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# Aqueous Glue Setting in Double-Coated Paperboard Systems

The Impact of Application System & Individual Coating Layer Thickness on Glue Bond Formation

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**RETHINK PAPER:**  
**Lean and Green**



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# Motivation

- The performance of the glue bond (glueability) is critical throughout the life cycle of coated paperboard
  - Converting, Filling, Distribution, Display, End Use
- Manufacturers typically use qualitative methods to ascertain the performance of the glue bond
  - Percent fiber tear, Time to reach fiber tear, Coated to Coated/Uncoated
- The design of the coating formulation along with basestock properties has been shown to greatly influence the glueability
  - Binder type and level, pigment choice, single layer or multilayer, degree of sizing, furnish composition
- Although there has been considerable work reporting on the effect of application system on coating structure; very little has been reported on the impact to glueability in coated paperboard



# Objectives

- Determine the impact of coating application technique and coating layer thickness for double-coated paperboard on:
  - Glue setting time
  - Glue strength
  - Coating structure
- Evaluate different microscopy techniques for evaluating the degree of penetration of the aqueous portion of water-based adhesives into coating structure



# Presentation Outline

- Formulations & Pilot Trial Details
- Coated Board Testing Results
- Glue Setting & Strength Results
- Microscopy Approaches
  - Scanning Electron Microscopy
  - Confocal Laser Scanning Microscopy
  - Optical Microscopy
- Conclusions
- Q&A



# Formulations

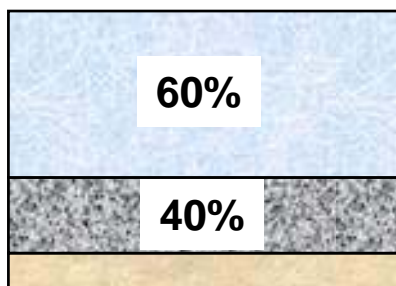
	Basecoat	Topcoat	Topcoat (air knife)
#1 Clay	77	65	65
Calcined Clay	23	20	20
TiO <sub>2</sub>		15	15
SB Latex	14	17	17
Thickener	0.5	0.5	0.5
% Solids	64	60	46
pH (ammonia)	8.5	8.5	8.5



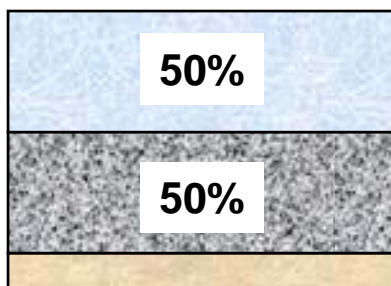
# Pilot Coating Trials

System	Basecoat Application	Topcoat Application
A	Rigid Blade	Rigid Blade
B	Rigid Blade	Air Knife
C	Rod	Rod
D	Rod	Air Knife
E	Film Coater	Air Knife

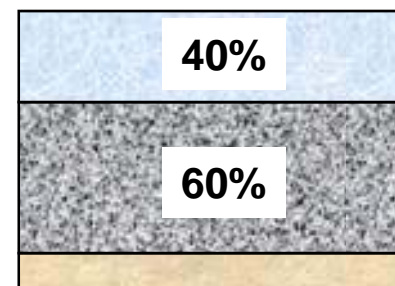
- Coating Speed: 275 mpm
- Coat Weight: 24 gsm (total)
- Substrate: 10 point Bleached Board



**Spl 1**



**Spl 2**



**Spl 3**



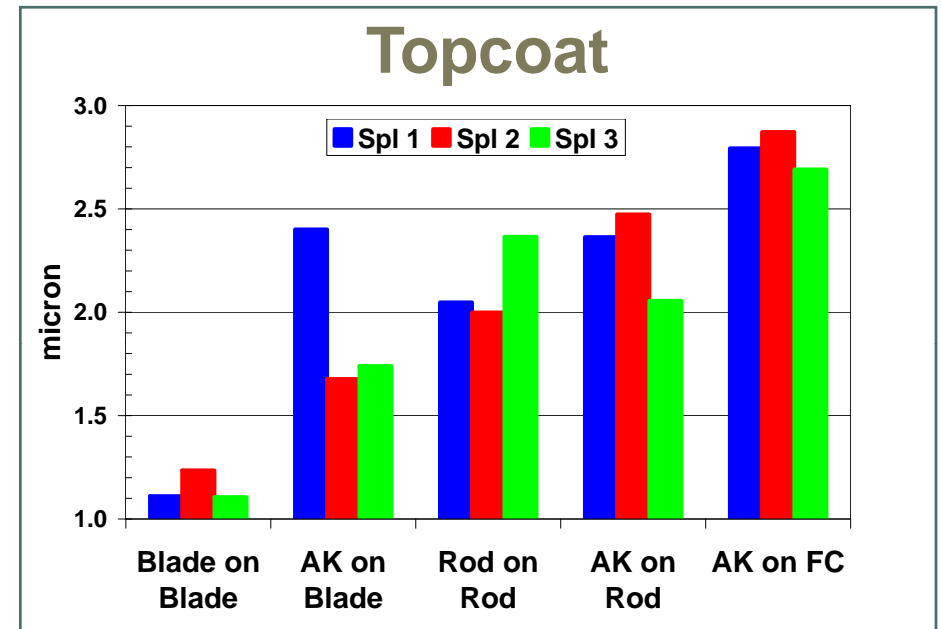
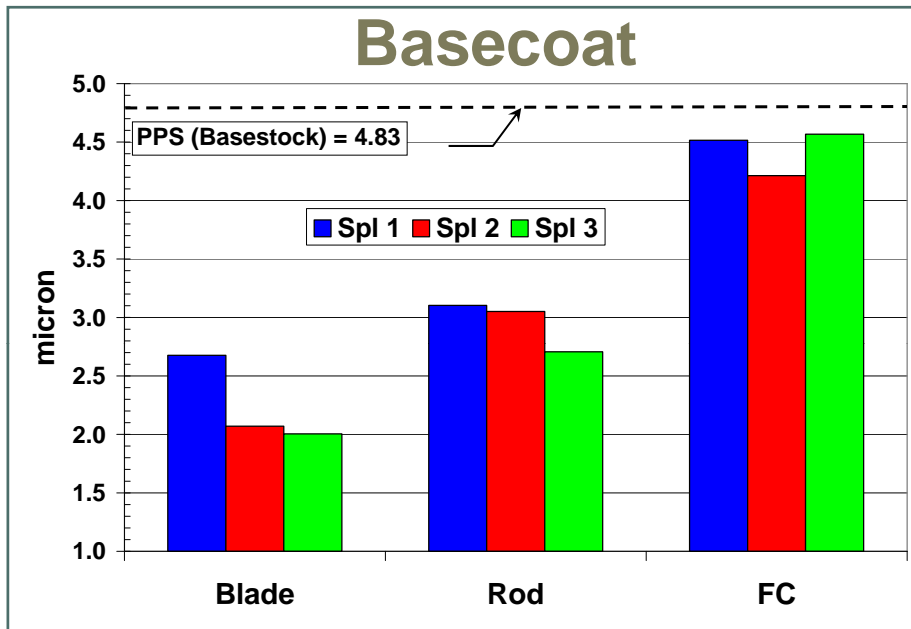
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# Testing Results



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# Surface Roughness (PPS)



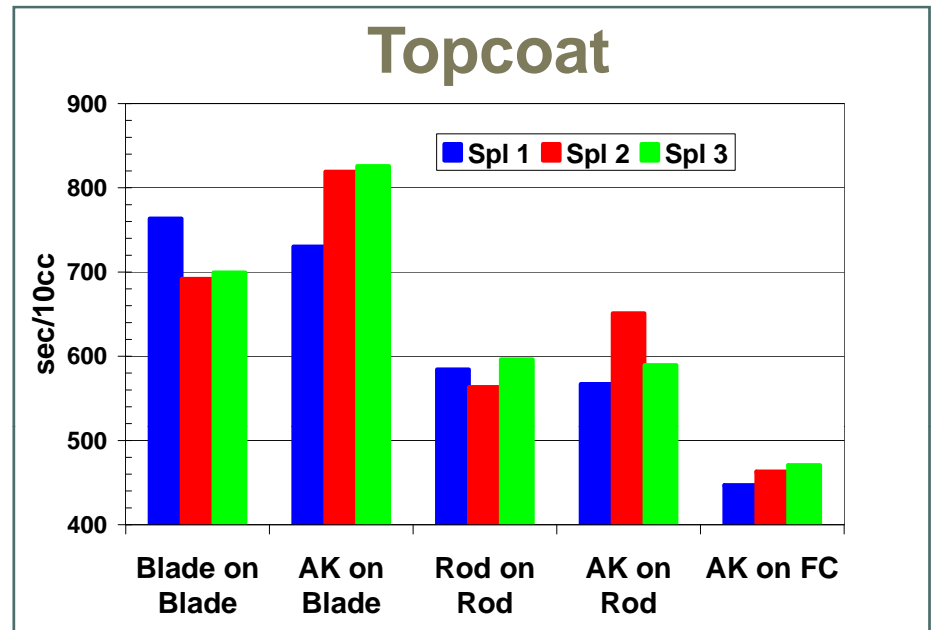
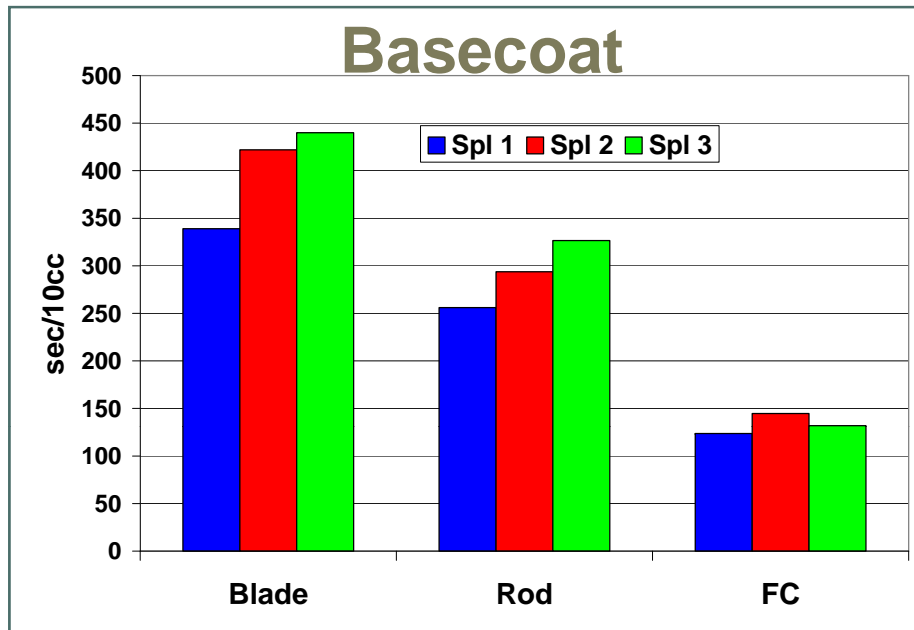
- Surface roughness = f(contour vs. leveling) & coat weight, ~~as expected~~
- Rougher surface with thicker (more randomly oriented coating structure) air knife topcoat



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# Coating Porosity (Hagerty)



- Particle alignment reduces porosity in basecoat (Blade > Rod > FC)
- Porosity reduced with increasing coat weight of basecoat
- Air Knife topcoats; thicker coating results in increased porosity
- Basecoat structure is dominant factor for overall porosity
- Film coat contributed highest porosity – least pigment alignment

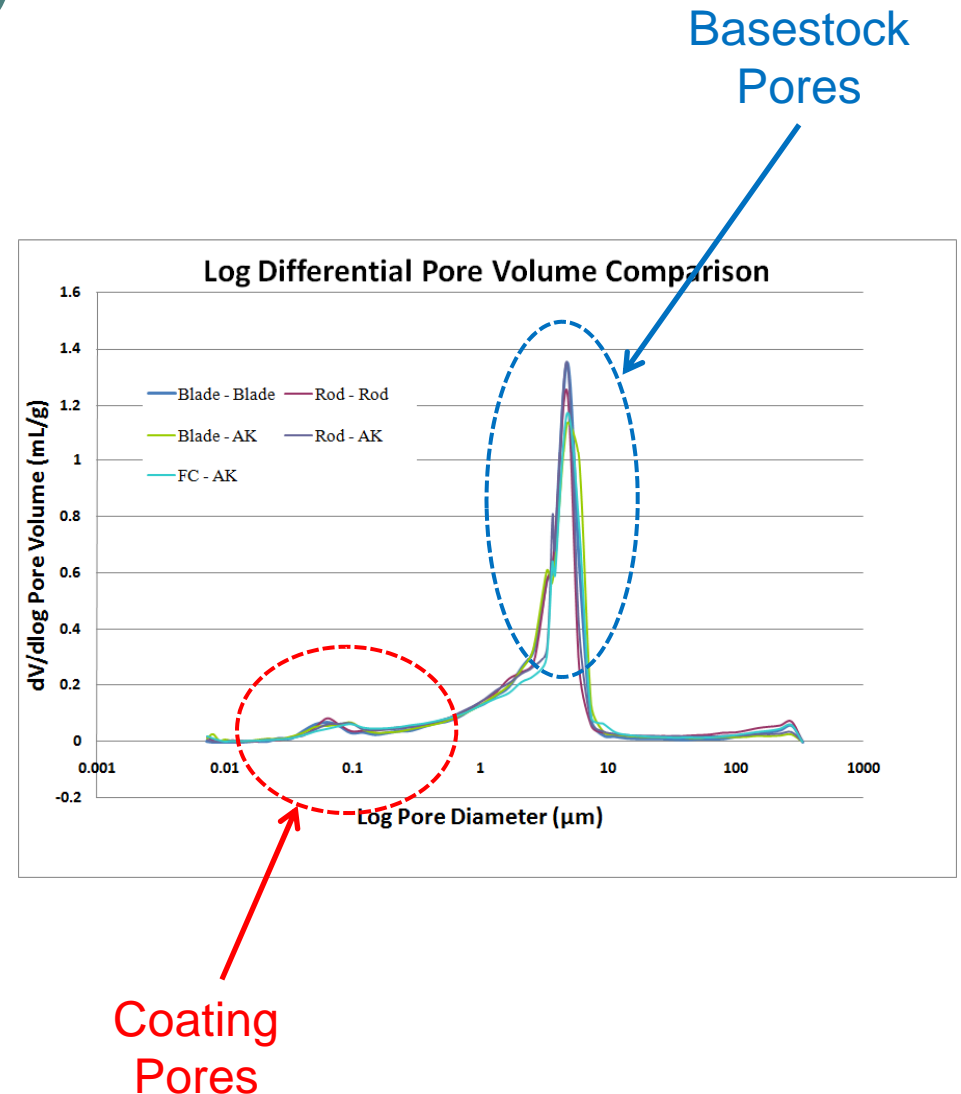


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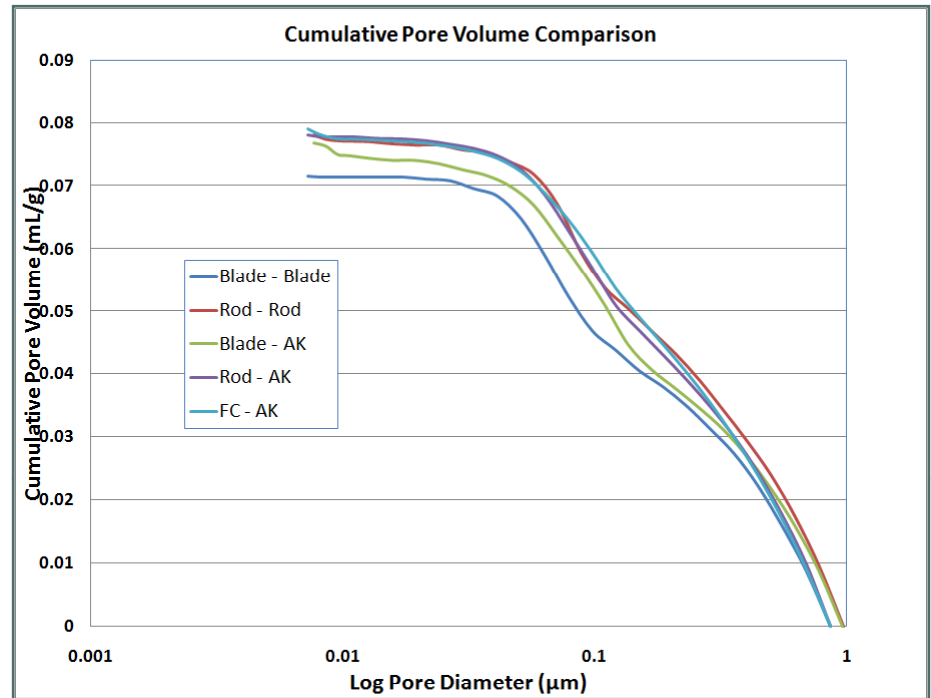
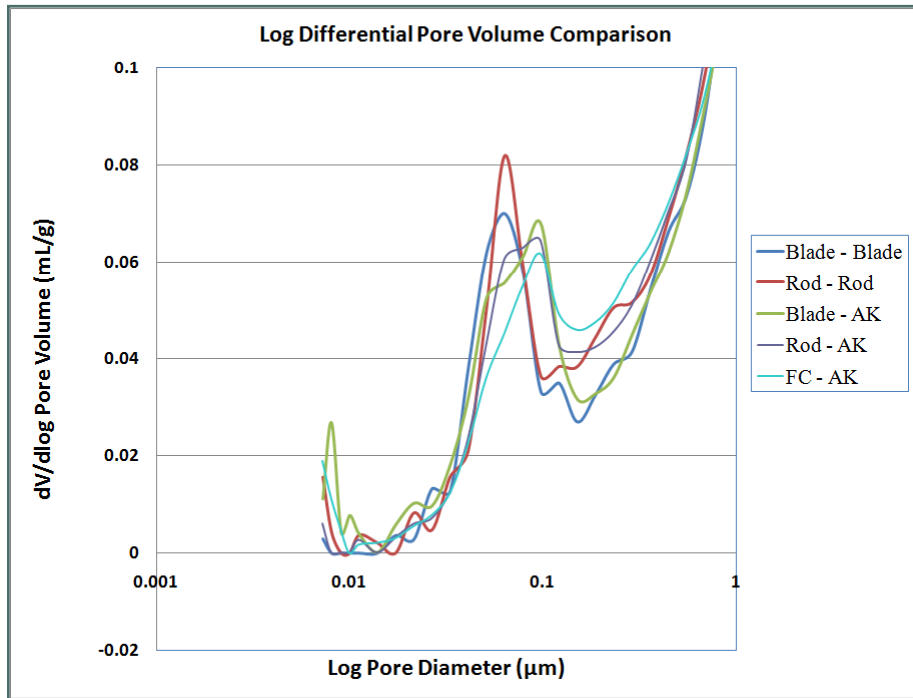
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# Mercury Porosimetry

- Mercury Porosimetry (Micromeritics Autopore III) used to characterize pore volume and pore size distribution of samples.
- Low pressure utilized for Hg intrusion into larger pores (basestock), while correspondingly higher pressure used for coating pores.
- Pressure required is converted to equivalent pore size using Laplace equation and surface tension of Hg.
- Medium basecoat/Medium topcoat sample of each coater combination analyzed (Equivalent coat weight & Identical formulations).



# Mercury Porosimetry Results



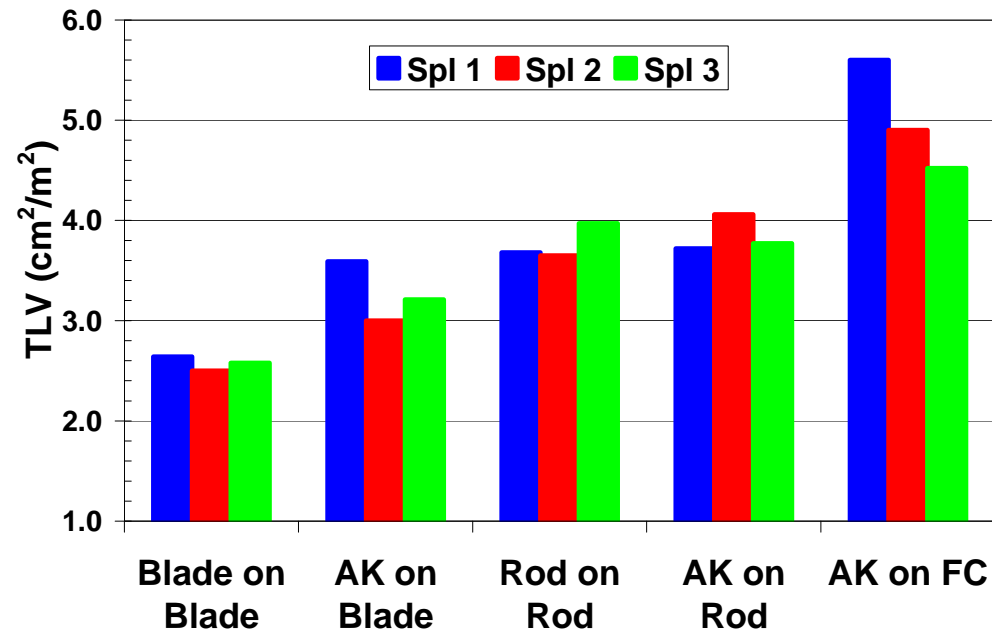
- Blade basecoat results in slightly decreased total pore volume
- Air Knife topcoat causes shift in dominant pore size to larger pores
- Cumulative Pore Volume (after subtracting basecoat) shows:
  - $(\text{Rod} - \text{Rod}) > (\text{Rod} - \text{AK}) > (\text{FC} - \text{AK}) > (\text{Blade} - \text{AK}) > (\text{Blade} - \text{Blade})$



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# Bristow Wheel Absorption



- Total liquid volume transferred at 0.1 sec contact time
- Diluted, dye-based inkjet ink utilized
- Absorption results mirror the Hagerty porosity results
- Thicker air knife topcoat contributed to faster absorption

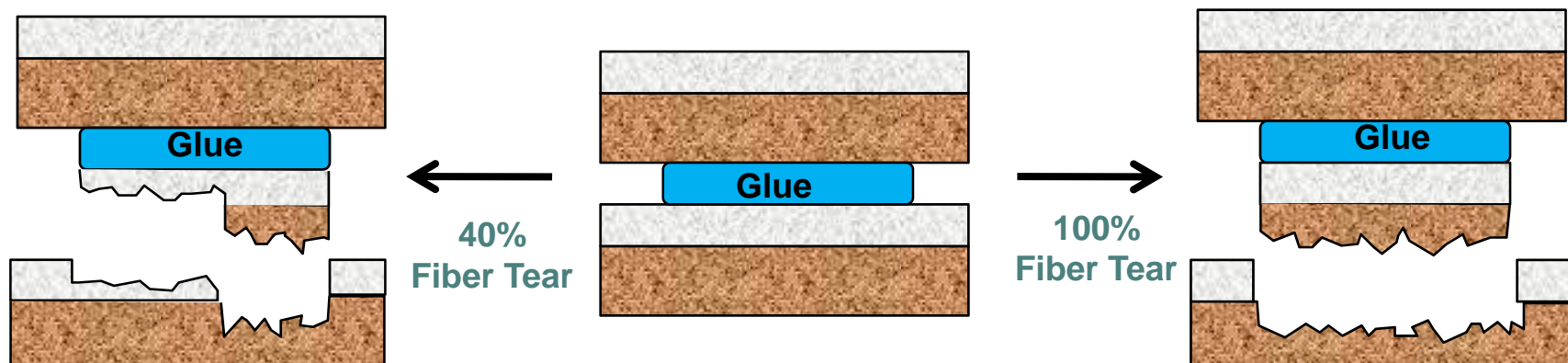


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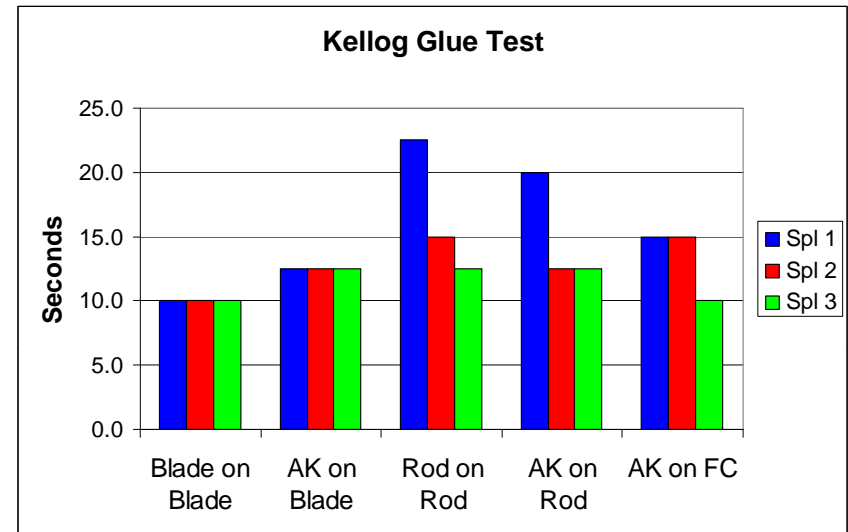
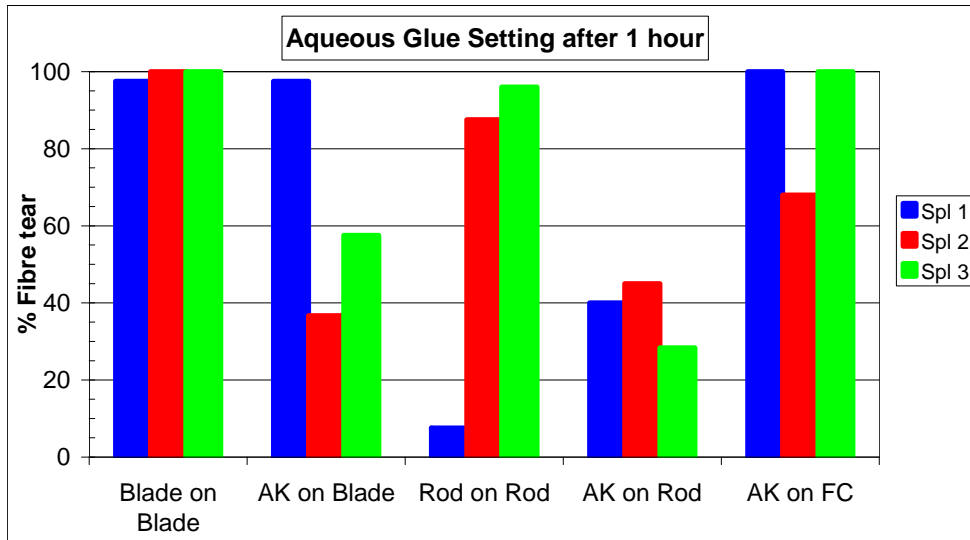
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# Glue Testing Methods

- Aqueous Glue Setting Test
  - 37% solids, Polyvinyl acetate acrylic copolymer (Reynolds Company)
  - Glue applied to coated surface (0.38 mm bird bar)
  - 3 lb. steel plate in place for three minutes
  - % fiber tear evaluated after 1 & 2 hours set time
- Kellogg Glue Test
  - 57% solids, polyvinyl acetate liquid adhesive (Capital Adhesives)
  - Glue applied to coated surface (15 mils, 3 inches wide)
  - 5 lb. steel roller passed over sample once in both directions
  - Glued sample pulled back at specified time intervals and degree of bond determined visually.



# Aqueous Glue Setting Results



- Blade – Blade system exhibits fastest setting and strong bond formation
- Difficult correlation with regards to coat weight split; however
  - A thin Rod basecoat (regardless of topcoat) shows slowest setting times
  - Same results with hot melt glue (polypropylene base) with regards to rod basecoat
- Air Knife topcoat tends to reduce glue strength compared to blade & rod topcoats (larger surface pores ?)
- Faster glue setting did not correlate to better final glue strength



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# Microscopy Approaches for Investigating Aqueous Glue Penetration



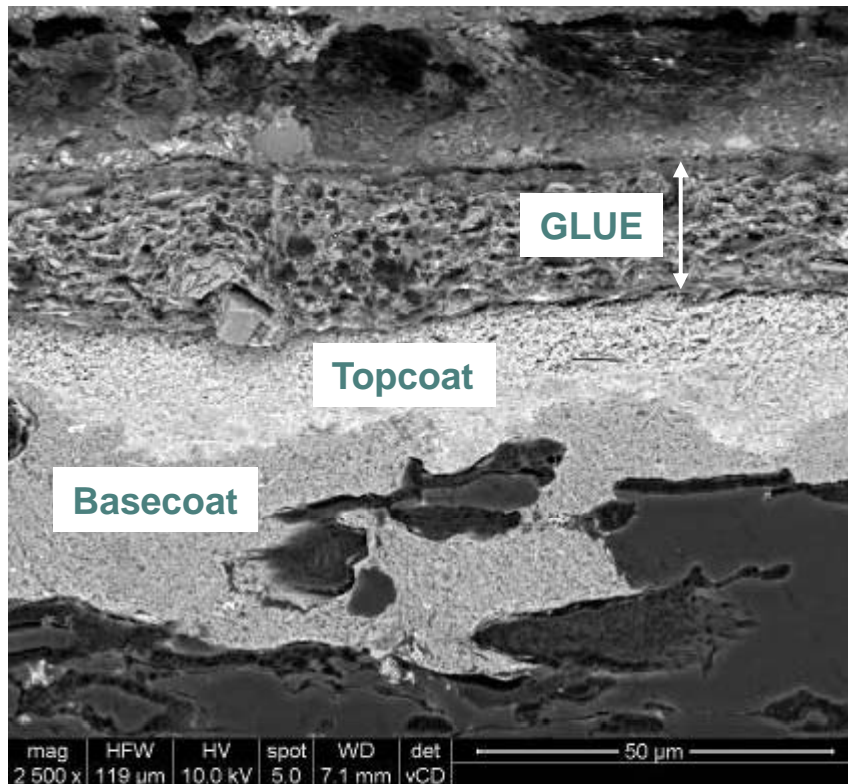
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# Microscopy Approaches

**Objective:** Follow penetration of aqueous glue as it migrates into coating structure and basesheet.

**Goal 1:** Assess the influence of coating structure on the degree of penetration.

**Goal 2:** Correlate penetration (anchoring) with glue setting results.



## Tracers for Glue:

1. Metal Oxide Nanoparticles
2. Rhodamine-B dye

## Techniques

1. Electron Microscopy (SEM)
2. Confocal Laser Scanning Microscopy
3. Optical Microscopy (Dark Field Illumination)

## Cross Section Preparation

1. Microtome
2. Epoxy Embed and Polish
3. Ion Polish

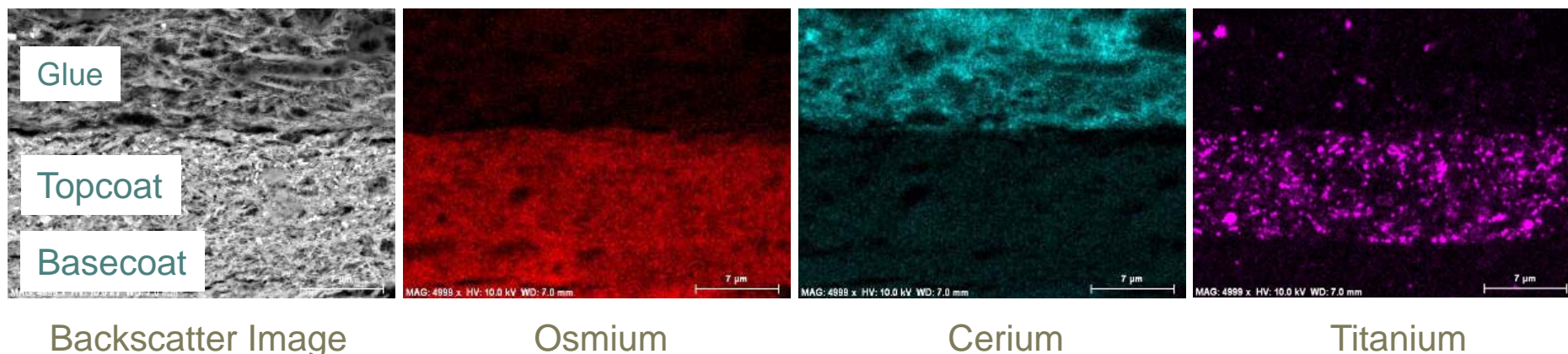


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# SEM (Metal Oxide Nanoparticles)

Metal	Formula	State	P.S. (nm)
Iron	$\text{Fe}_2\text{O}_3$	Dispersion	< 50
Cerium	$\text{CeO}_2$	Dispersion	< 25
Zinc	$\text{ZnO}$	Dispersion	< 100

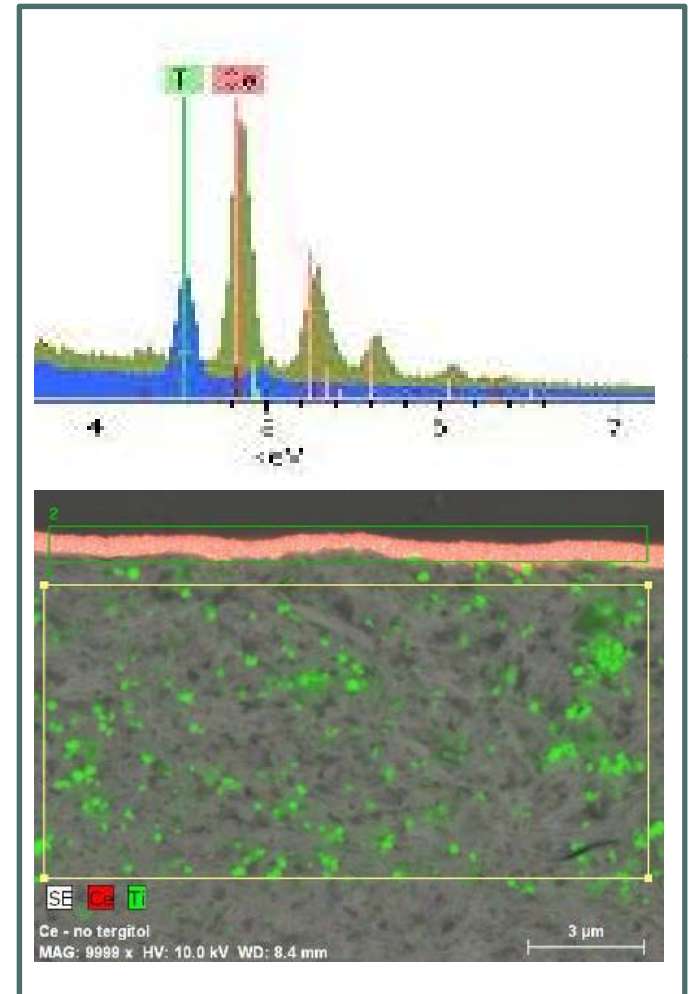
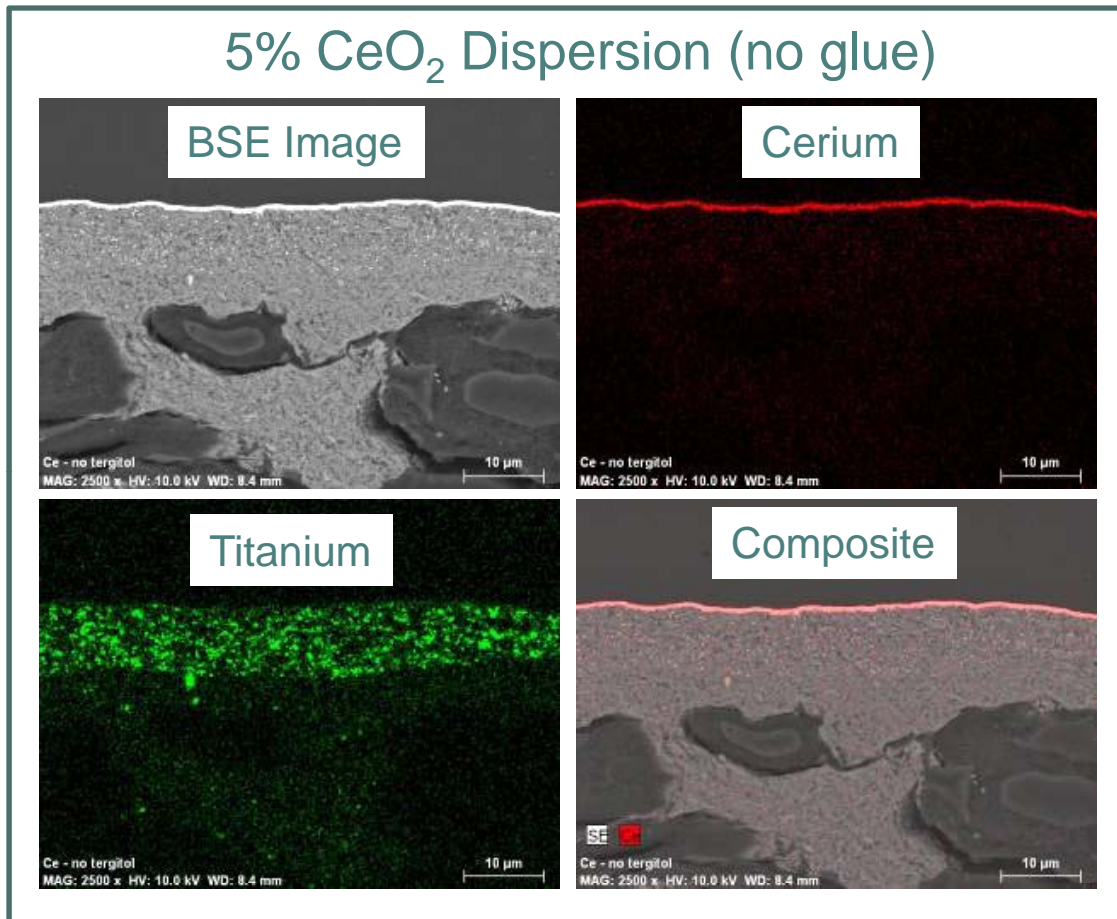
## 1% $\text{CeO}_2$ in PVAC Glue



- Elemental X-ray spectra showed no penetration (Fe, Ce, Zn)
- Very high Silicon signal from glue layer (clay fillers)

# SEM (Metal Oxide Nanoparticles)

## 5% CeO<sub>2</sub> Dispersion (no glue)



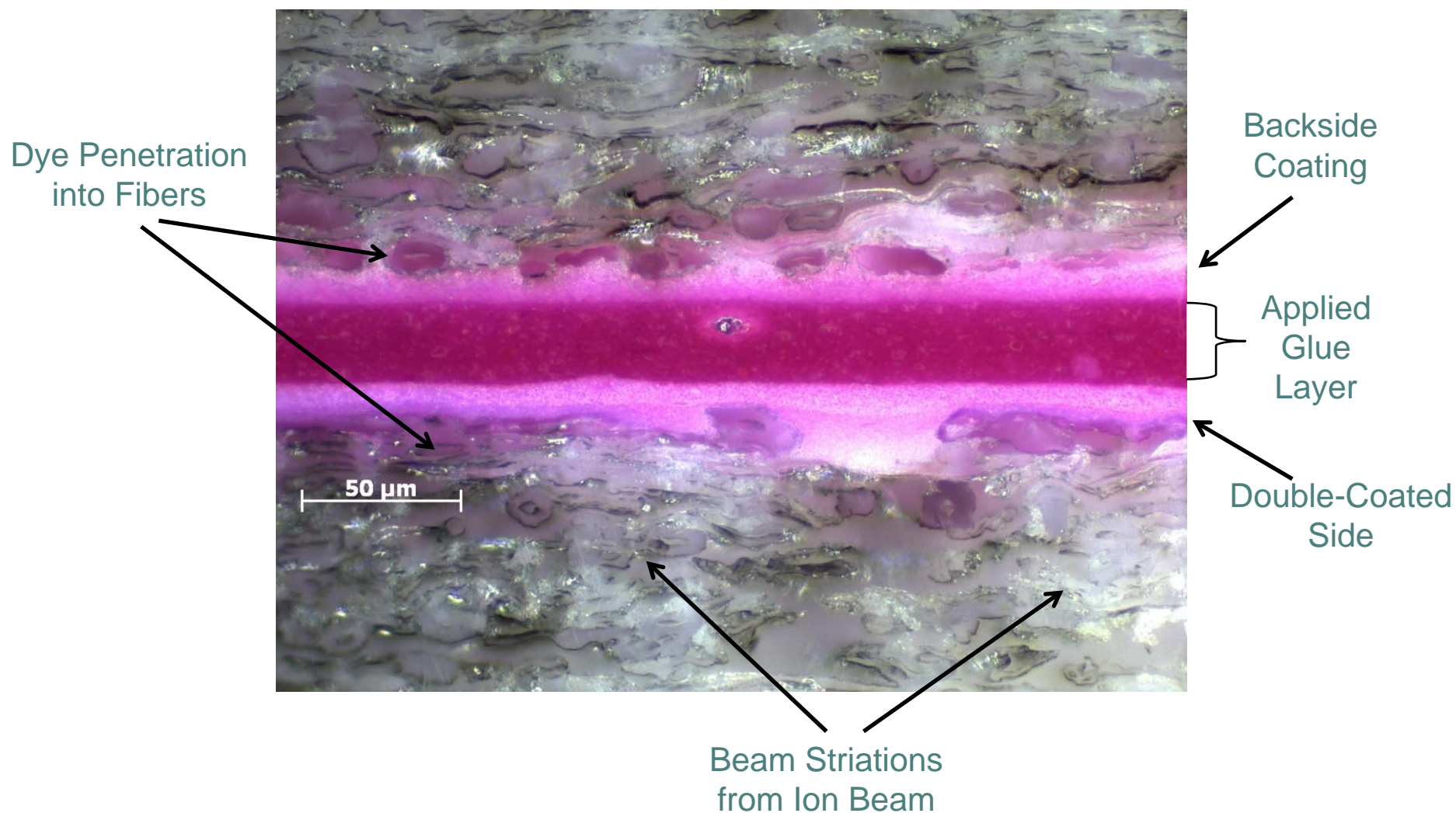
- Metal oxide dispersions formed dense layer at surface
- With/without additional surfactant to aid wetting
- Spectra (left figure) exhibited no penetration into coating structure

# Optical Microscopy (Dark Field Illumination)

- Aqueous glue stained with Rhodamine-B dye.
- Glue applied via standard testing procedure.
- Cross sections prepared using JEOL Ion Polisher
  - Minimize sample damage, smearing & deformation
  - 18 hours per sample
- Images obtained with Zeiss Axio Optical Microscope
  - Dark Field Illumination
- Measurements were made using Image J Software
  - (National Institutes of Health: <http://rsb.info.nih.gov/ij/>)



# Optical Microscopy Image Example



# Optical Imaging Results

Basecoat Application	Topcoat Application	Coating Penetration (μm)	Glue Layer (μm)	Backside Penetration (μm)	Total (μm)
Blade	Blade	35.8	25.0	38.2	99.0
Blade	Air Knife	53.2	20.6	44.1	117.9
Rod	Rod	39.7	25.9	42.2	107.8
Rod	Air Knife	46.3	20.5	31.9	98.7
Film Coater	Air Knife	39.9	21.8	38.7	100.4

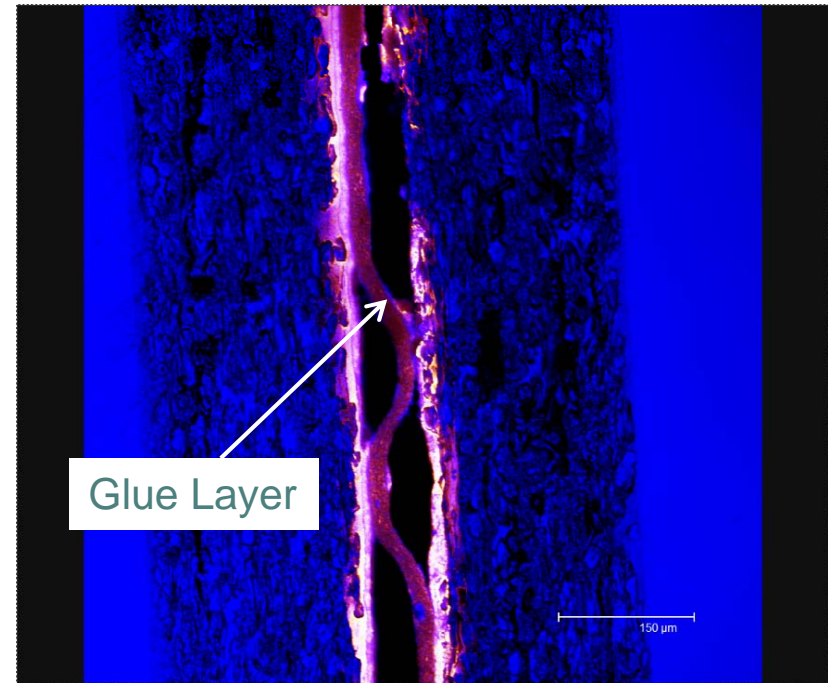
- Dye penetration into fiber matrix observed for all samples
- Indication of slightly deeper penetration into samples with air knife topcoats
- Ion polishing reduces sample damage at the expense of time (18 – 24 hrs)
- No correlation between dye penetration and glue strength and setting



# Confocal Laser Scanning Microscopy (CLSM)

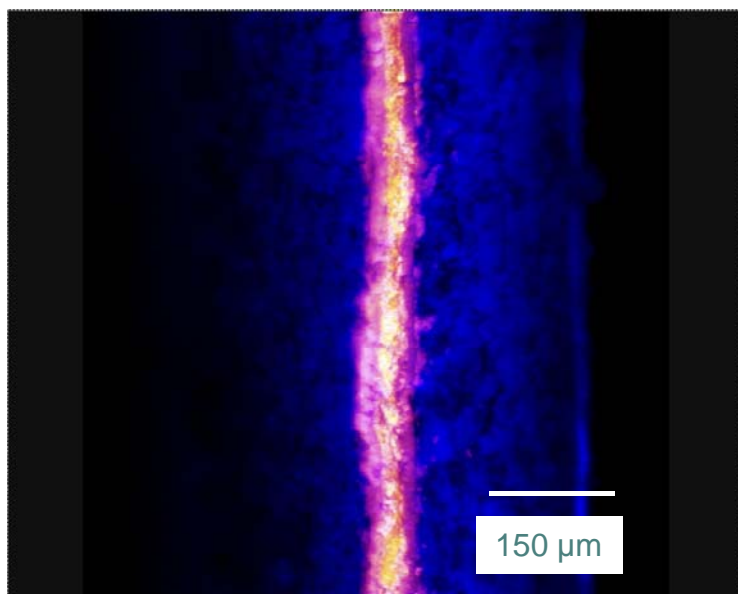
- Aqueous glue was stained with Rhodamine-B dye.
  - Emission wavelengths (535 – 680 nm).
- Glue application performed via standard protocol for all Spl2 samples (50% basecoat: 50% topcoat).
- Cross sections prepared
  - **Microtome**
  - Epoxy embed (metallographic polishing)
- Images obtained
  - Confocal
  - **Cross Sections**

Delamination observed when epoxy embedding and polishing

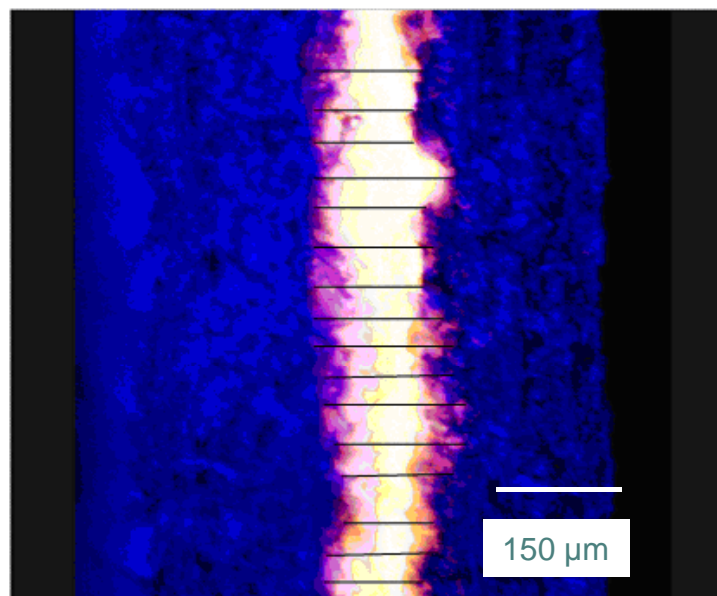


# CLSM Results

Bascoat	Topcoat	Avg. Width ( $\mu\text{m}$ )	Std. Dev. ( $\mu\text{m}$ )
Blade	Blade	145	21
Rod	Rod	141	33
Blade	Air Knife	81	49
Rod	Air Knife	69	7
Film Coater	Air Knife	61	9



Rod – Air Knife



Blade – Blade



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# Summary & Conclusions (1)

- Basecoat observations:

- Basecoat properties dominate the overall properties of the double-coated system, regardless of the split in the coat weight between the two layers.
- Blade applied basecoat produces a structure with the lowest porosity, highest smoothness, and lowest water absorption.
- Film-coater applied basecoat produces structure with highest porosity, lowest smoothness, and highest water absorption.
- Rod applied basecoats produce results intermediate between Blade & Film Coater.

- Topcoat observations:

- The contour coating provided by the air-knife produces a more open coating structure, rougher surface and a brighter overall paperboard.
- The overall strength of the air-knife topcoat surpassed that of both the blade and rod applied topcoats (lower ink slopes, higher passes to failure, higher IGT dry strength, and higher wet strength (IGT & Prufbau)).



# Summary & Conclusions (2)

- Glue Setting & Strength

- The blade – blade system produced samples with the fastest glue setting time and best overall glue strength.
- The thin rod applied basecoats produced the worst performing samples, regardless of topcoat application.

- Microscopy Technique Development

- Following the extent of aqueous phase penetration was facilitated by the use of a water soluble dye.
  - Better penetration to form deeper fiber-tearing bonds.
- Good preparation of cross sections is critical
- CLSM with cross sections prepared with ion polishing may provide the best combination for quantitative analysis.
- Developing techniques to follow extent of adhesive penetration into structure would be valuable.



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# Thank You Questions ??



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