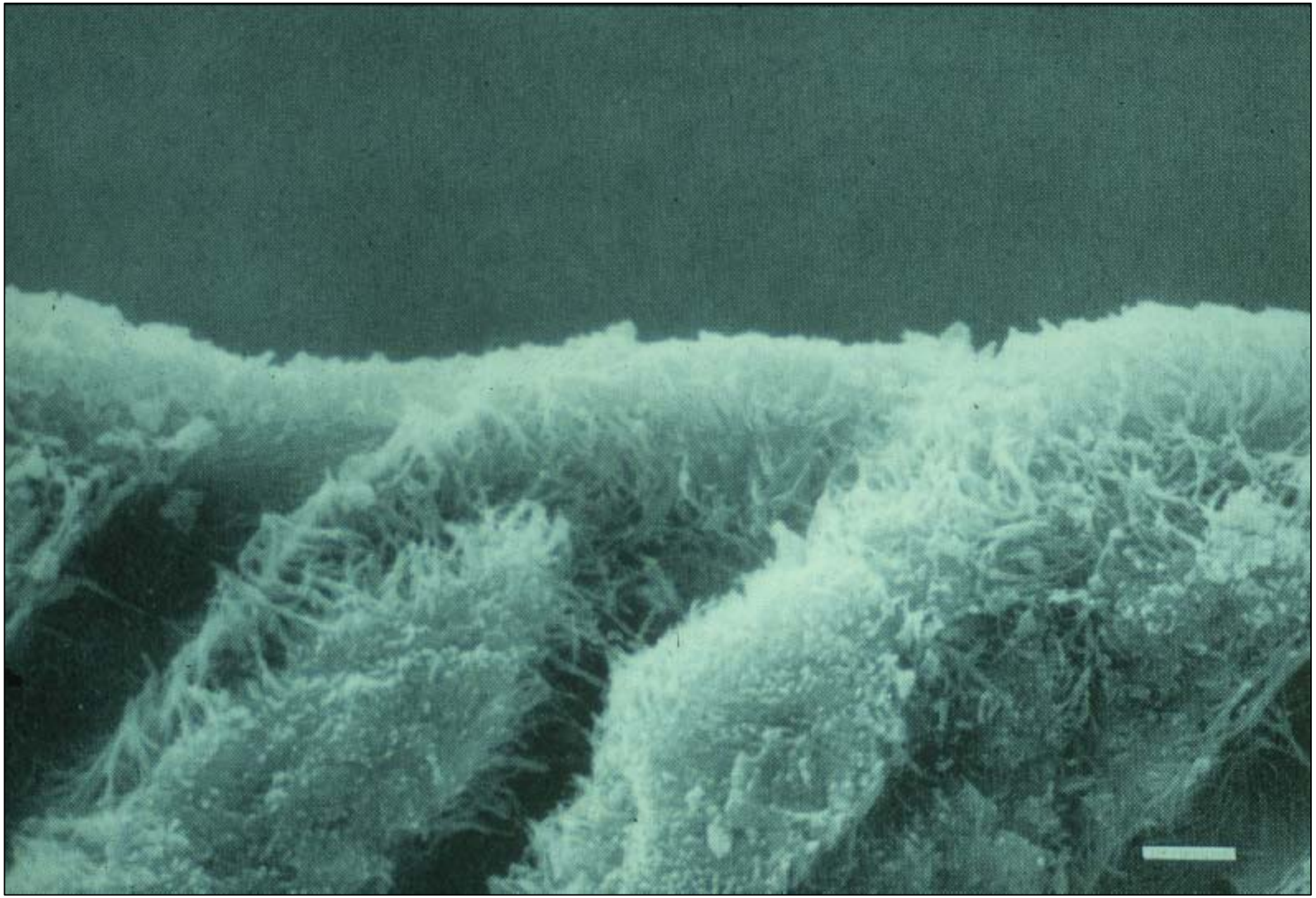


Outer third

Inner third

Demineralized Dentin





Primers

- Function:
  - As *surface-active* compounds which may promote adhesion through functional groups
  - Provide hydrophilic monomers
  - Initiator for monomeric polymerization
    - Promote interfacial polymerization
  - Permit improved wetting for hydrophobic resin

Primer Composition

Bifunctional monomers

- Hydrophilic -COOH groups have an affinity for exposed collagen fibrils
- hydrophobic groups capable of bonding to methacrylate groups of the adhesive resin

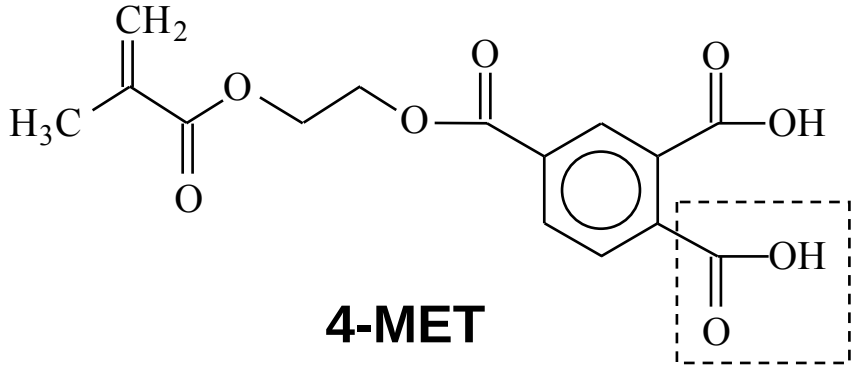
Photoinitiators

- Camphorquinone, phosphineoxides, tertiary amine

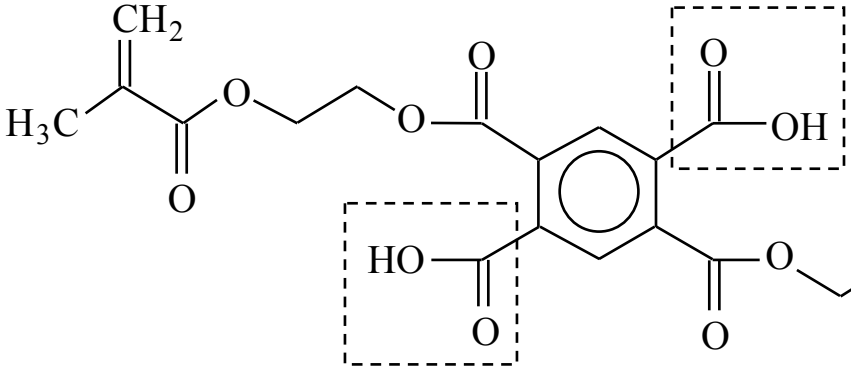
Solvents

- Acetone, ethanol or H_2O

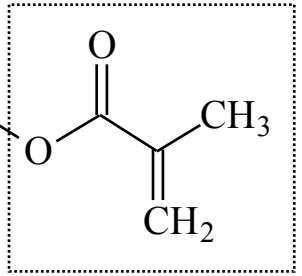
Adhesive Agents



4-MET



Carboxylic acid group



Methacrylate group

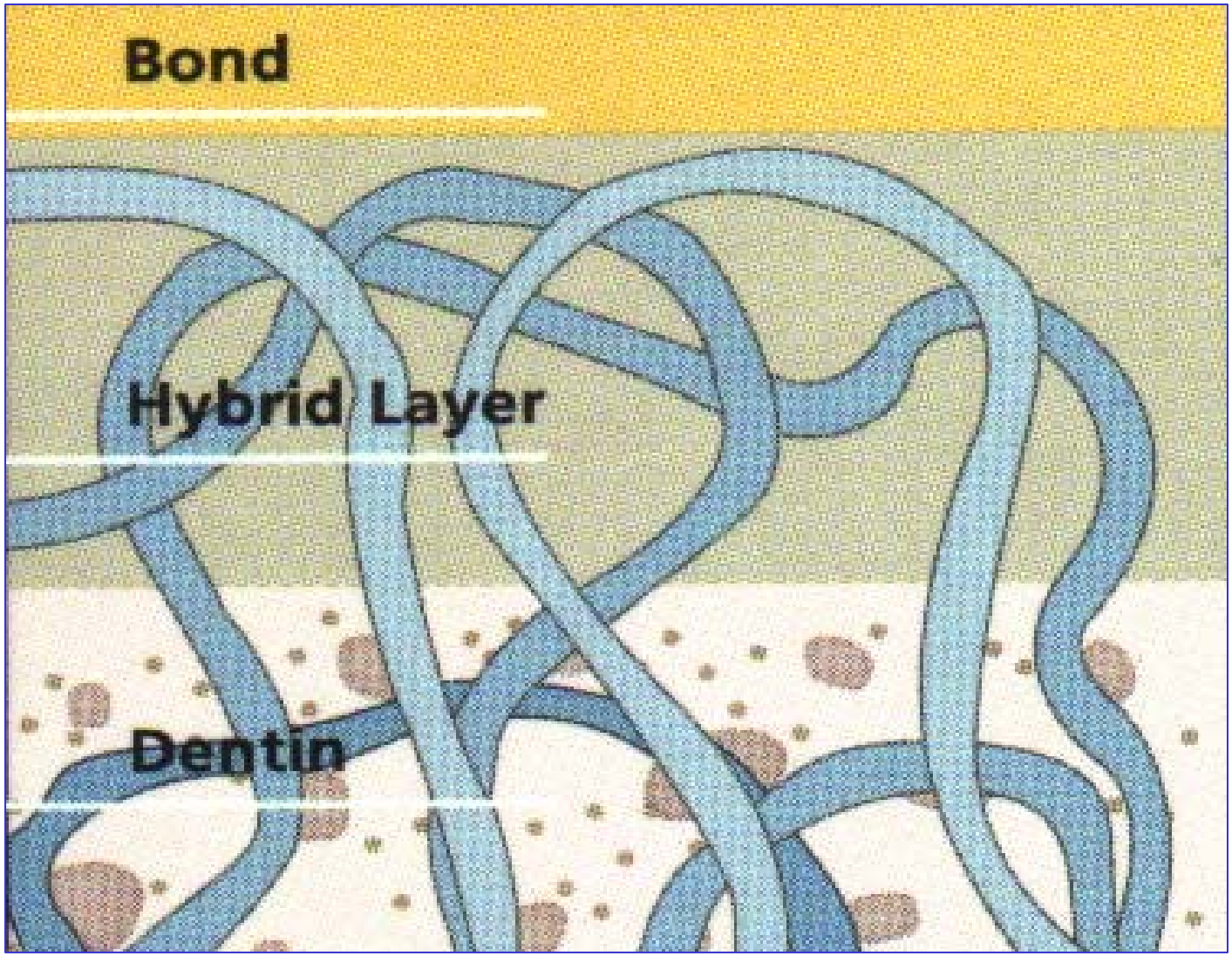
PMDM

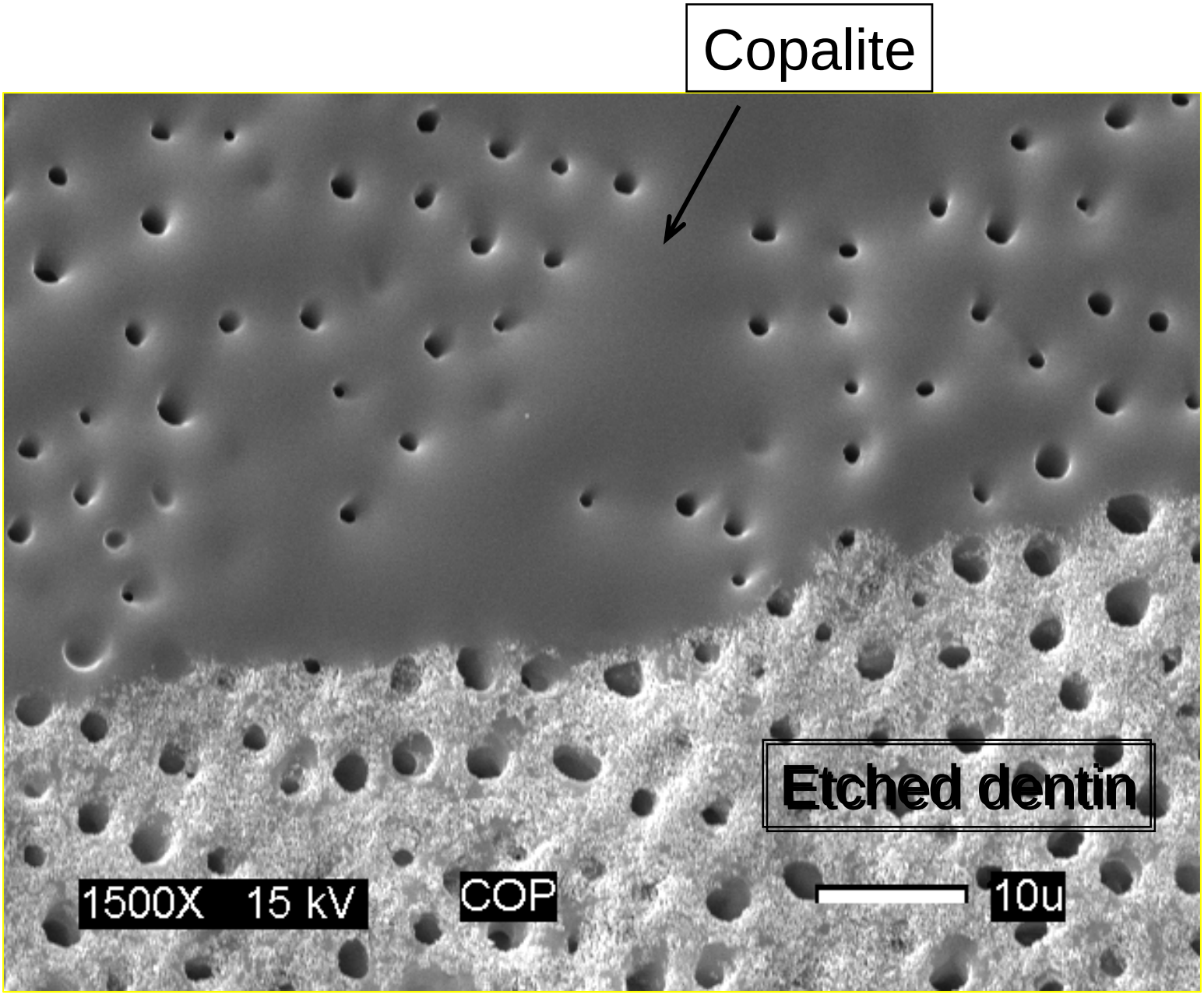
What is Hybridization?

- Formation of a hybrid layer following demineralization with an acidic conditioner.

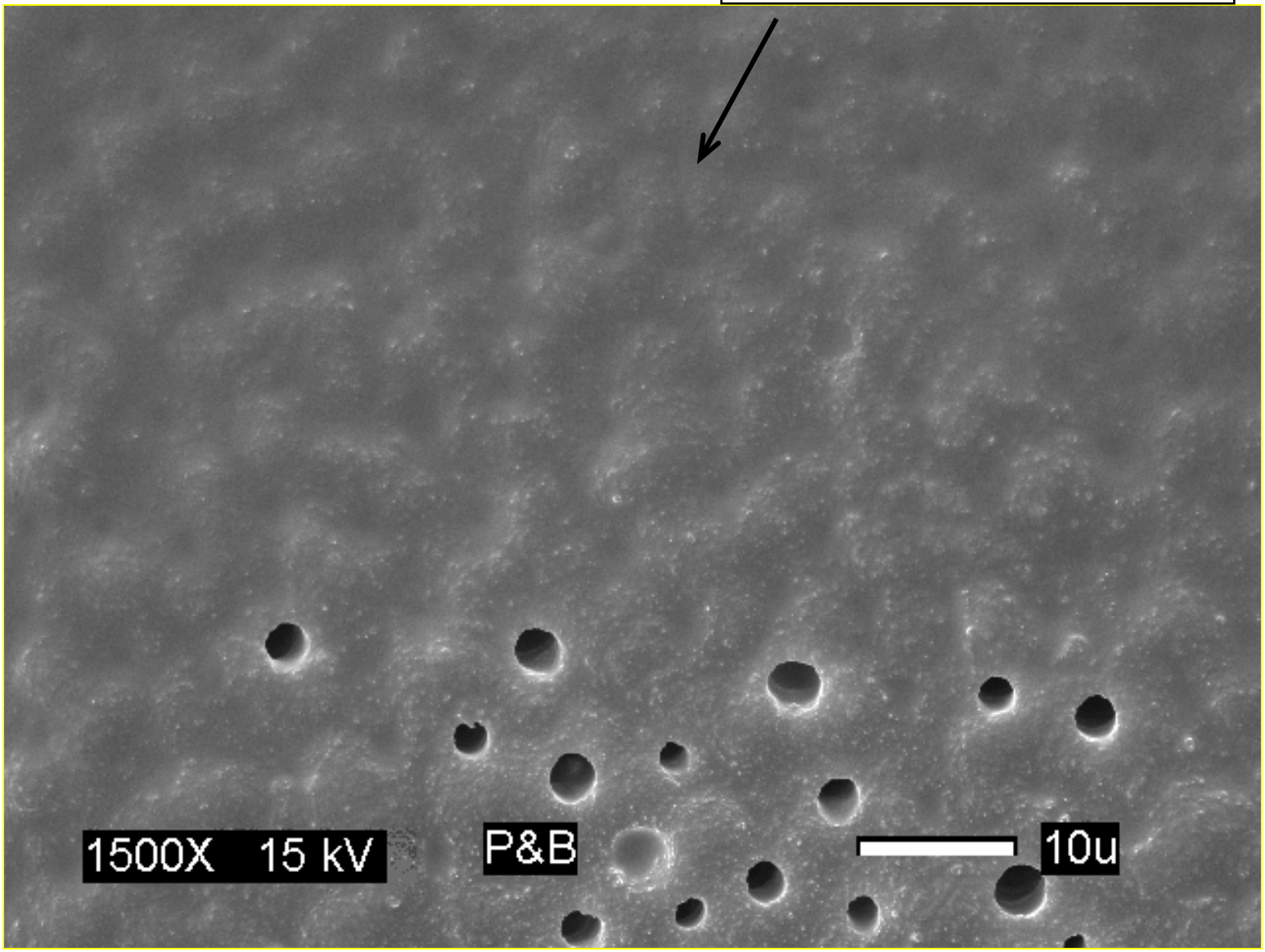
What is the Hybrid Layer?

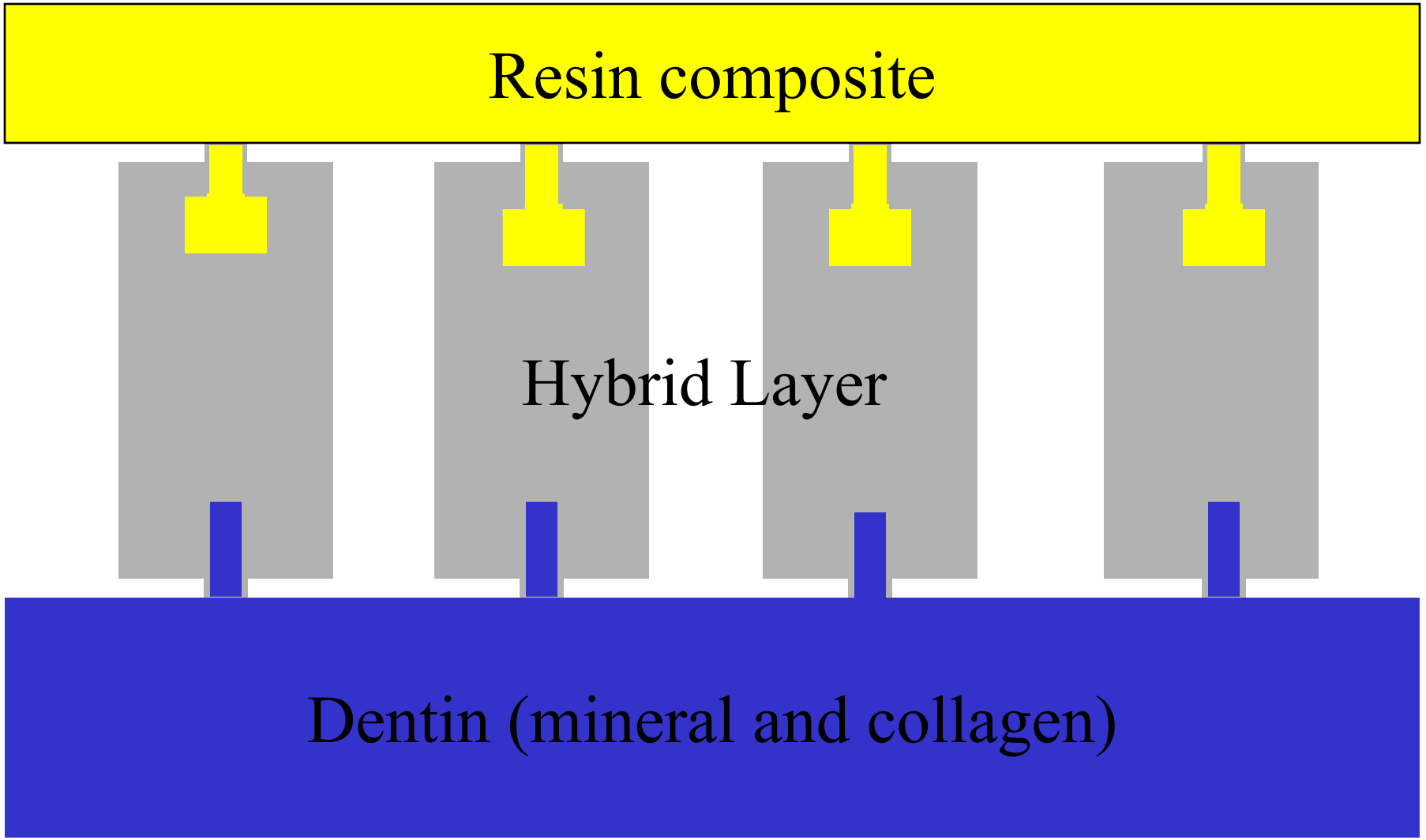
- A zone
- Resin micromechanical interlocking with dentinal collagen


Bond
Hybrid Layer
Dentin

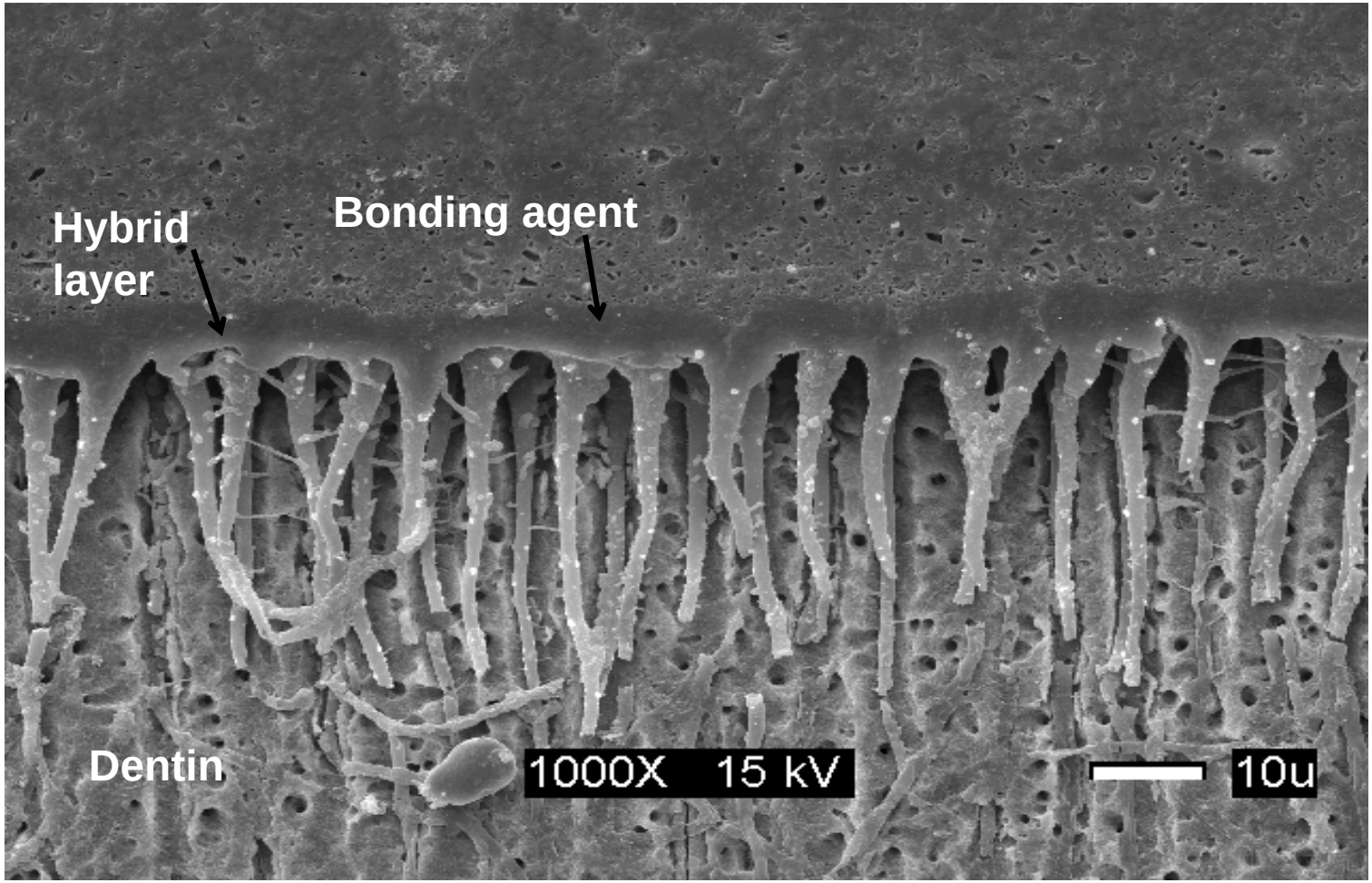

Copalite
Etched dentin
1500X 15 kV
COP
10u

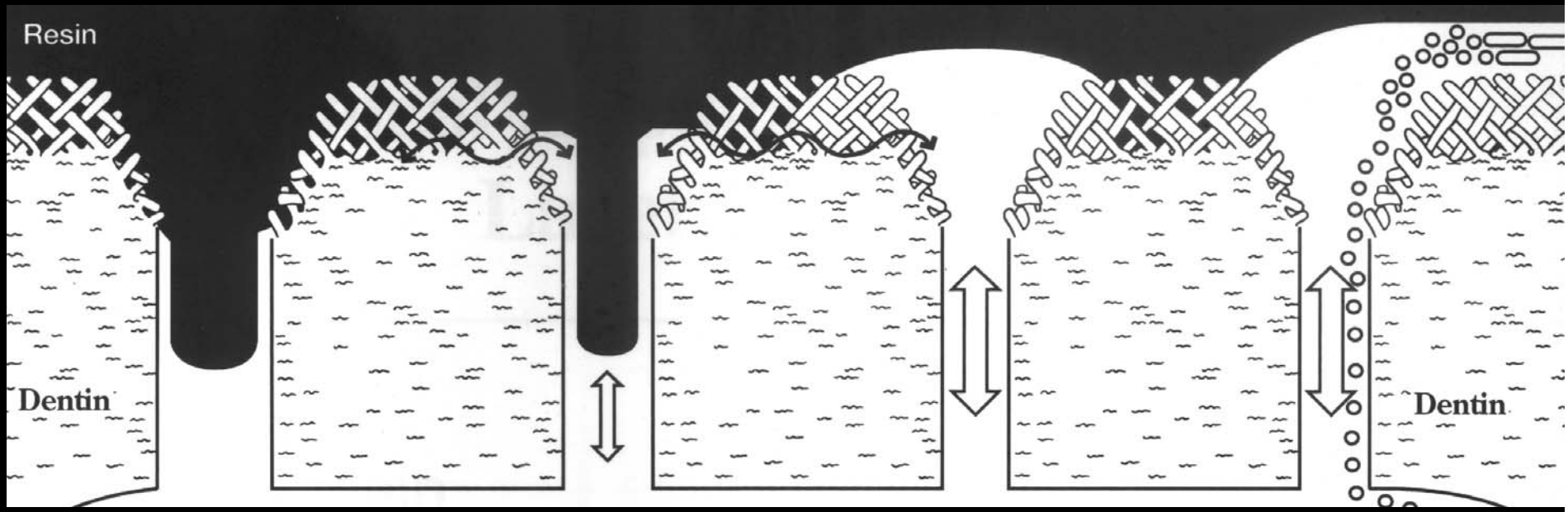
Prime & Bond 2.1




Resin composite
Hybrid Layer
Dentin (mineral and collagen)

Hybrid Layer




Resin
Dentin
Dentin

What bond strength is required?

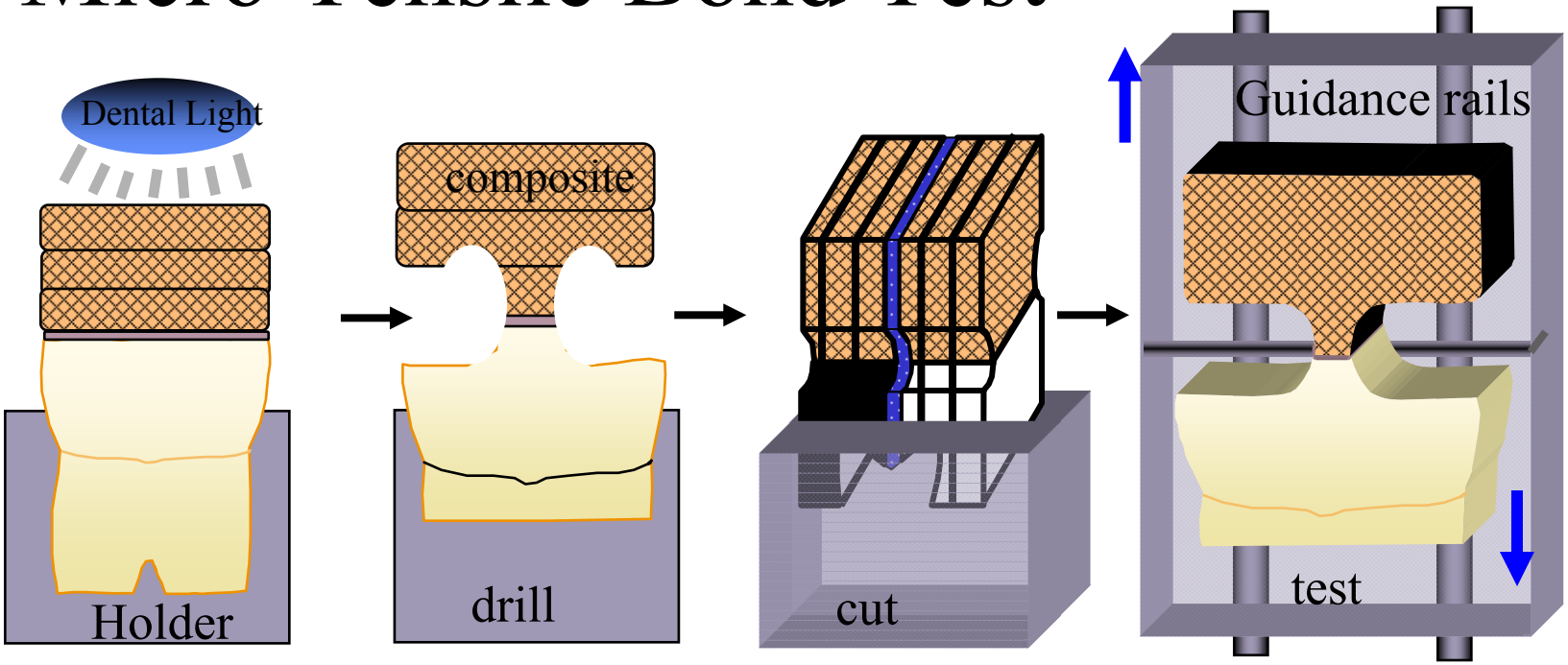
- Adequate bond strength is required to overcome the stresses associated with polymerization shrinkage of the resin composite restorative material.
- 20 MPa*

*DAVIDSON CL, de GEE AJ, FEILZER A. (1984) J Dent Res 63(12):1396-1399

Types of Tests

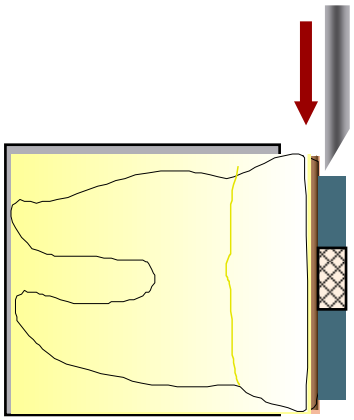
- Adhesion or Bond Strength
  - Tensile, Microtensile
  - Shear, Microshear
- Thermal cycling
- Microleakage
- Bond Observations
  - SEM
  - TEM
  - Mode of Failure

Micro Tensile Bond Test

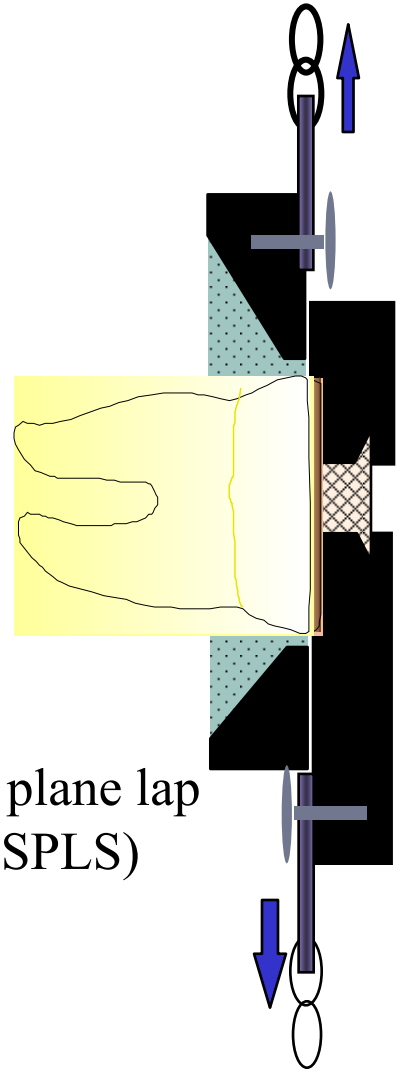


1. Cut the tooth/composite assembly into a cube-shaped block.
2. Cut two grooves on the long sides of the cube parallel to and centered on the resin-dentin interface, and machine the resin-dentin block into an hour-glass shape.
3. Cut the resin-dentin block into several slices and measure bond interface areas.
4. Fix the specimen to a specially designed holder with Loctite.®
5. Mount in a universal testing machine and stress at a crosshead speed of 1 mm/min.

Shear Bond Tests



Metal
Iris

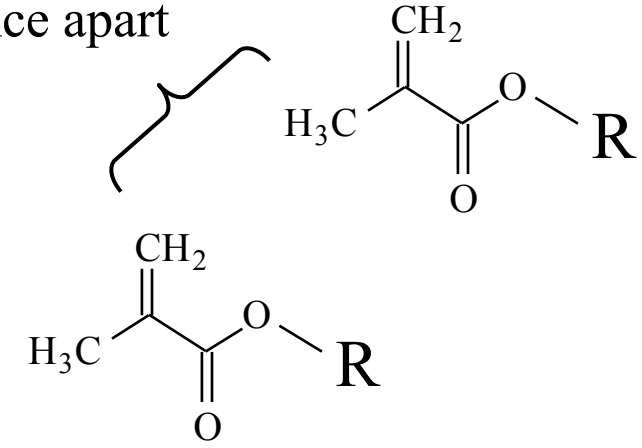


Single plane lap
shear (SPLS)

Milos and Dickens, IADR 1997

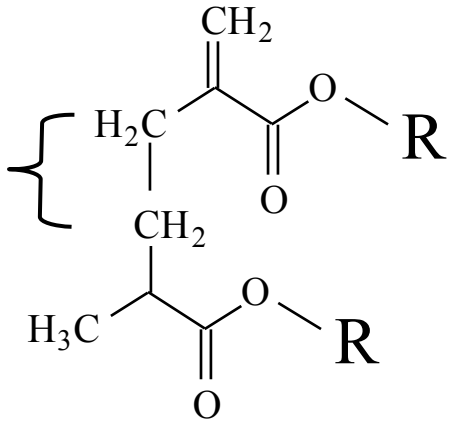
Polymerization Shrinkage

Van der Waal's
distance apart



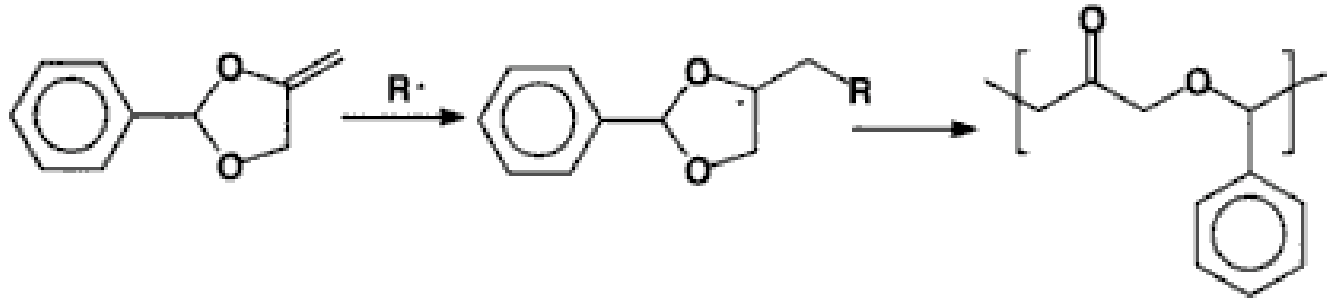
pre-cured monomer

covalent bond
distance apart



polymer

Free Radical Ring Opening Polymerization to Minimize Polymerization Shrinkage



Polymers for Dental and Orthopedic Applications, Shalaby W. Shalaby, Ulrich Salz, 2006

- Based in ring size, number of rings, degree of conversion, etc., reduced shrinkage and even net volume increase possible.
- Mechanical properties tend to suffer
  - compressive modulus
  - wear resistance

Acknowledgements

Gary E. Schumacher, DDS, MS

Associate Director and Chief, Clinical Program
American Dental Association Foundation
Paffenbarger Research Center

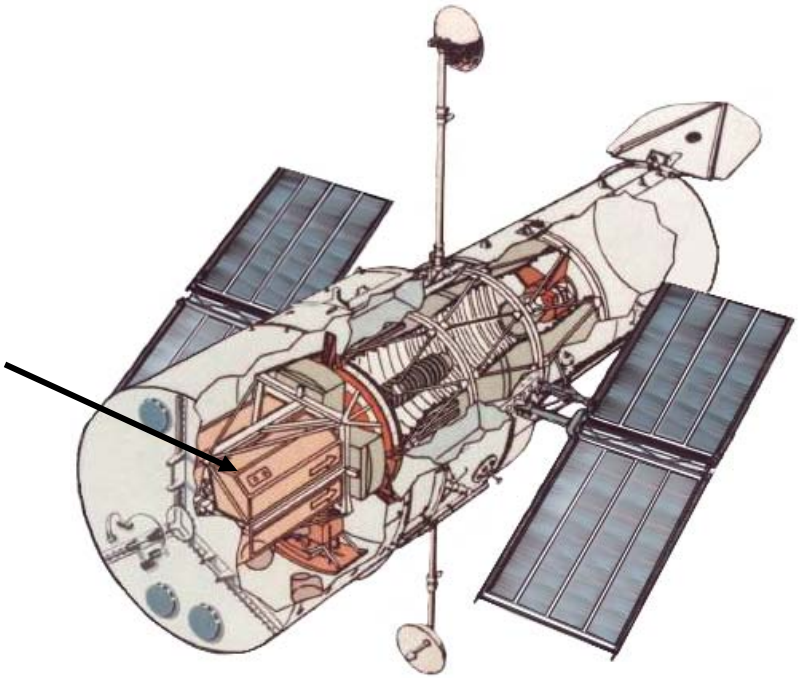
National Institute of Standards and Technology
Gaithersburg, MD 20899-8546

COS Interpoint DC-DC Power Converter Failure in Vacuum

B. Reed
Materials Engineering Branch
March 4, 2008

Cosmic Origins Spectrograph (COS)

- HST axial scientific instrument the size and shape of a phone booth.
- Designed to study the large-scale structure of the universe and how galaxies, stars and planets formed and evolved.
- Will replace COSTAR which is no longer needed.
- The prime contractor for the design and assembly is Ball Aerospace and Technologies Corp., Boulder, CO.



QuickTime™ and a
TIFF (LZW) decompressor
are needed to see this picture.

Background

- COS successfully completed instrument level ambient functional testing in 2003.
- Subsequently, moved into a thermal vac chamber at Ball where during the initial instrument checkout an anomaly was noted.
- The Calibration Lamp Converter 30V output was reading only 14V.
- The low voltage power supply board #2 was removed from the instrument.
- Following trouble shooting at Ball the Interpoint dual power converter, MFL2815D, was removed and shipped to GSFC.

COS LVPS2

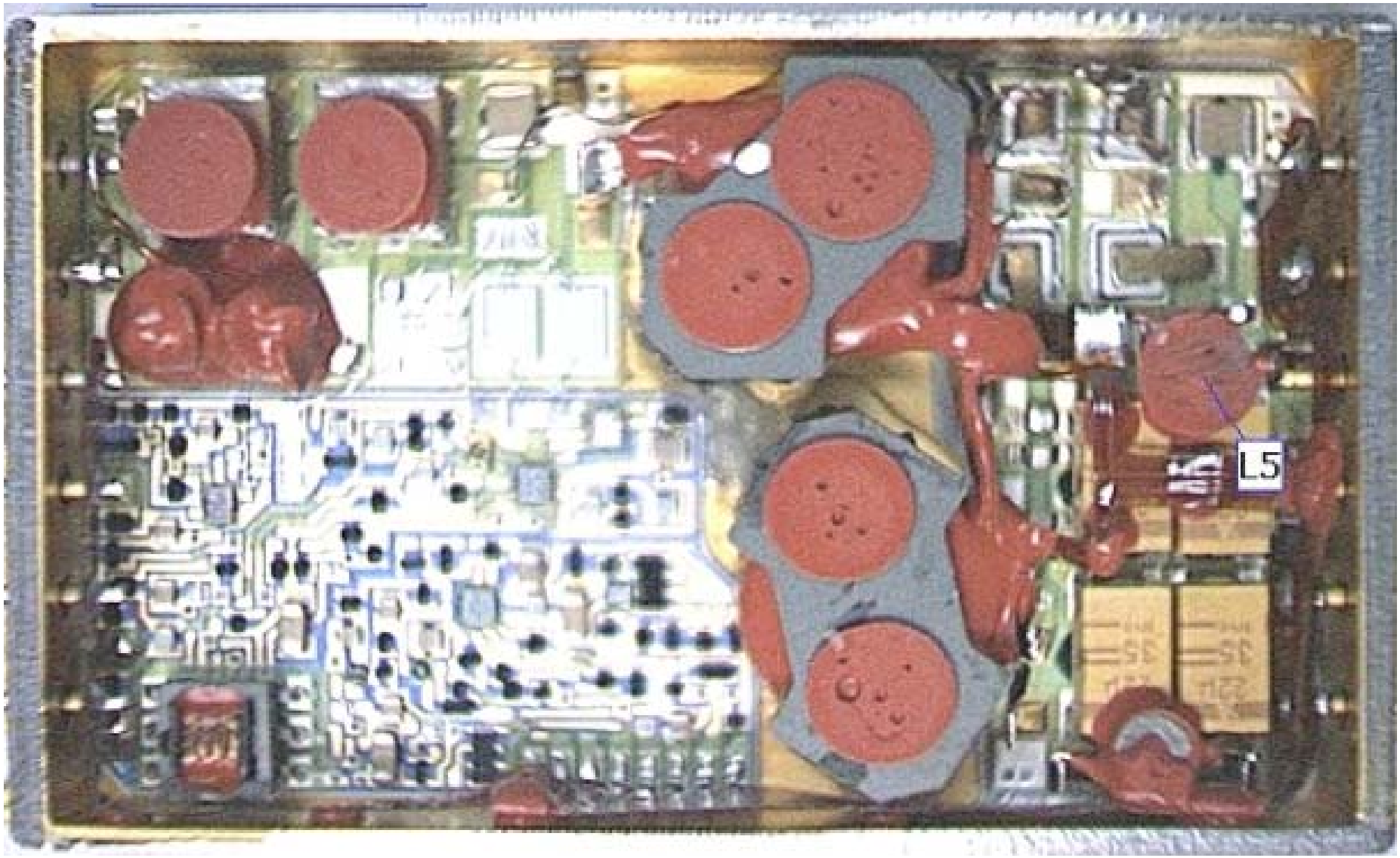
photo dated 2001

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

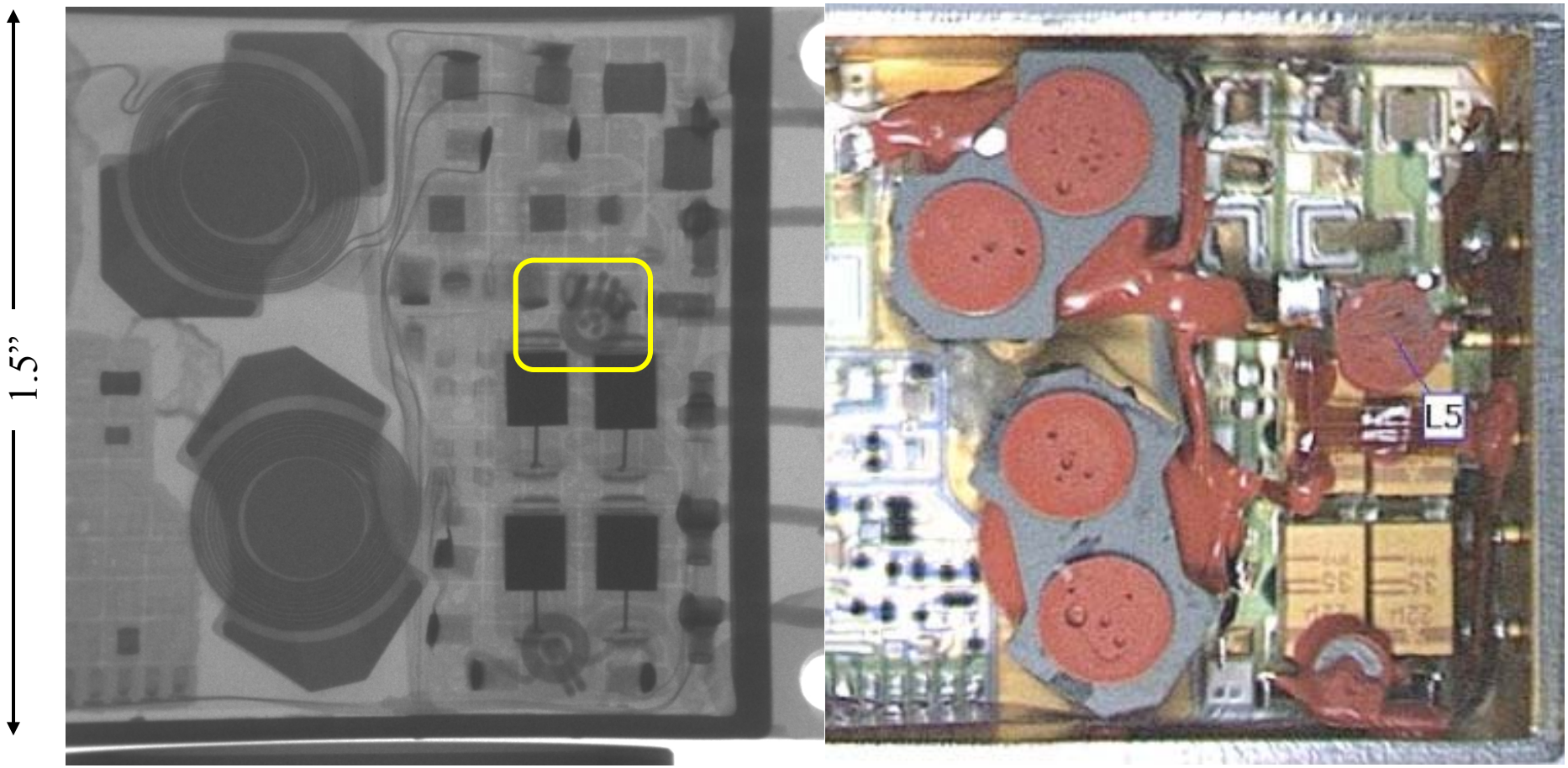
Materials Branch Investigation

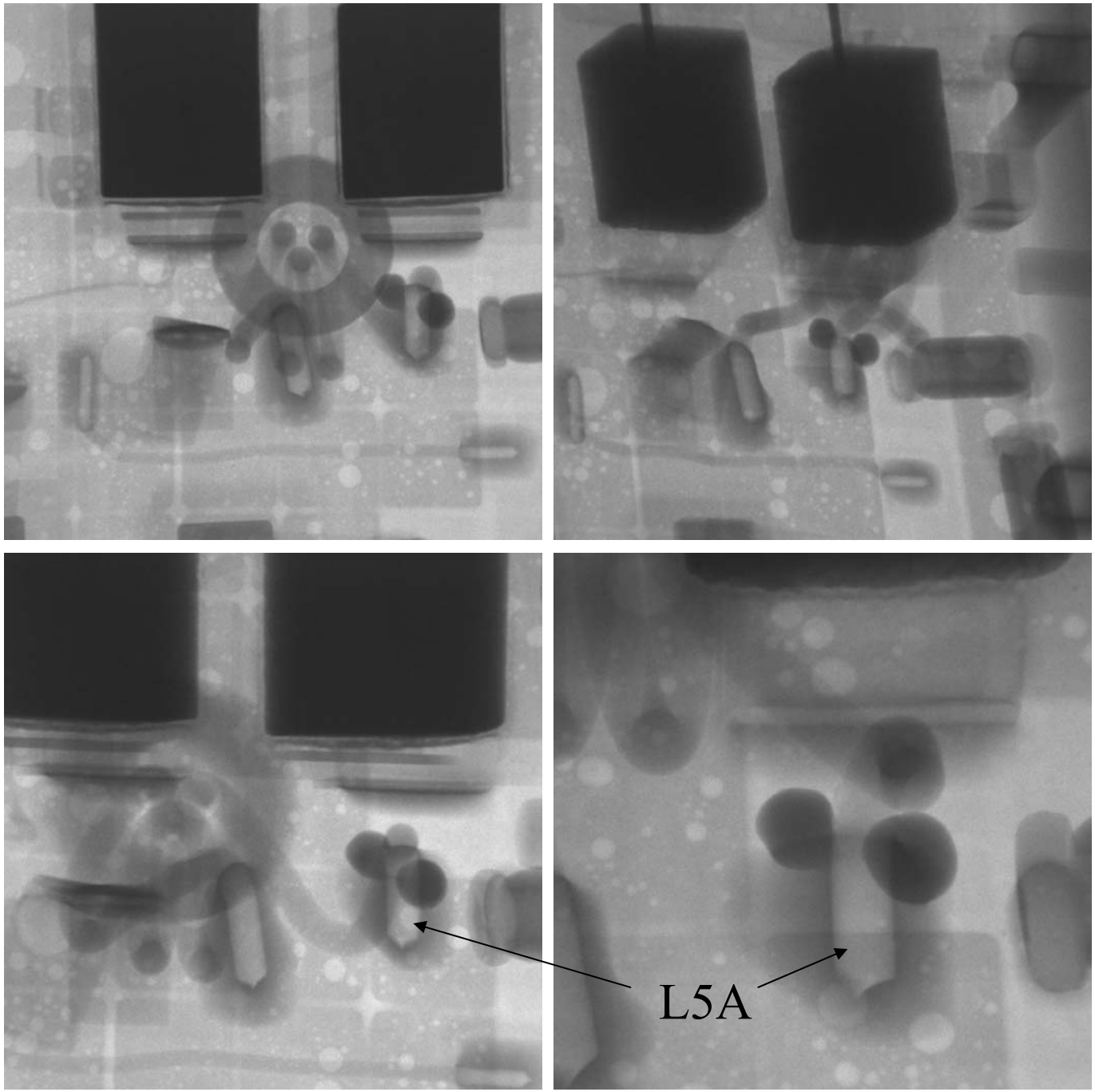
- A halo was observed around the ‘foot’ of the L5A inductor lead.
- The converter was then installed in a vacuum bell jar, mounted to the real time radiography manipulation stage, and configured with continuous impedance monitoring of the -15V output.
- This allowed realtime telemetry of the circuit resistance while observing any internal movement as a function of external pressure.
- BTW, Interpoint power converters are hermetically sealed with ambient pressure inside.

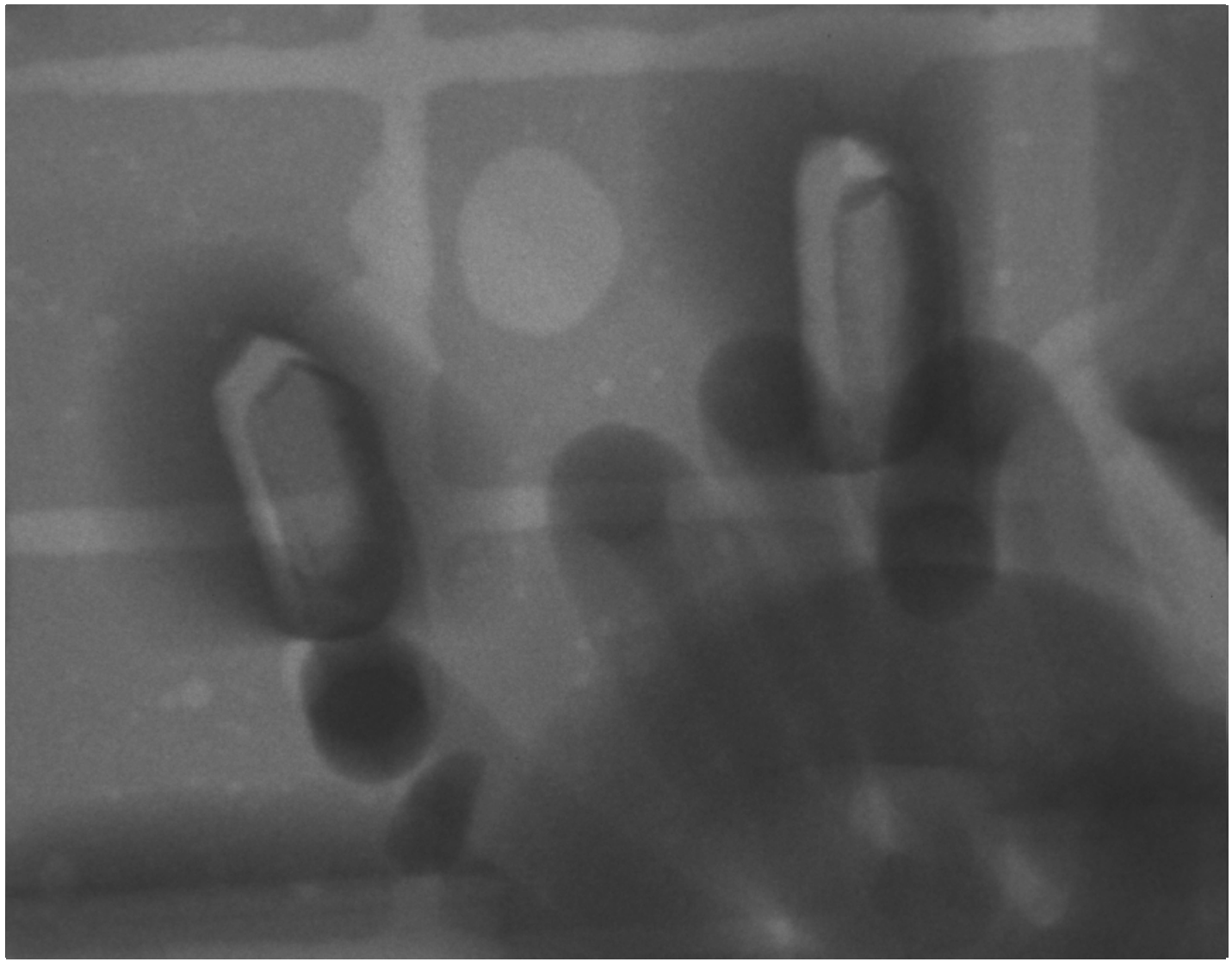
MFL2815D with Lid Removed



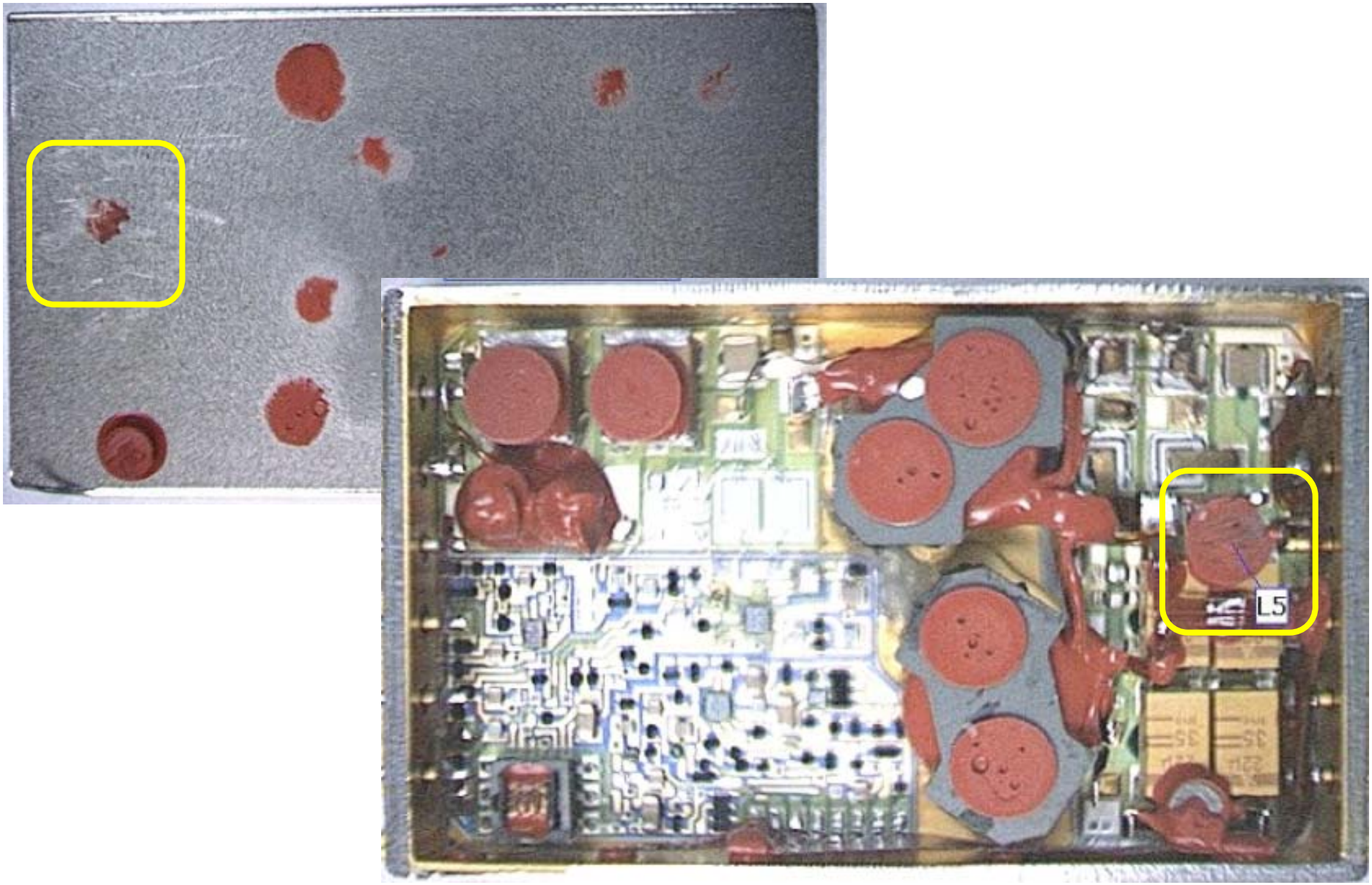
Internals of Interpoint 2815D, LDC 9846




L5A



2815D with Lid Removed



Root Cause

- Detrimental combination of:
  - L5 inductor bonded to lid
  - lid bowing outward upon vacuum acquisition
  - lack of adequate strain relief of L5 leads

Corrective Actions

- All dual power converters, on all PWAs, were radiographed to assess the inductor strain relief.
- Subsequently, Kovar plates were bonded to the lids to stiffen and thereby minimize deflection.

- For unrelated reasons, all Interpoint power converters were removed and replaced.

Acknowledgements

Bruno Munoz

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Rick Higgins
Emmanuel Cofie
Henning Leidecker
Justin Cassidy
Roger Chiei

HUBBLE SPACE TELESCOPE
Flight Systems and Servicing Project

HST SM4
Exterior Materials
Awareness Briefing

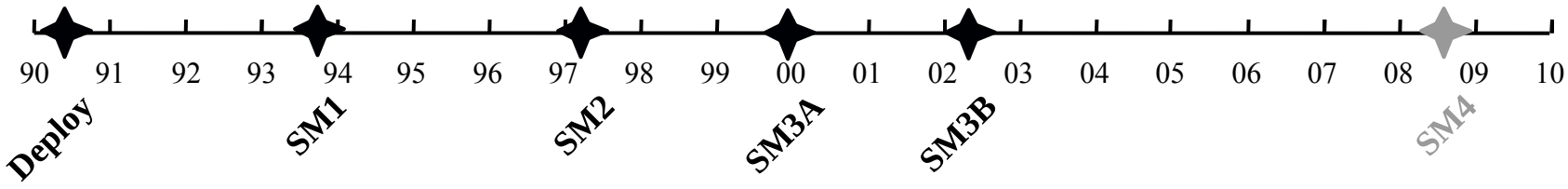
Benjamin Reed

NASA/GSFC

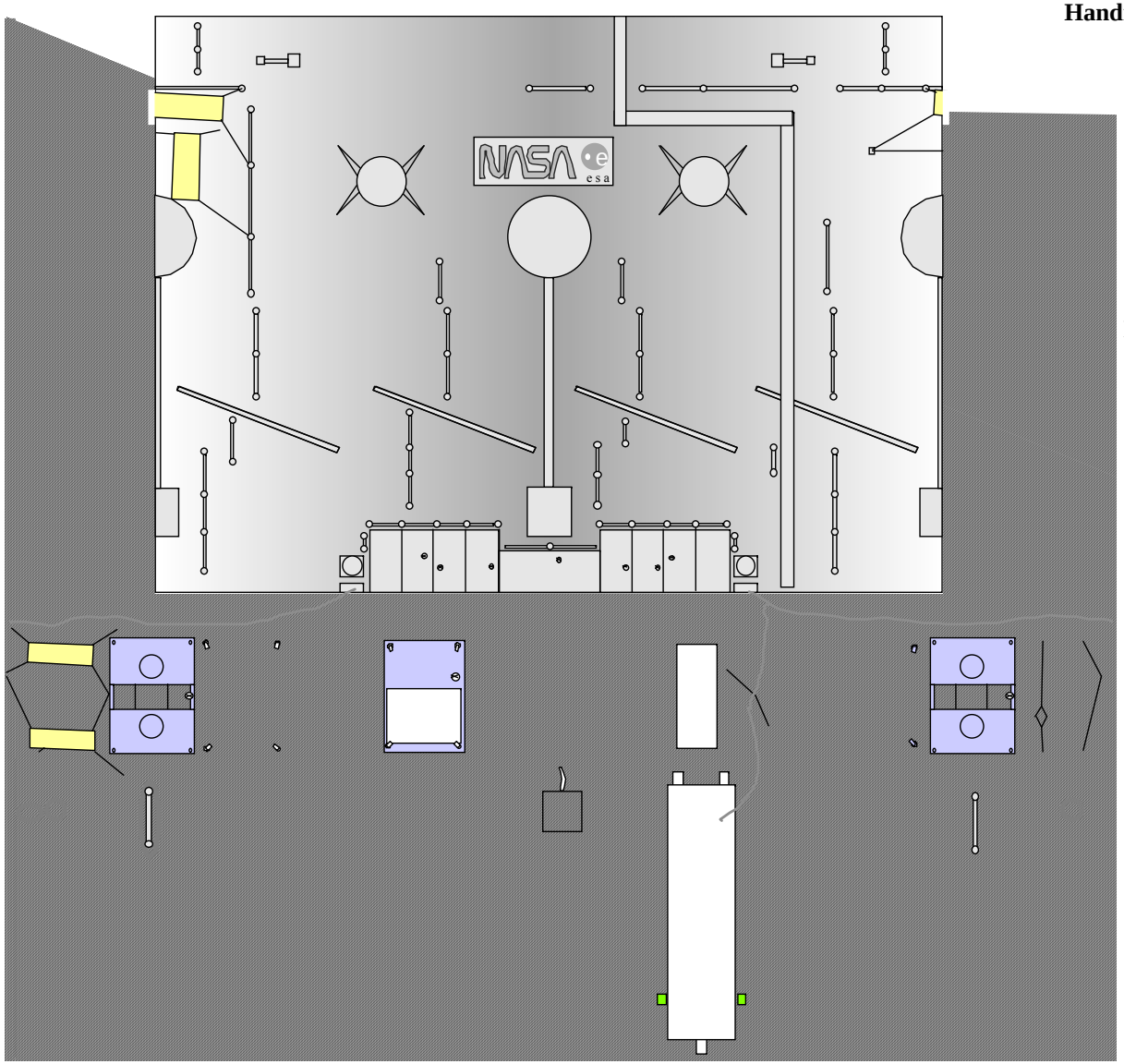
March 3, 2007

HST Servicing History

		total years	years between
• Deploy	Apr 1990		
• SM1	Dec 1993	3.6	3.6
• SM2	Feb 1997	6.8	3.2
• SM3A	Dec 1999	9.7	2.9
• SM3B	Feb 2002	11.9	2.2
• SM4	Sep 2008	18.4	6.5



Overview of HST Exterior



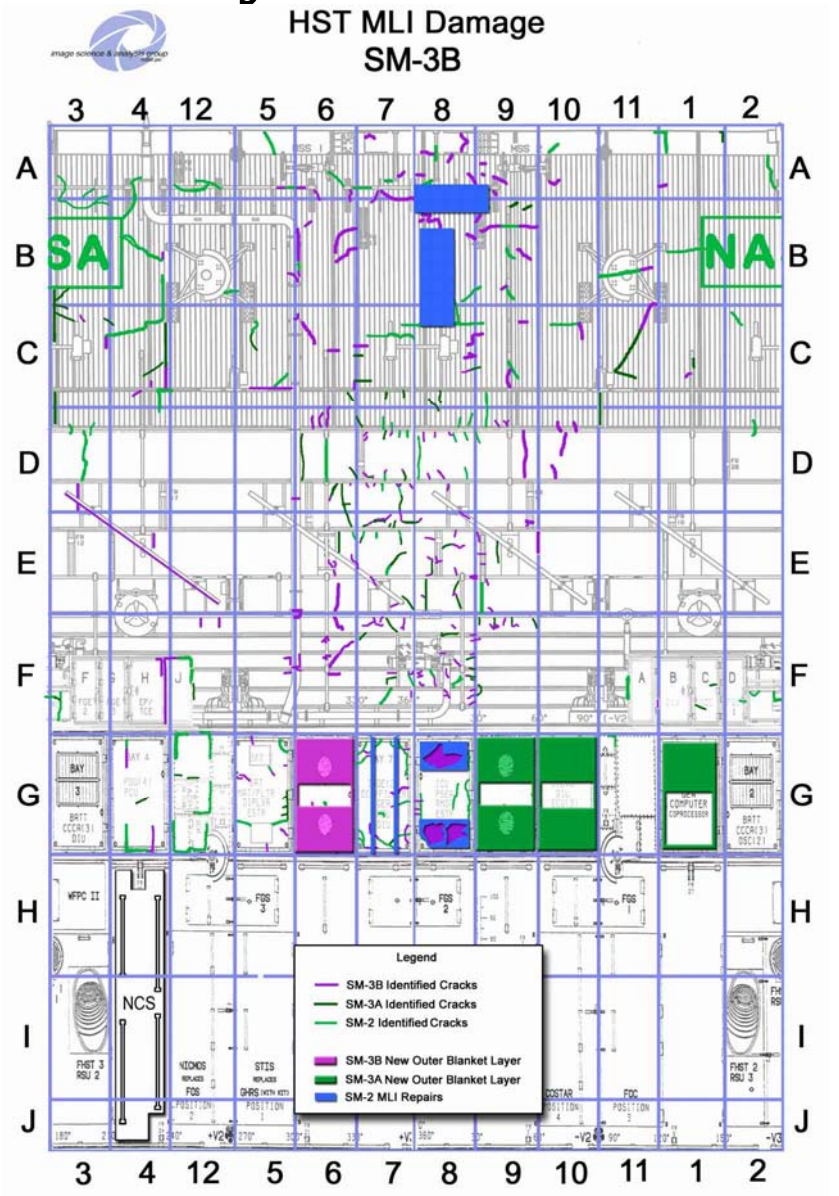
Handrail & scuff plate paint:
space
Silicone overcoat
Polyurethane binder with inorganic pigment
telescope

MLI construction:
space
5 mil Al Teflon
1/3 mil double Al Kapton embossed x 15 layers
1 mil Al Kapton
telescope

Equipment Bays:
combination of MLI, FOSR, NOBL

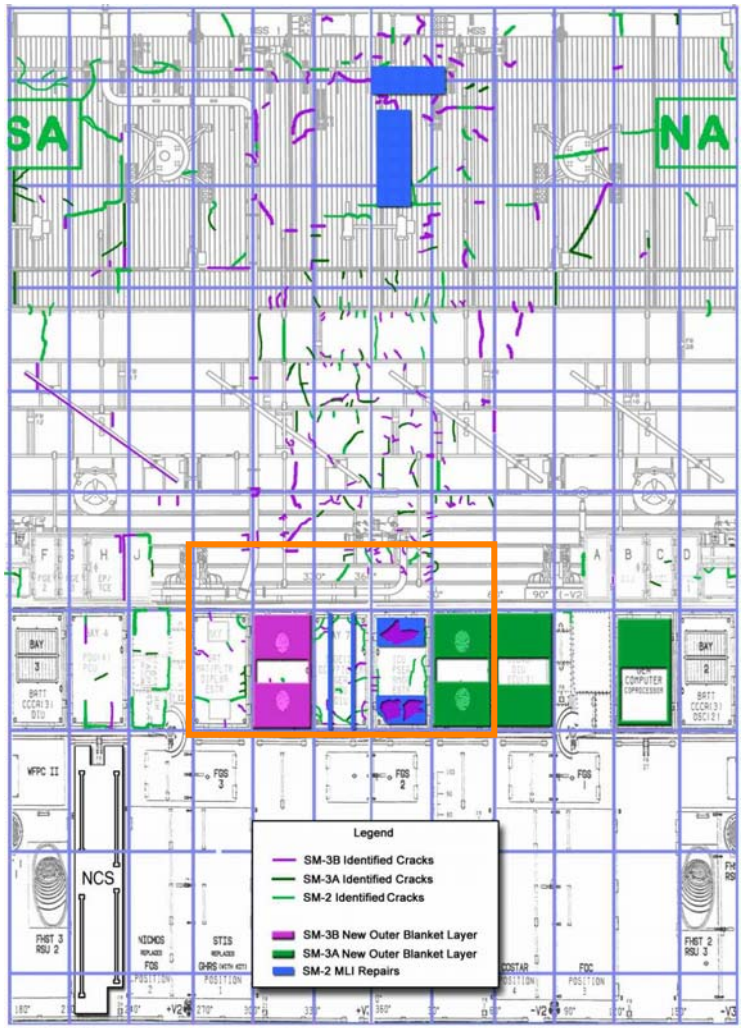
FOSR Tape:
space
5 mil Ag Teflon acrylic adhesive
telescope

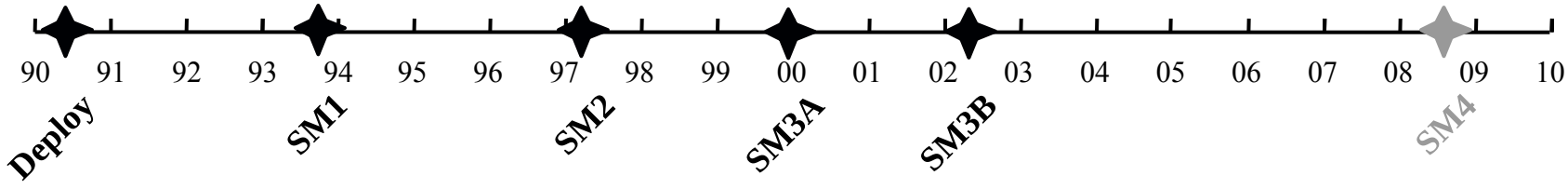
HST Damage Map as of Mar 2002
JSC ISAG Survey



Damage Map Summary

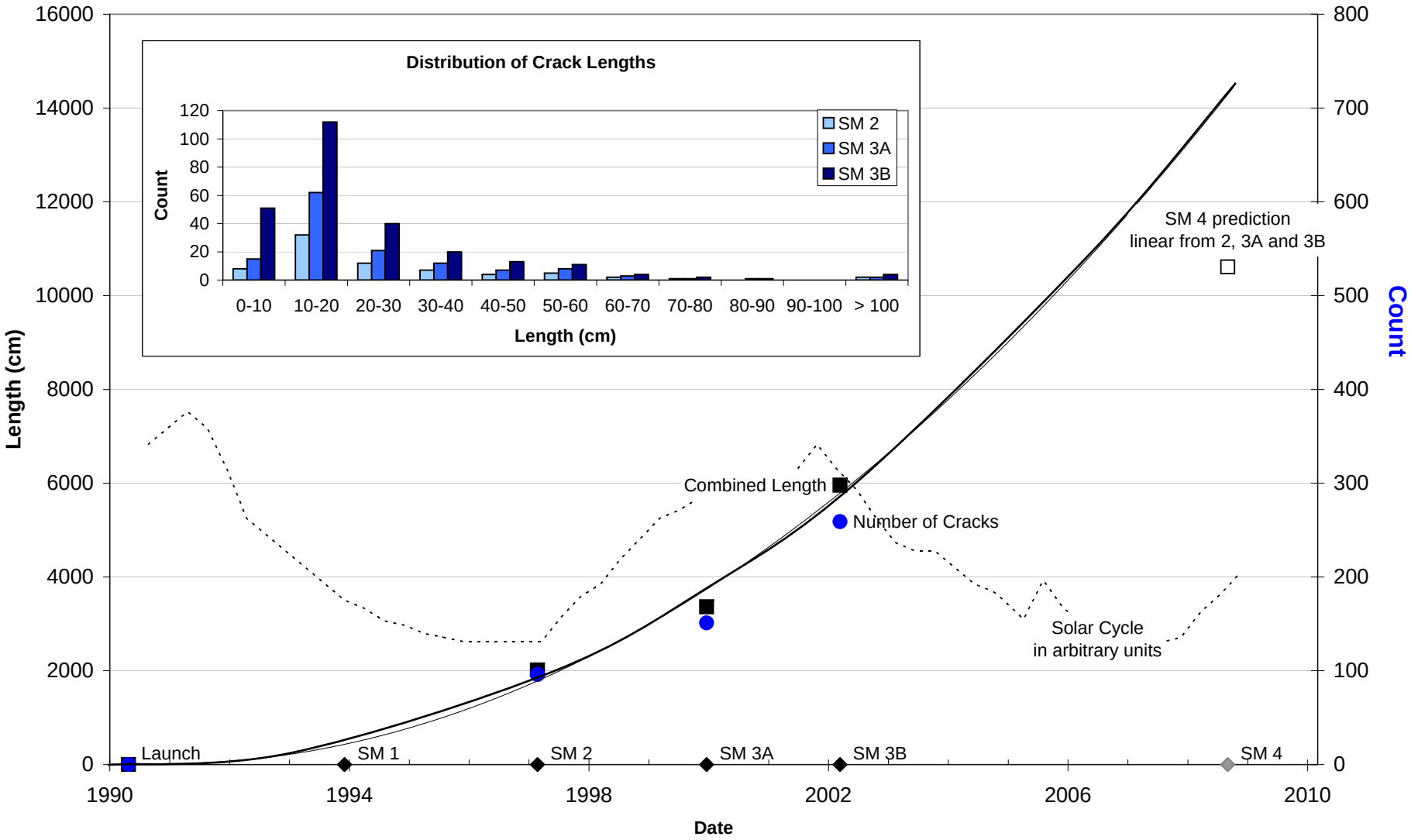
- SM2 96 cracks all 5 inches or longer (light green)
- SM3A 150 total cracks, 9 existing much longer (dark green)
- SM3B 260 total cracks, 38 existing grew longer (violet)
  - 75% of cracks are on +V3





SM4 MLI Crack Prediction

Hundreds of new cracks; average length 20 cm; a few much larger cracks



MLI Degradation Example
+V3 Light Shield and SM2 installed patches

SM1

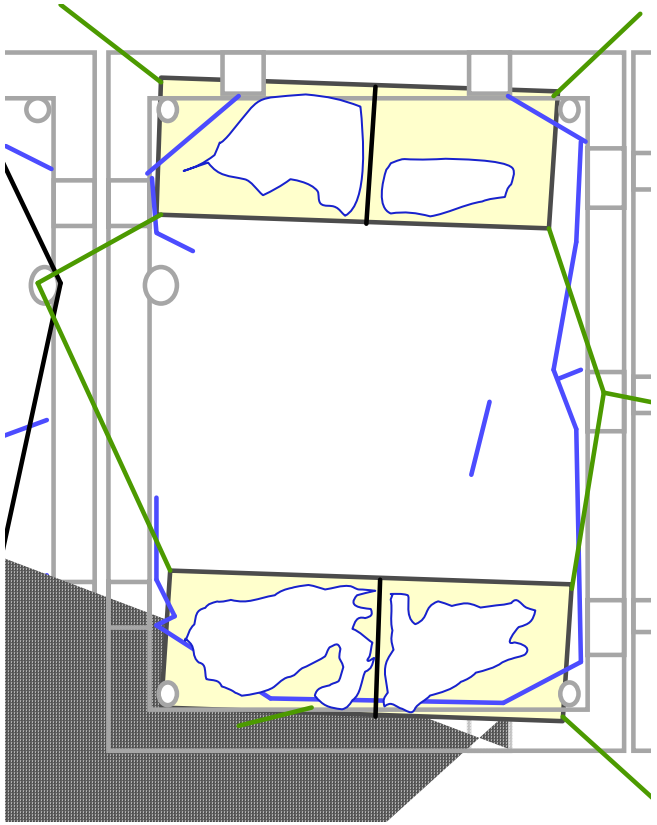
SM2

SM3B

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

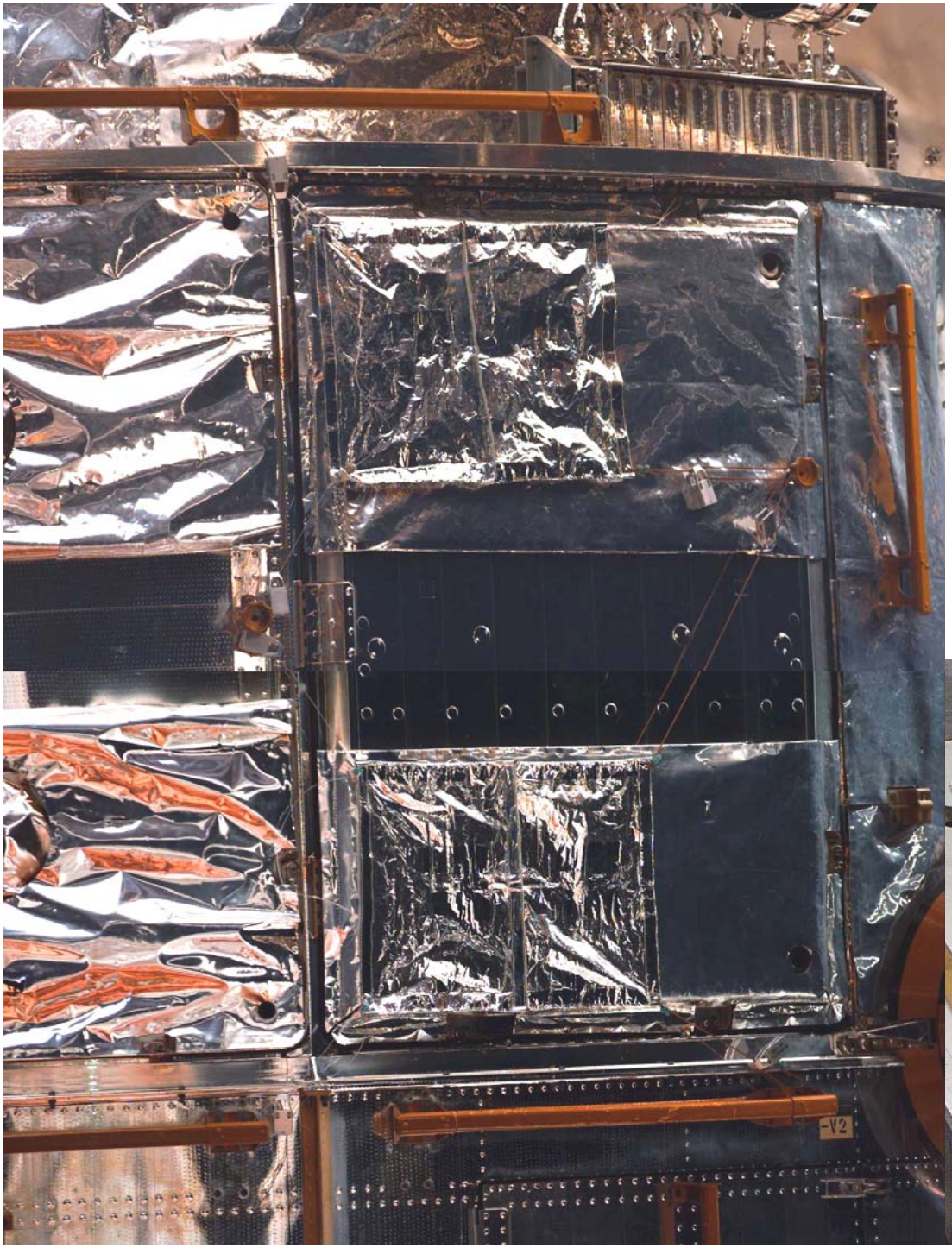
Bay 8 Details

- Original 17 layer MLI on Bay door cracked but in place
- Patches installed Feb 1997 - single layer 2 mil FEP Teflon aluminized
- Electronic equipment inside at risk of exceeding upper temp limit
- Patches & MLI to be removed and replaced with a NOBL



Bay 10 Patch Prior to Installation

S82E5689 1997:02:17 14:47:50

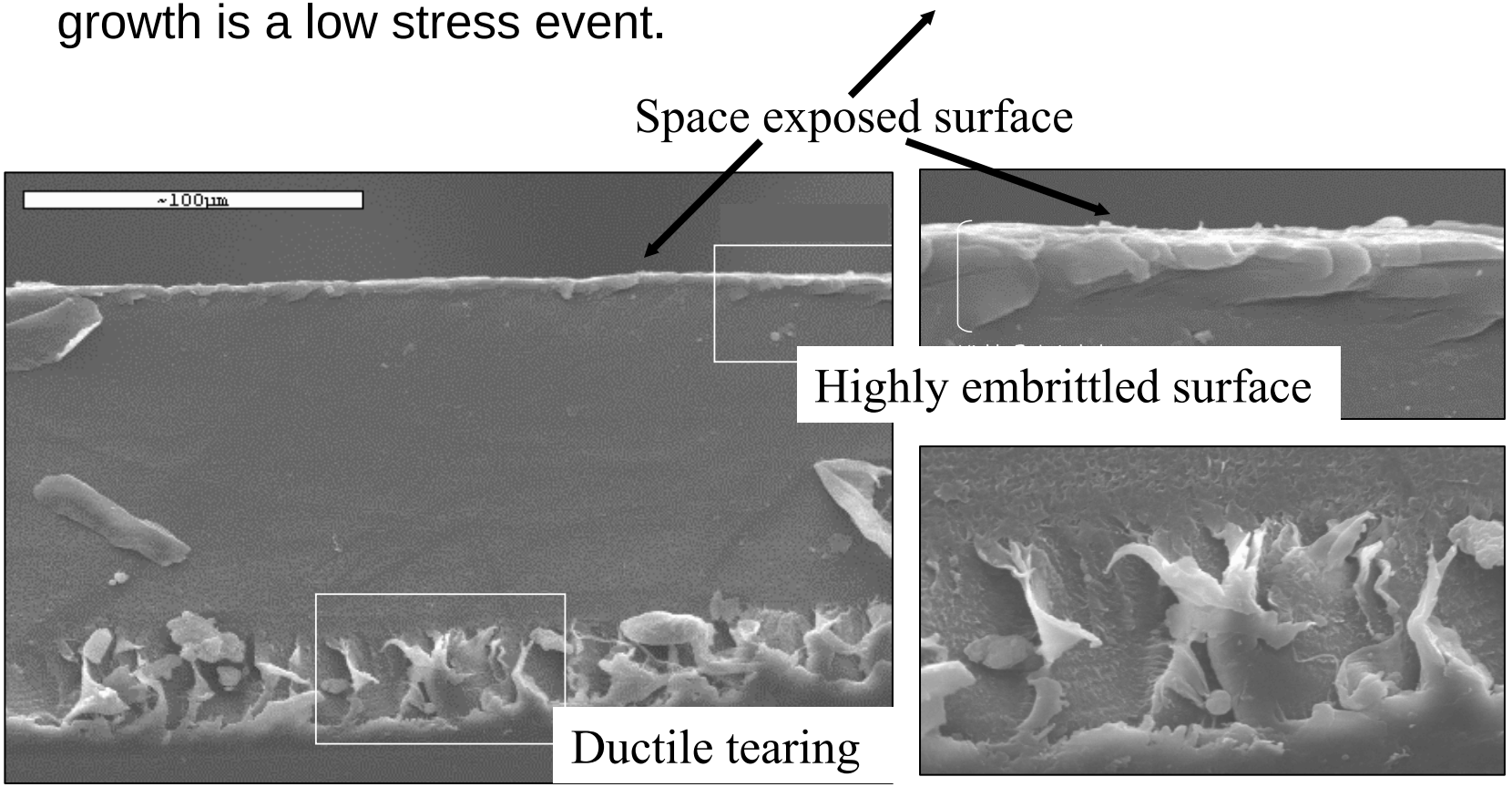


Bay 10 On Orbit Prior to MLI Removal

Bay 10 MLI in its EVA Transport Bag

SEM images of 5 mil Teflon 9.8 years exposure

- Cracks initiate on the space exposed, embrittled, side - propagate through the thickness, then laterally. The slow crack growth is a low stress event.



Brief History of HST MLI

	Observations	Installed	Retrieved	Analyses
SM1	No mention of cracks by crew; cracks visible in post-flight observations & photographs	Magnetometer cover-R & SA2	Magnetometer cover MLI & SA1 drive arm MLI	
SM2	Crew noted significant damage to MLI, ad-hoc photo survey - 95 cracks greater than 5” & yellow pigment on EVA gloves	Patches: two 17 layer (5 mil outer layer) on LS & 4 single layer (2 mil) on Bays 8 and 10. Wire/Ty-Raps	~12 in^2 5 mil aluminized Teflon from LS & FOSR tape from cryo-vent cover	Synergistic effect of thermal cycling, deposited e^- and H^+, and solar radiation =>slow crack growth
SM3A	150 total cracks, 9 existing much longer, yellow pigment on EVA gloves	Contamination covers - handrail (5), door knob (2) and handle (1); and NOBLs (3)	~1800 in^2 Bay 10 MLI (17 layers 5 mil VDA FEP top layer) & 2 Bay 10 patches (2 mil single layer)	synergistic effect confirmed, contribution of peak temperature established
SM3B	260 total cracks, 38 existing grew longer, yellow pigment on EVA gloves. Complete photo survey executed.	Installed NOBL on Bay 6	SA2 drive arm MLI & bi-stem thermal shield (BSTS), diode box FOSR tape, one flake of paint	synergistic effect confirmed, AO erosion of 1/3 mil VDA2 Kapton measured. Effect of temperature on embrittlement confirmed: solar facing BSTS fully embrittled, anti-solar not.

Outer Layer MLI
SM4 Predicted Condition

- Nominal MLI Outer Layer
  - **best case**: continued linear progress in crack initiation and propagation
    - Many small cracks initiated
    - Some propagation of existing cracks
    - Partially embrittled Teflon **will not** crack upon crew contact
  - **worst case**: high dose rate and increased total dose significantly increase degradation rate
    - Many small and large cracks occur
    - Significant propagation of existing cracks
    - Numerous intersecting cracks lead to “islands” of free material
    - Fully embrittled Teflon **will** crack upon crew contact - much debris generated
- Patches
  - Best case: small cracks progress, not visible on orbit
  - Worst case: attachment failure; significant material loss

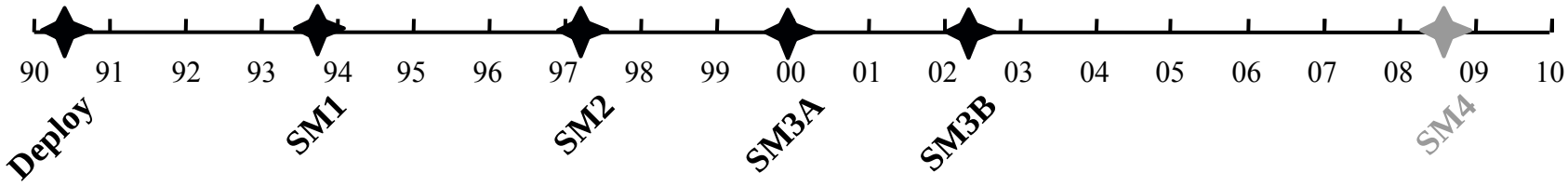
SM4 Mitigation Techniques

- Ordering of tasks
- Flight Rules
- FOD checks for all connectors
- Inspection/Cleaning of EMUs prior to donning

Outer Layer MLI Degradation Mechanism

- Cracking Mechanism:
  - Synergistic effects of load (stress concentrations), total dose, dose rate, temperature and film thickness
  - Load from stress concentrations inherent in manufacturing process, shrinkage, blanket build and installation, and thermal cycling
  - Dose rate is a function of solar cycle
  - The higher the upper temperature limit during thermal cycling, the more severe the degradation
  - The thinner the film for identical exposure conditions, the more degraded it will be

- Factors by Mission
  - Launch to SM1: high dose rate but not total dose
  - SM1 to SM2: detrimental combination of load, dose and t-cycles
  - SM2 to SM3A: lower dose rate and lower load (relieved by cracking)
  - SM3A to SM3B: moderate dose rate
  - SM3B to SM4: high dose rate and total dose and ~ 107,500 thermal cycles



Handrail, Doorknob & Handle Paint

SM4 Predicted Condition

- Free pigment on all painted surfaces
  - Handrails
  - Doorknobs
  - Door handles

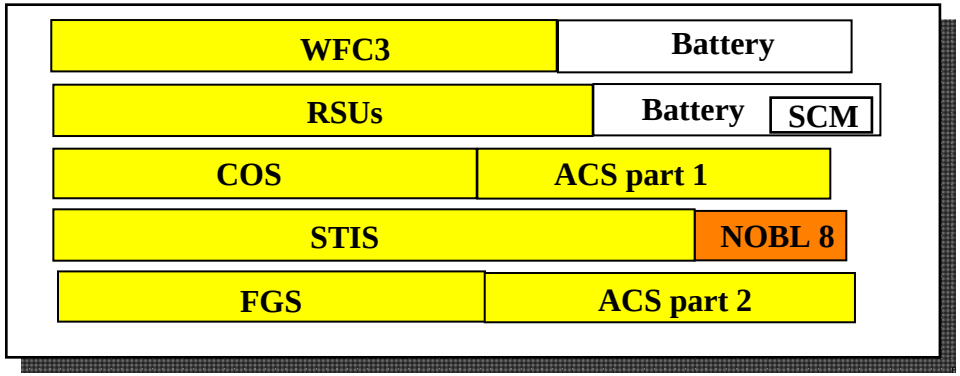
Mitigation Techniques

- Flight rules
- Glove inspection prior to opening of Aft Shroud
- Contamination covers if necessary
  - Installed on SM3A
    - 5 handrail covers
    - 2 knob covers
    - 1 handle cover

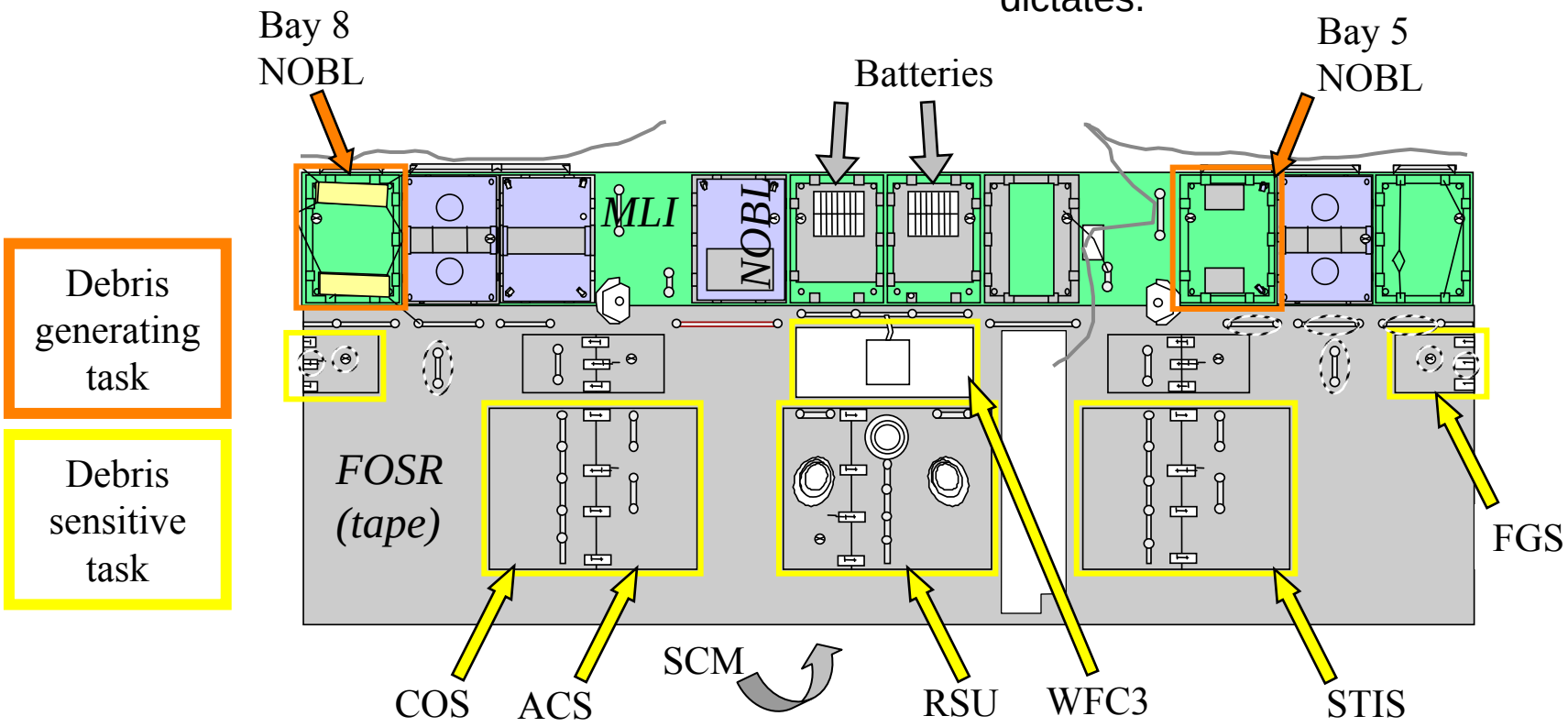
Handrail Paint Degradation Mechanism

- Handrail Paint:
  - Silicone overcoat on top of polyurethane paint with inorganic pigment
- Free pigment
  - Atomic oxygen (AO) undercuts silicone, erodes binder
  - Pigment left on surface and transfers to gloves when touched
  - Occurs all around telescope
- Flaking (peeling, scaling, lifting; not discoloration)
  - UV + AO damage shrinks silicone
  - Good bond between silicone and paint;
  - Failure between paint and aluminum
  - Occurs with high solar exposure (+V3)

SM4 Task Summary

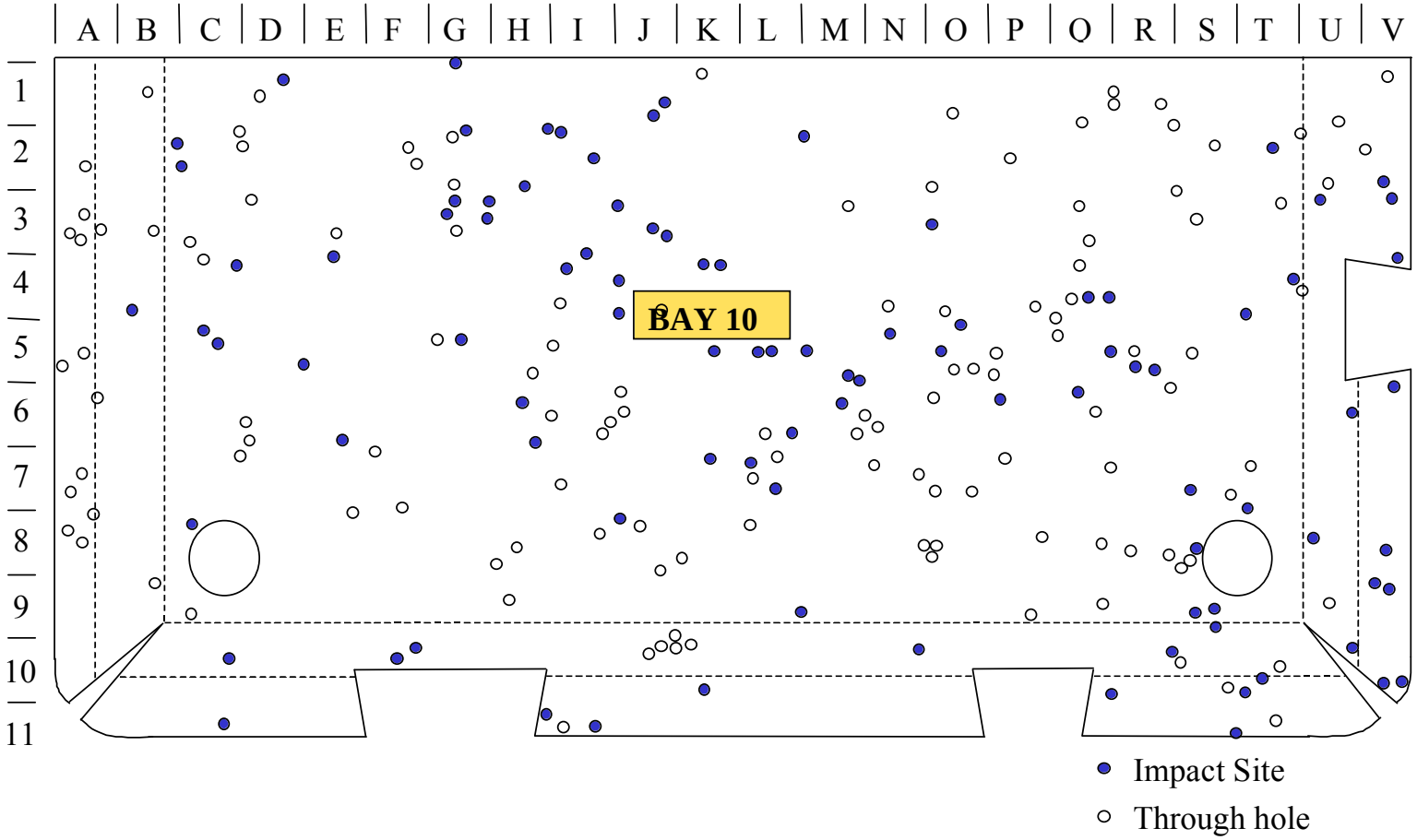


- Debris generating tasks follow debris sensitive tasks.
- Debris sensitive tasks are surrounded by FOSR (tape), not MLI.
- Handrail/handle covers may be installed as their condition dictates.

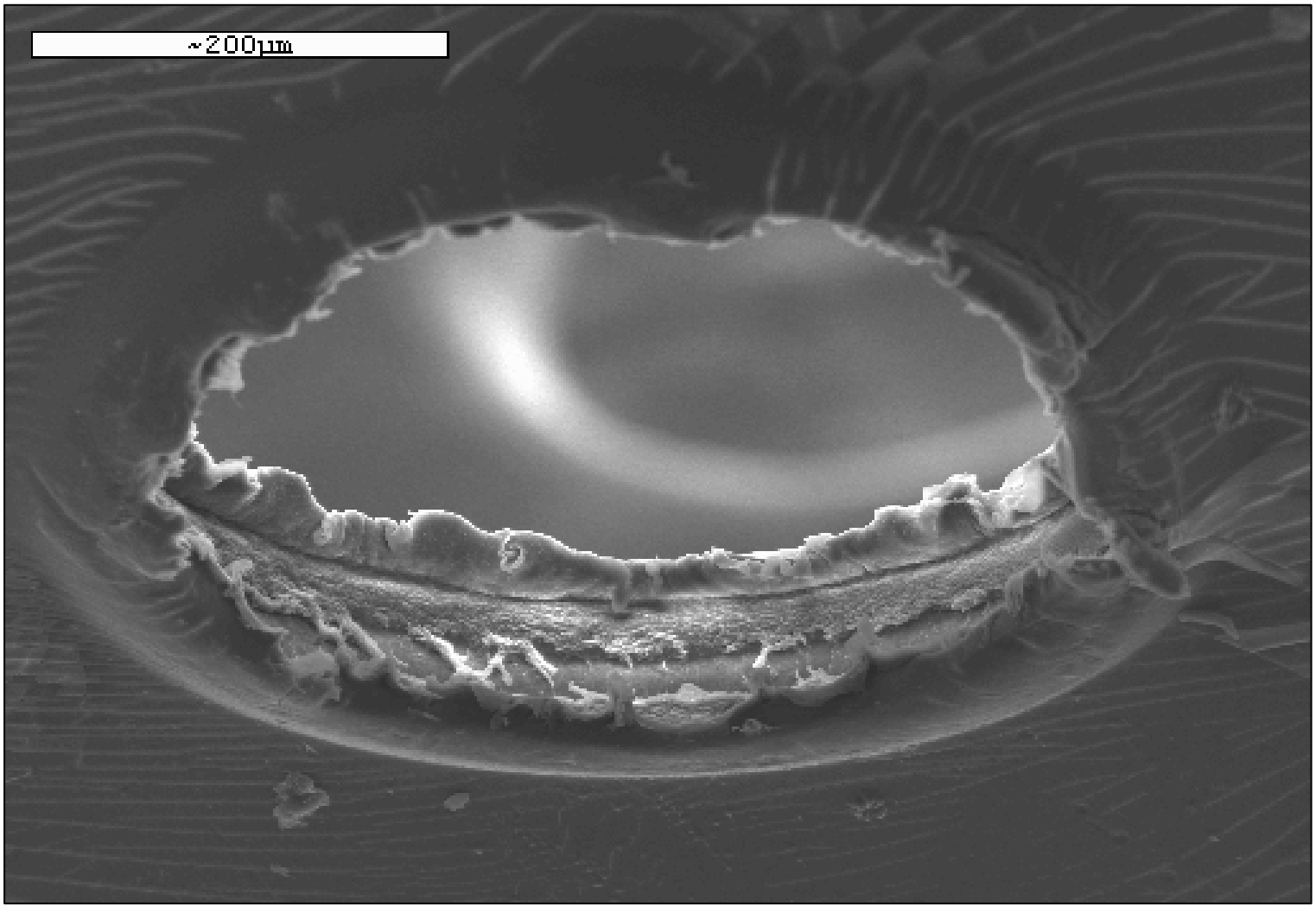


SM4 Mission Summary

- MLI outer layer will look bad
- Some locations (hot spots) will be brittle, most will not
- Majority of SM4 tasks are independent of MLI, mitigation strategies are in place


A B C D E F G H I J K L M N O P Q R S T U V
1 2 3 4 5 6 7 8 9 10 11
BAY 10
Impact Site
Through hole

Detail of a Through-Hole



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