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# “Investigating the Performance of Aged Fluorescent Penetrant Materials Phase II”

Sandia National Laboratories, Infrastructure Assurance & NDI, Albuquerque, NM, USA  
FAA-Airworthiness Assurance and NDI Validation Center (AANC)

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**FAA William J. Hughes  
Technical Center**

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# ABSTRACT

The Airworthiness Assurance NDI Validation Center (AANC) at Sandia National Laboratories has been tasked by the Federal Aviation Administration (FAA) to investigate the effectiveness of aged fluorescent penetrant materials (e.g., penetrant, emulsifier, developer) to detect fatigue cracks. The question that must be answered is “If a field inspector accidentally used a penetrant material that had an expired shelf-life would that reduce the likelihood of detecting fatigue cracks?” In March 2007, testing of unopened and aged Method A (water washable) systems revealed no evidence of diminished crack detection capability. In this second phase of testing we investigate aged Method D (post-emulsifiable, hydrophilic) systems which are formulated differently than their Method A counterparts and are commonly used in the field for detecting cracks in critical rotating engine components. The Method D penetrant materials have been stored at the AANC for a 7-12 year period and have not been opened or used prior to this effort. Well characterized titanium and Inconel<sup>®</sup> low cycle fatigue crack specimens were inspected using the aged penetrant materials and brightness readings were captured for each crack indication using a photometer. The specimens were also inspected using equivalent new penetrant materials and crack indication brightness readings were compared.





# Background

- Research question asked by the FAA (Al Broz) and Sandia (David Moore)...

**If a field inspector accidentally used aged penetrant materials/systems would that reduce the likelihood of detecting fatigue cracks?**

- An AANC study conducted in March 2007 revealed no strong evidence that the use of unopened and aged (8-10yr) Method A, Level 4 penetrant systems would automatically lead to reduced likelihood of crack detection
- This study focuses on the testing of aged Method D penetrant systems

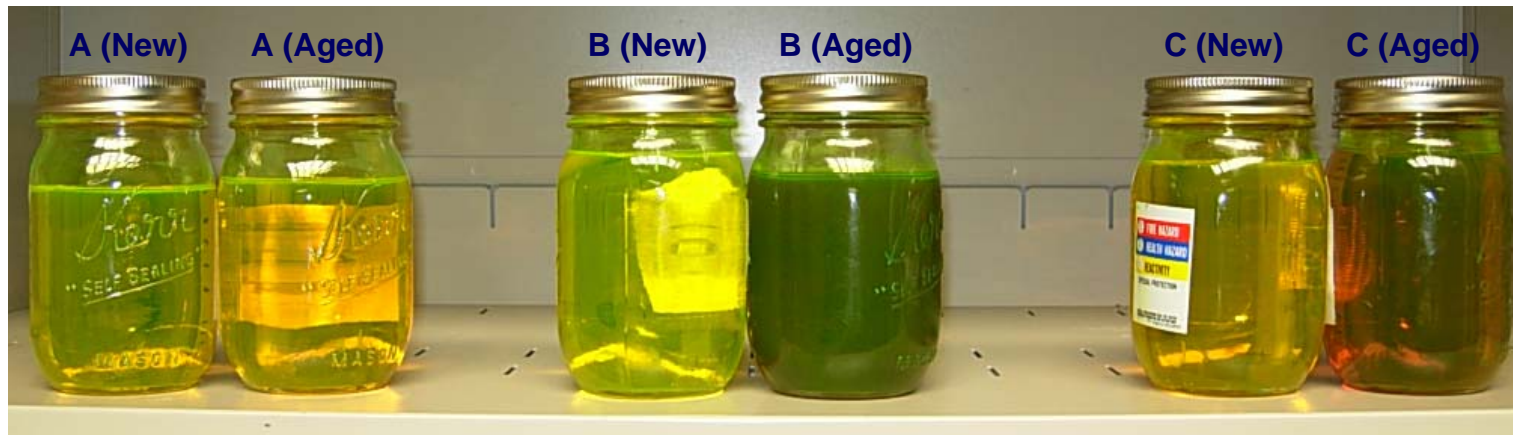


# Approach

- Penetrant materials used in this experiment have been stored at the AANC for a 7-12 year period and have not been opened or used prior to this effort
- Penetrant materials from 3 major manufacturers were available for testing
- The shelf life dictated by the manufacturers range from 3-5 years

Aged Method D, Level IV, Penetrant Material History			
Manufacturer	Penetrant	Emulsifier	Developer
A	2	3	8
B	6	7	11
C	5	0	11
Age (Years)	Actual Age - Shelf Life	Actual Age - Shelf Life	Actual Age

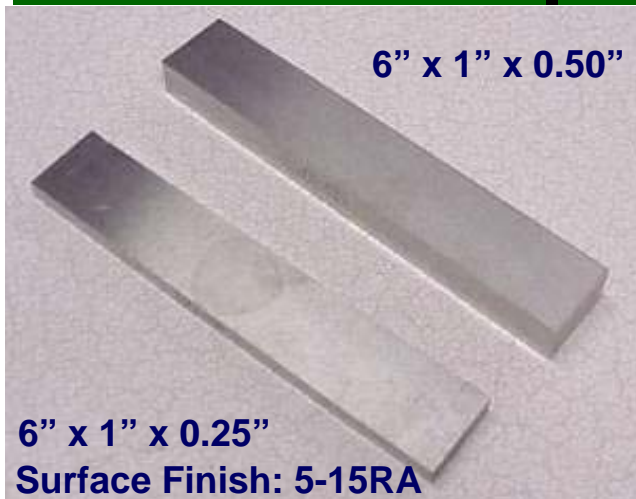
Visual comparison of aged penetrant and equivalent new penetrant



## Approach (continued)

- Critical rotating engine components are typically made of Titanium and Inconel® and are typically inspected with fluorescent penetrant in the field
- Well characterized titanium and Inconel® low cycle fatigue crack specimens were inspected using the new penetrant materials and brightness readings (5 run average) were captured for each crack indication using a photometer
- The specimens were also inspected using equivalent aged penetrant materials and crack indication brightness readings (5 run average) were compared

### Titanium 6-4 and Inconel® -718



### Crack Distribution

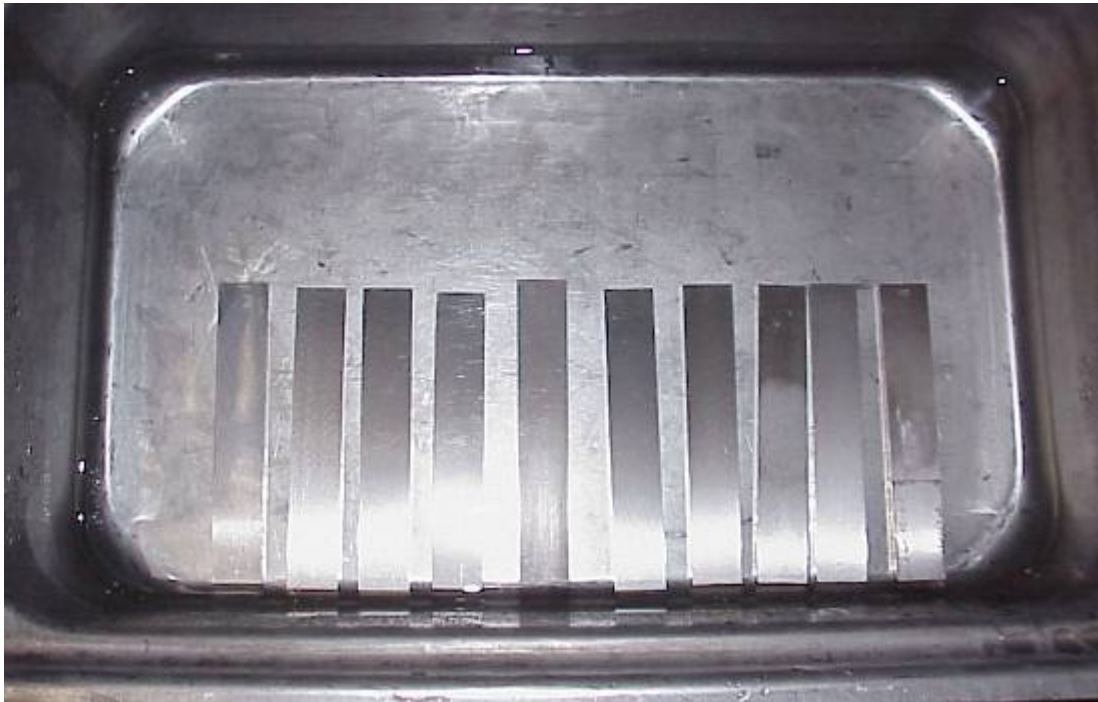
Inconel-718		Titanium 6-4	
Bar	Crack Length	Bar	Crack Length
1	0.022	1	0.013
2	0.024	2	0.031
3	0.025	3	0.035
4	0.028	4	0.038
5	0.028	5	0.039
6	0.028	6	0.040
7	0.029	7	0.042
8	0.030	8	0.042
9	0.034	9	0.046



# Specimen Cleaning

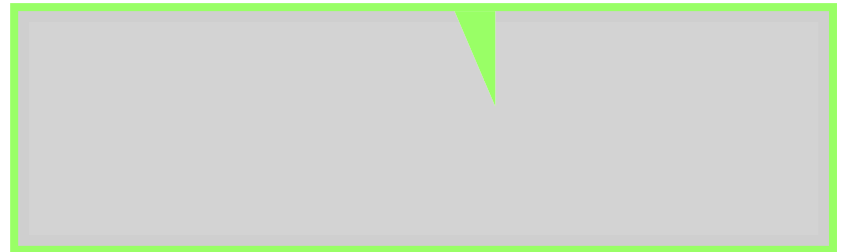
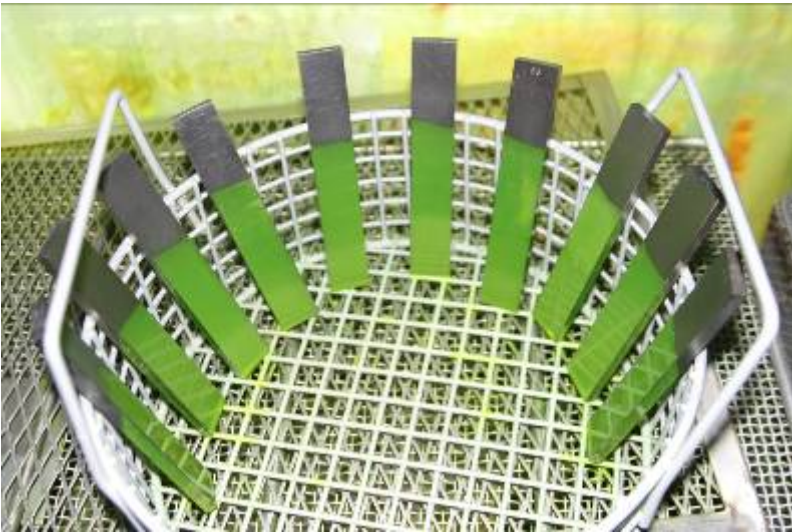
- Clean Bars in an Ultrasonic Acetone Bath for 15 Minutes

Performed before each run to ensure that the specimen and crack are clean from penetrant, developer, and possibly any other debris that could clog the crack



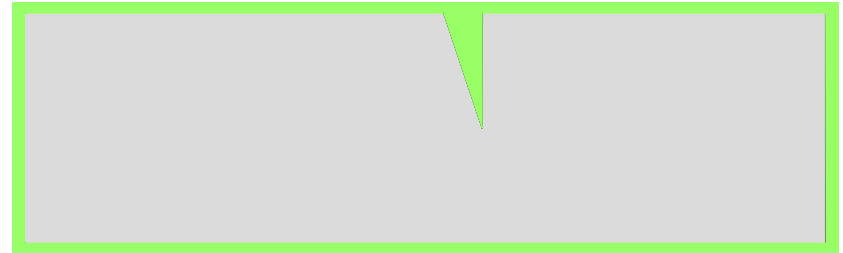
# Penetrant Application

- Application: Dip Specimen in Method D, Level 4 Penetrant
- Penetrant Dwell for 10 Minutes



# Water Rinse

- Water Rinse for 30 sec max at 78-82 °F



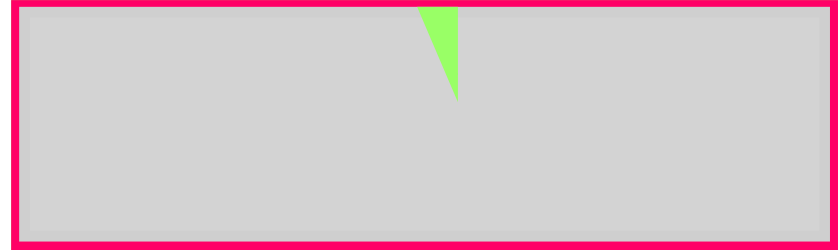
- Water wash removes excess penetrant from the surface of the object being tested
- Care must be taken to remove only surface penetrant and not penetrant from the discontinuities



# Emulsifier Application

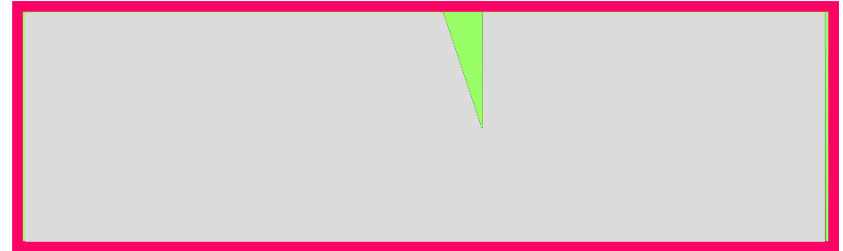
Application: Dip for 30 seconds and agitate at 15 seconds

Emulsifier concentration set at 20% for all products



# Water Rinse

- Water Rinse for 30 sec max at 78-82 °F

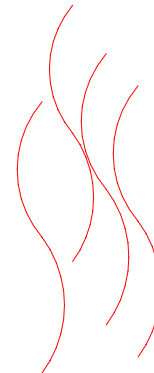
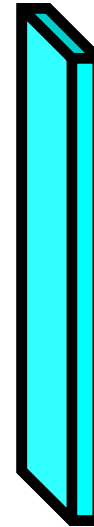


- Water wash removes excess emulsifier from the surface of the object being tested
- Care must be taken to remove only the emulsifier and not the penetrant in the discontinuities



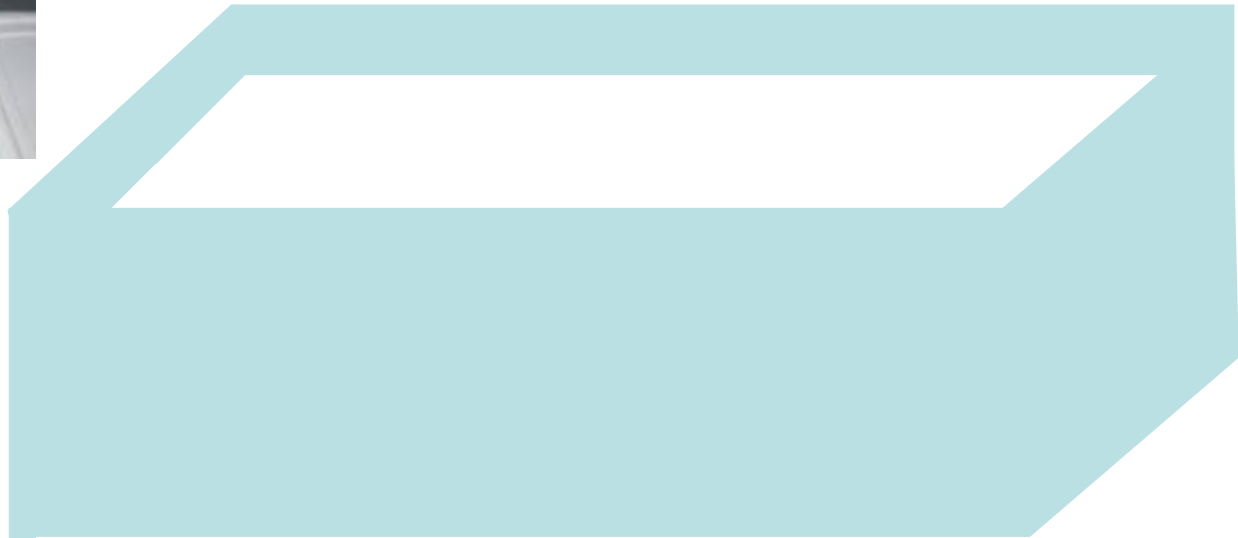
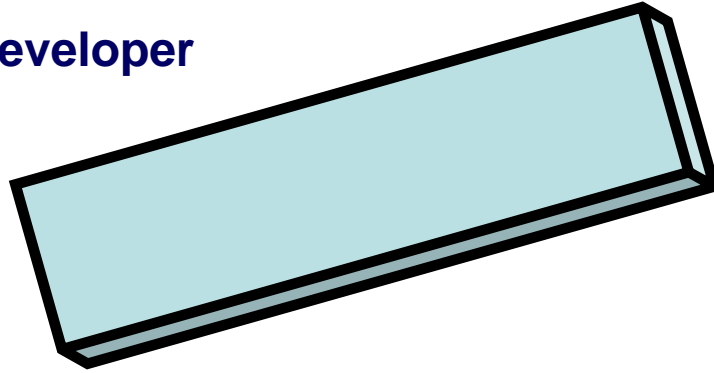
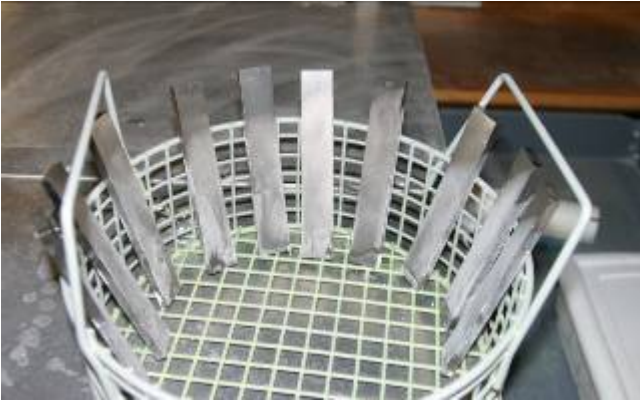
# Drying

- Flash Dry at 140°F (approximately 4 minutes)



# Developer Application

- Application: Dip and Drag in Dry Developer
- Developer Dwell for 10 Minutes

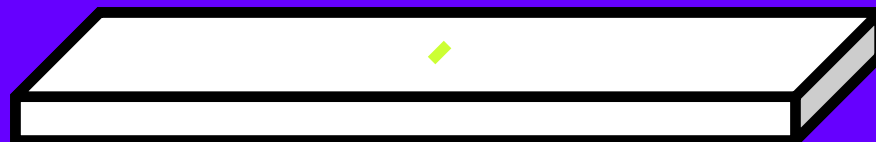
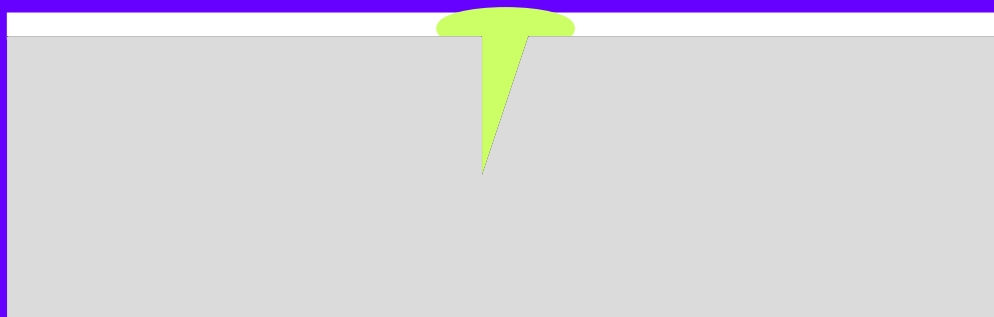




# Developer Application

Developer acts as a blotter by absorbing the penetrant and spreading it throughout the powder around the crack which forms an indication

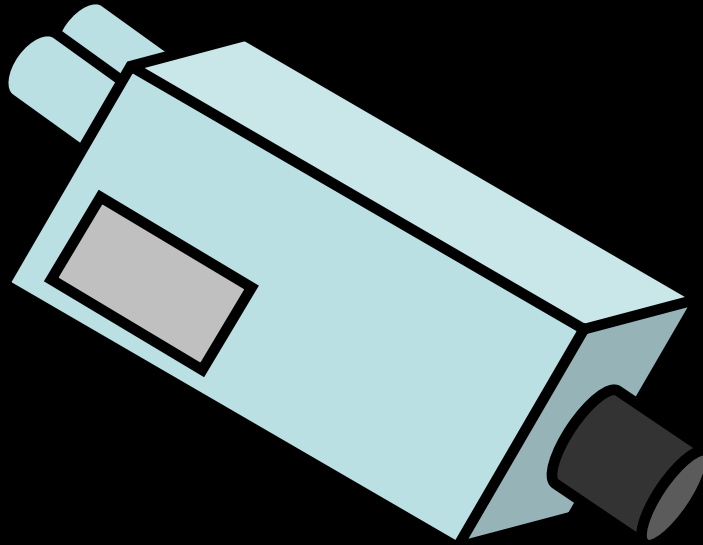
Nonvisible cracks can be detected with this technology



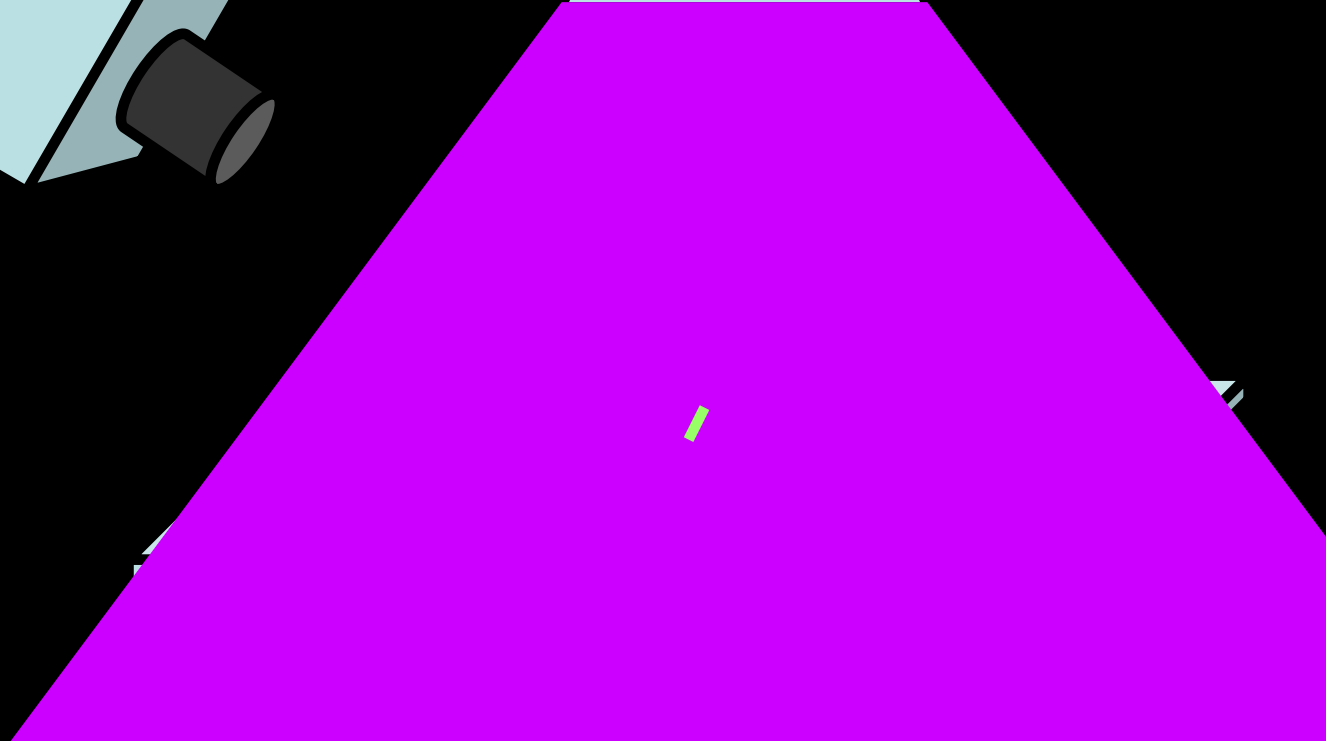
- Blotting is also caused by capillary action
- It is important that the surface is dry so that the developer is spread evenly and does not clump together



# Photometer: Brightness Measurement

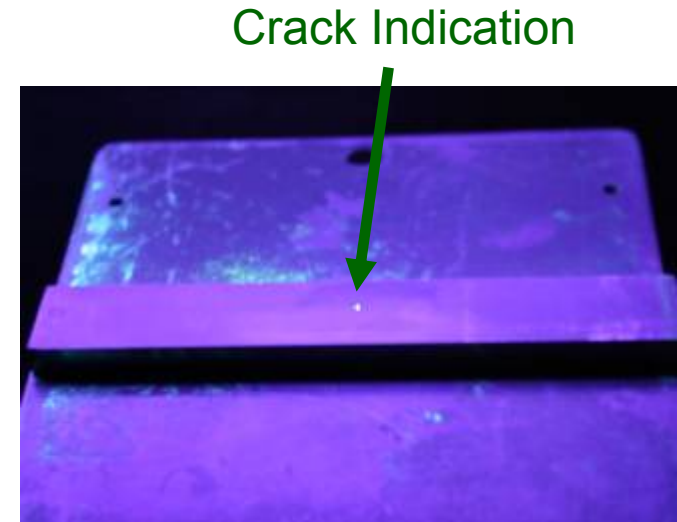


U.V.



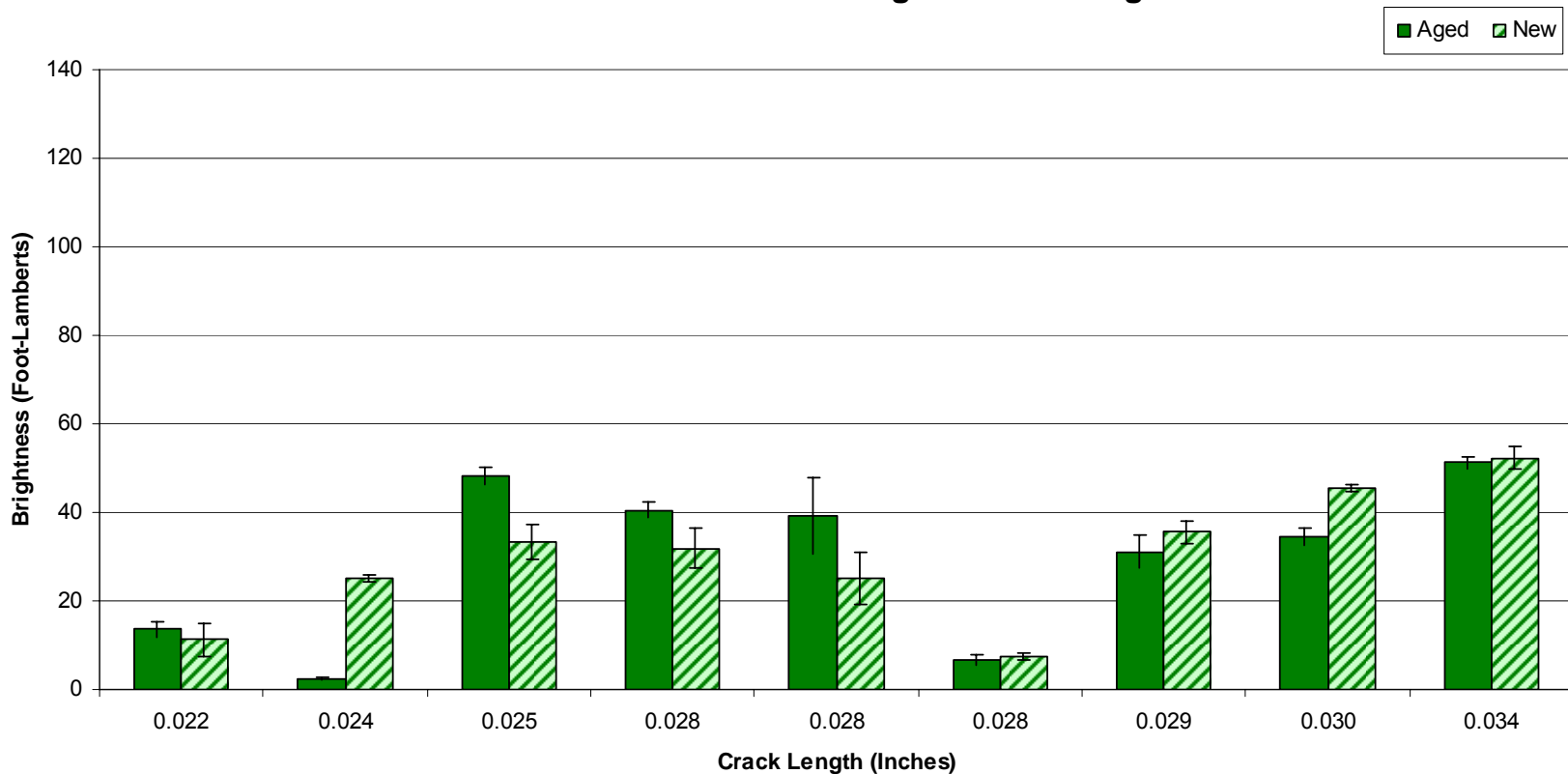
# Spotmeter: Brightness Measurement

- PR-1500 photometer is used to measure the brightness of the FPI indication
- Background brightness measurements typically less than 1 Ft-Lambert
- The higher the brightness value the more likely a field inspector would identify the flaw
- A five run average and standard deviation were calculated for each cracked bar



# Aged vs. New Penetrant Comparison for Cracked Inconel<sup>®</sup> Bars

Manufacturer A, Method D, Level 4  
5 Run Inconel Crack Brightness Average



**Group Average (Aged or New) =  $\sum$  all 9 brightness values shown above and divide by 9**

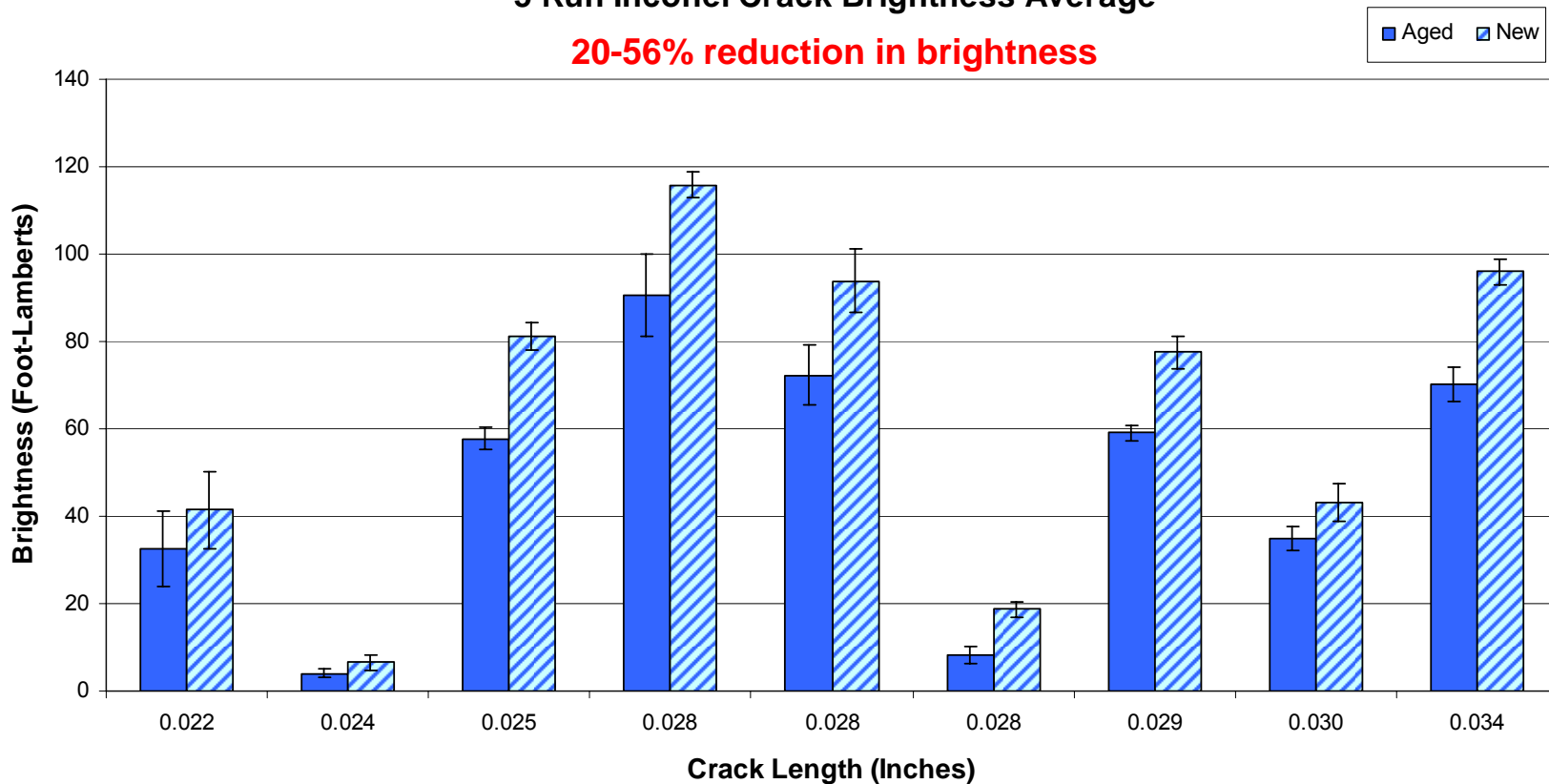
**Group Average Brightness Standard Dev. (Aged or New) =  $\sum$  all 9 standard dev. values and divide by 9**

INCONEL Manufacturer A	Aged	New
Group Average Brightness (Ft-Lamberts)	29.8	29.7
Group Average Brightness Standard Deviation (Ft-Lamberts)	5.0	5.7

# Aged vs. New Penetrant Comparison for Cracked Inconel® Bars

Manufacturer B, Method D, Level 4  
5 Run Inconel Crack Brightness Average

**20-56% reduction in brightness**

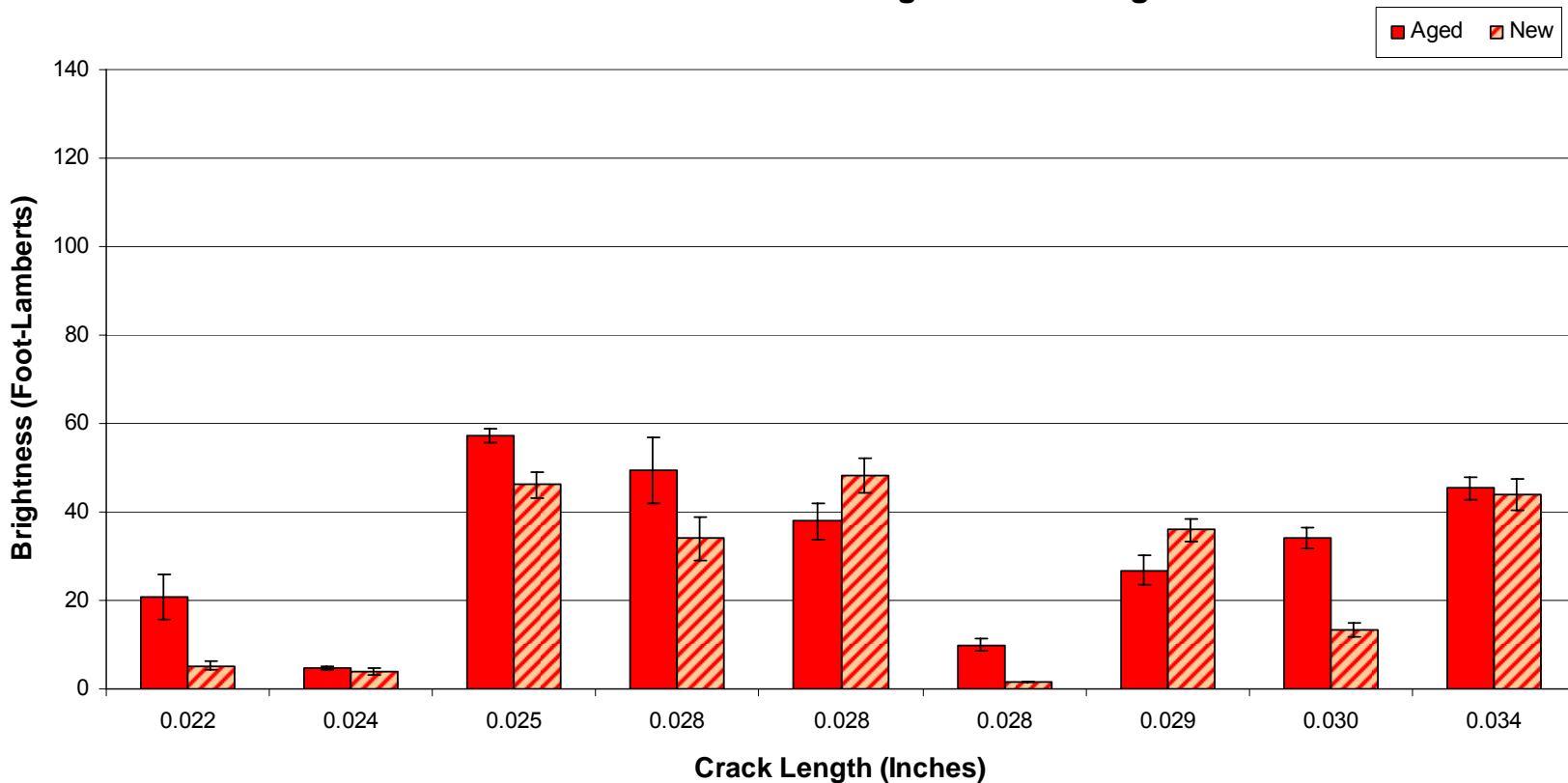


INCONEL Manufacturer B	Aged	New
Group Average Brightness (Ft-Lamberts)	47.8	63.8
Group Average Brightness Standard Deviation (Ft-Lamberts)	8.6	8.2



# Aged vs. New Penetrant Comparison for Cracked Inconel<sup>®</sup> Bars

Manufacturer C, Method D, Level 4  
5 Run Inconel Crack Brightness Average



INCONEL Manufacturer C	Aged	New
Group Average Brightness (Ft-Lamberts)	31.8	25.8
Group Average Brightness Standard Deviation (Ft-Lamberts)	6.2	4.7



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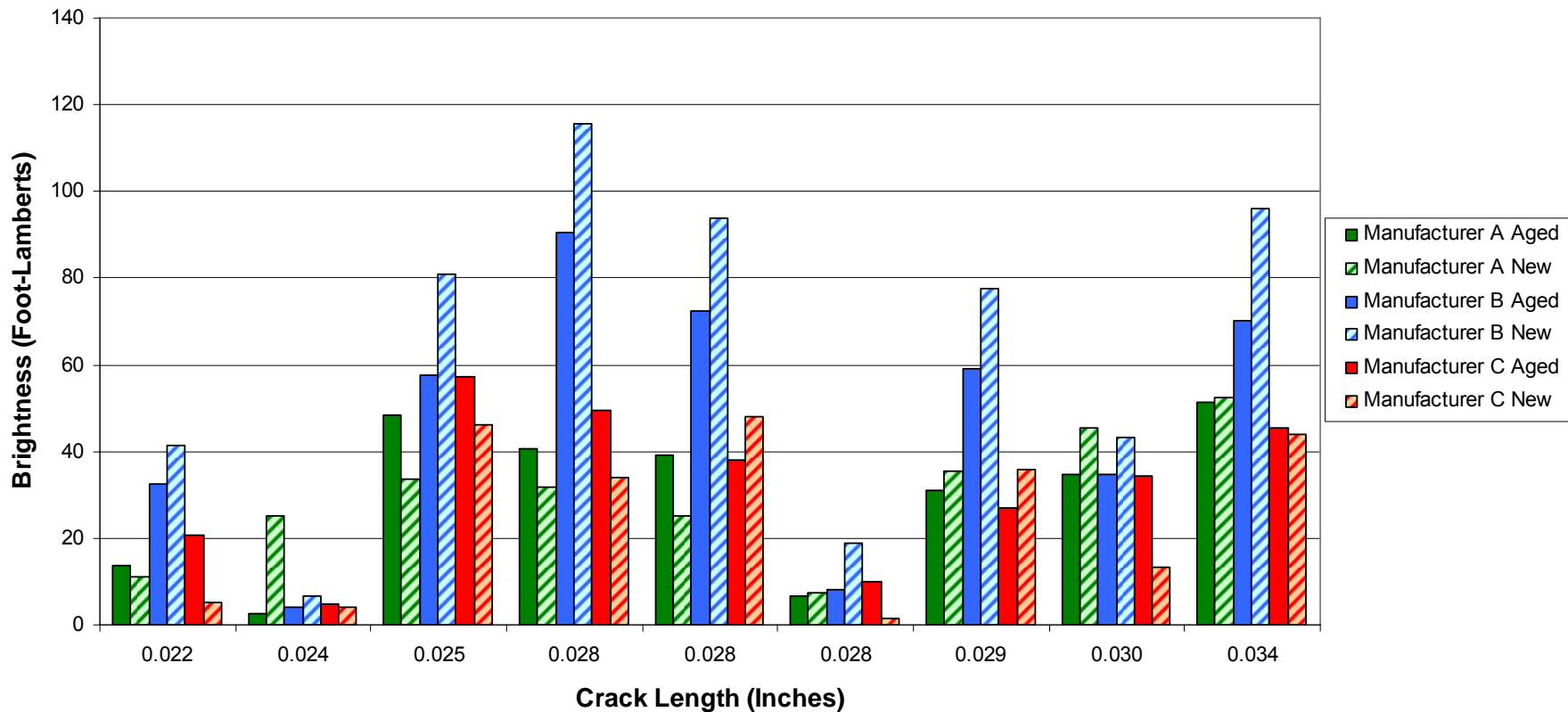


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# Comparison By Manufacturer for Cracked Inconel® Bars

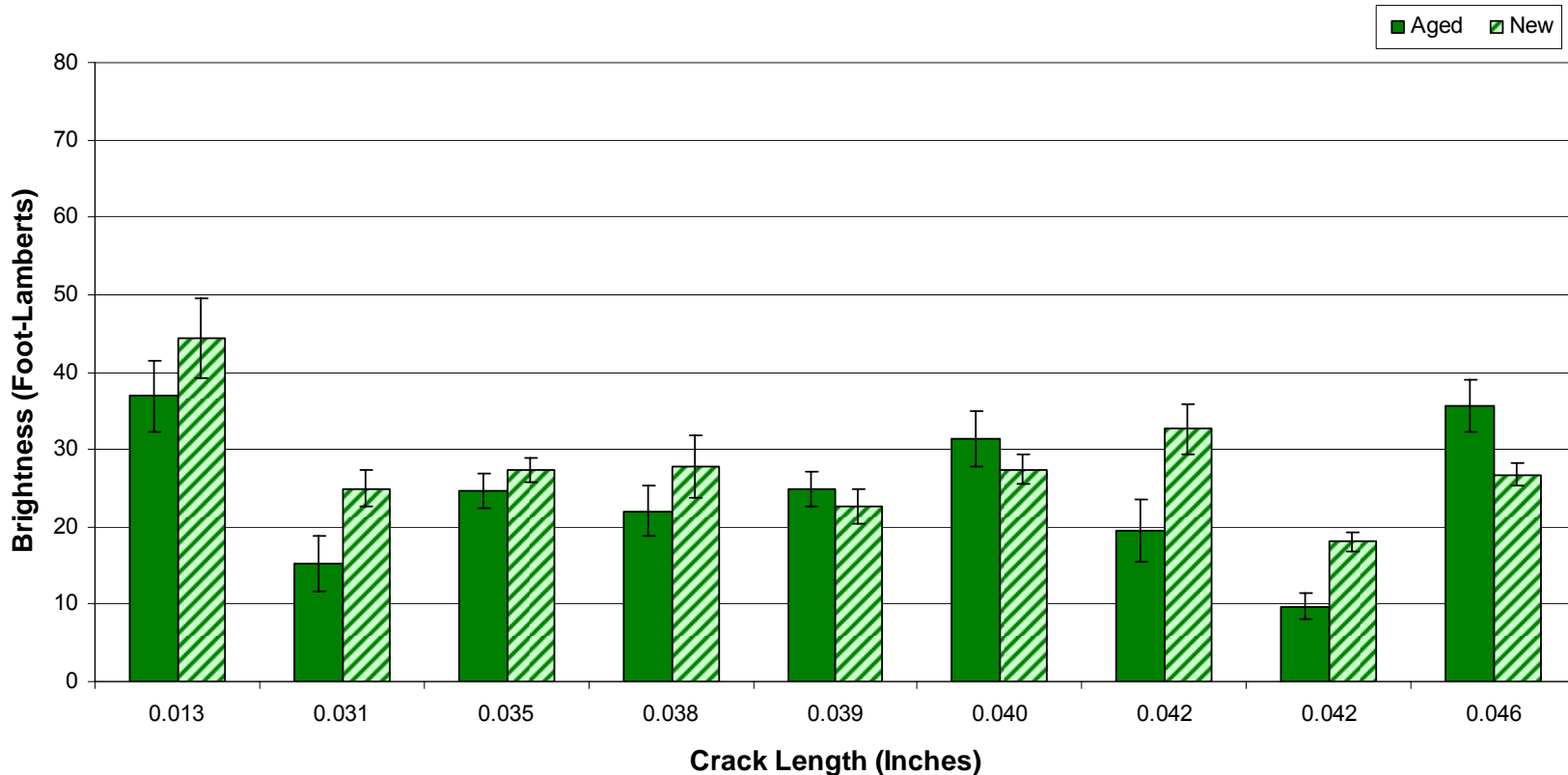
- Manufacturer B Generally had the Highest Crack Brightness Values

All Manufacturers, Method D, Level 4  
5 Run Inconel Crack Brightness Average



# Aged vs. New Penetrant Comparison for Cracked Titanium Bars

Manufacturer A, Method D, Level 4  
5 Run Titanium Crack Brightness Average



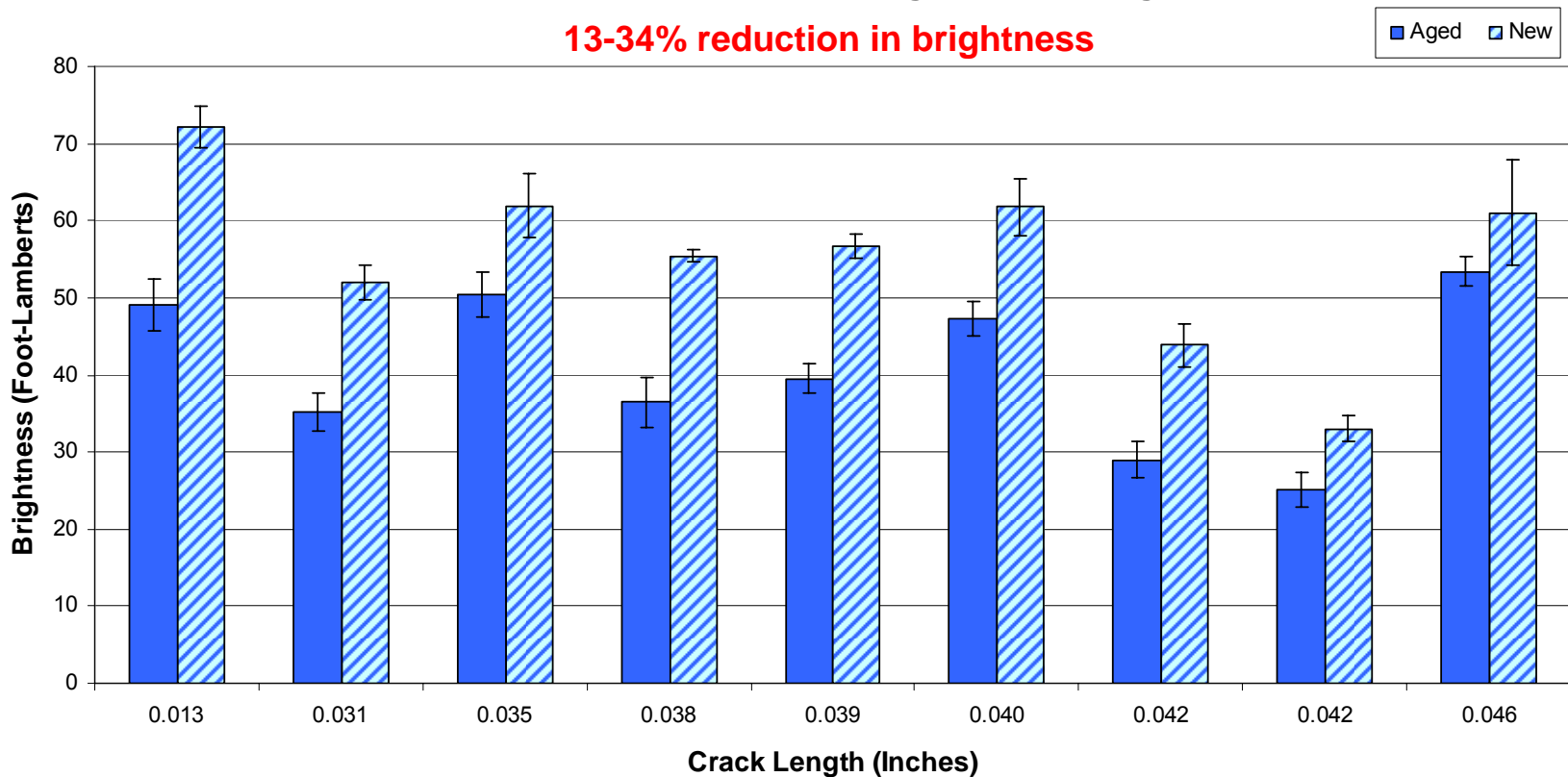
TITANIUM Manufacturer A	Aged	New
Group Average Brightness (Ft-Lamberts)	24.5	28.0
Group Average Brightness Standard Deviation (Ft-Lamberts)	6.4	5.1



# Aged vs. New Penetrant Comparison for Cracked Titanium Bars

Manufacturer B, Method D, Level 4  
5 Run Titanium Crack Brightness Average

**13-34% reduction in brightness**

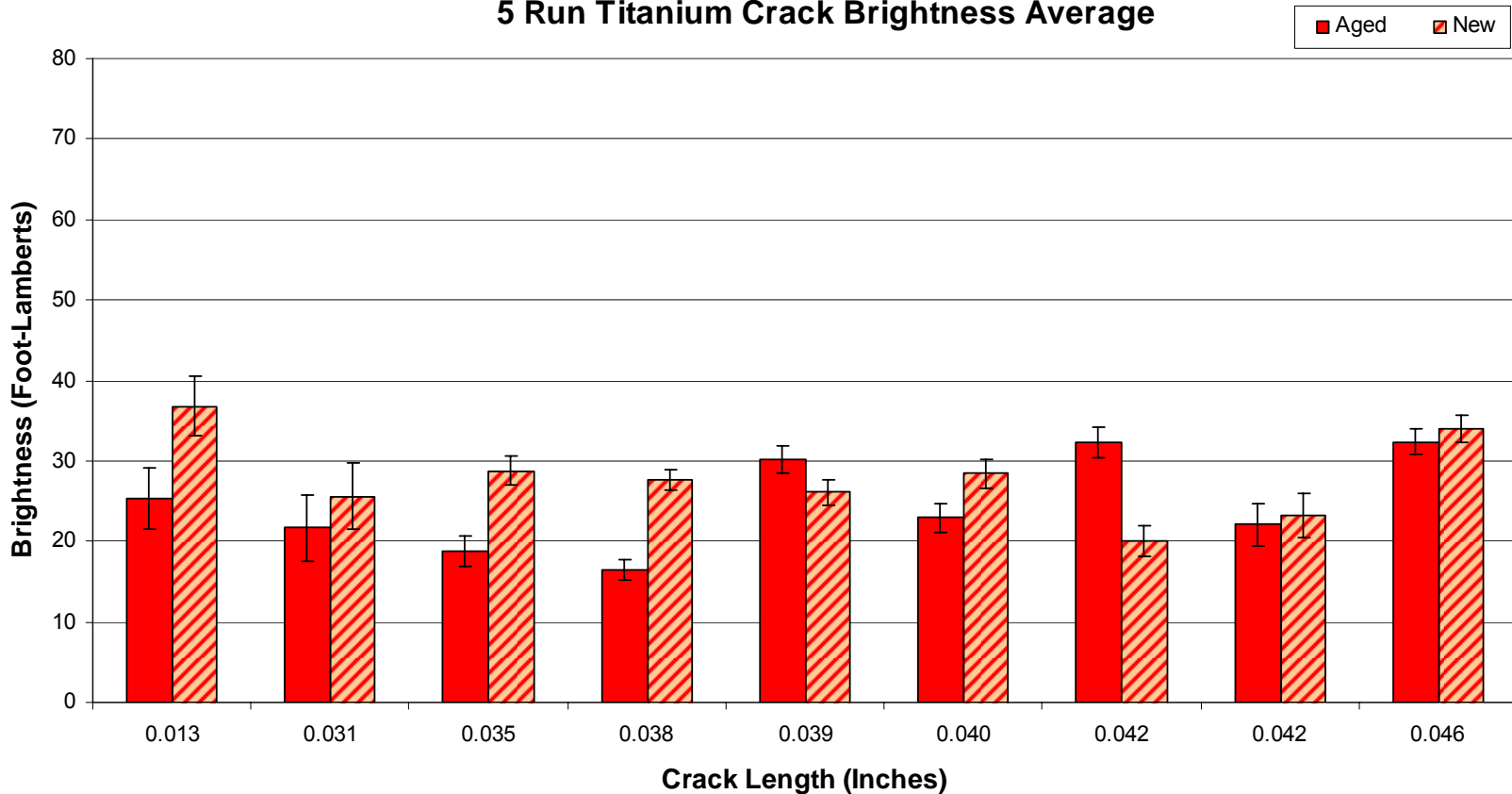


TITANIUM Manufacturer B	Aged	New
Group Average Brightness (Ft-Lamberts)	40.6	55.3
Group Average Brightness Standard Deviation (Ft-Lamberts)	5.0	5.9



# Aged vs. New Penetrant Comparison for Cracked Titanium Bars

Manufacturer C, Method D, Level 4  
5 Run Titanium Crack Brightness Average



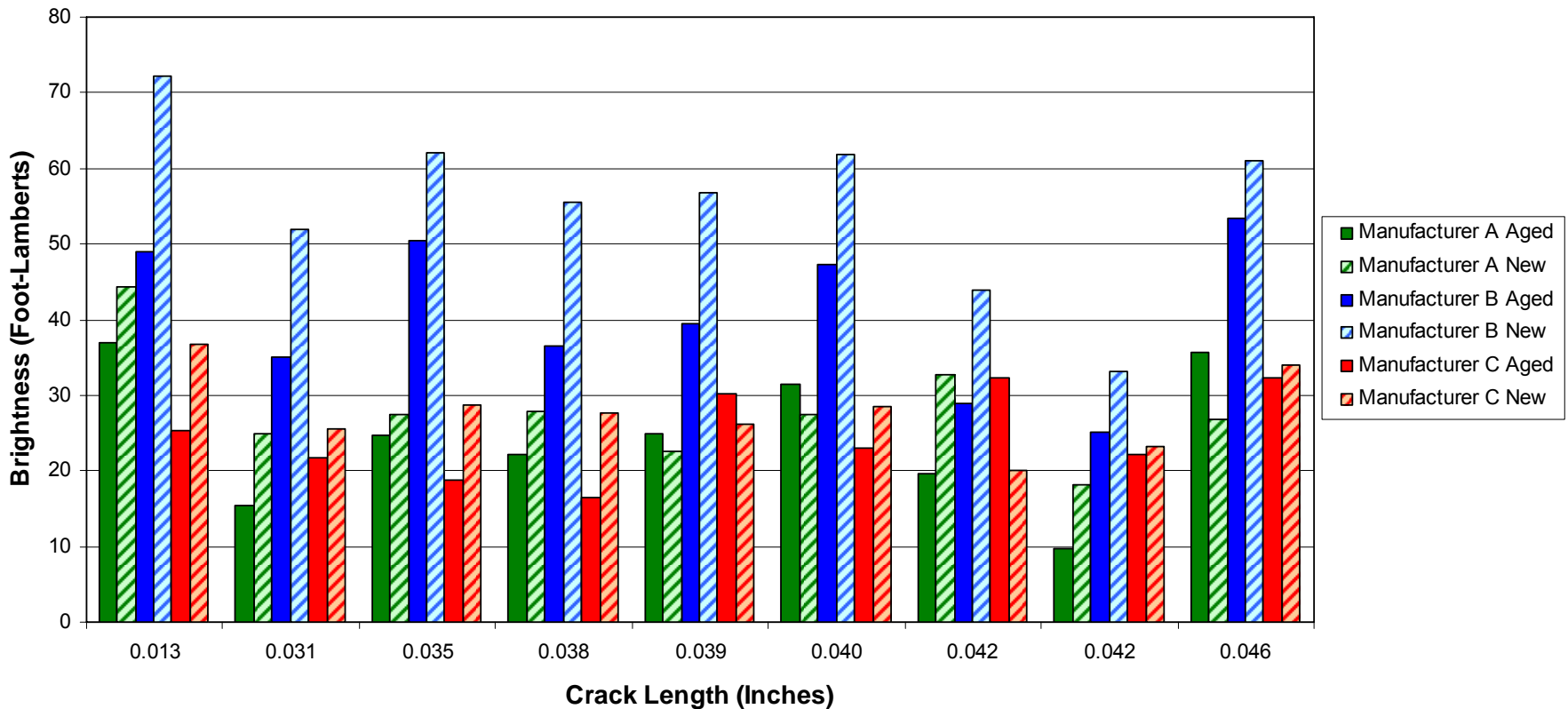
TITANIUM Manufacturer C	Aged	New
Group Average Brightness (Ft-Lamberts)	24.7	27.8
Group Average Brightness Standard Deviation (Ft-Lamberts)	4.6	4.8



# Comparison By Manufacturer for Cracked Titanium Bars

- Manufacturer B Generally had the Highest Crack Brightness Values

All Manufacturers, Method D, Level 4  
5 Run Titanium Crack Brightness Average

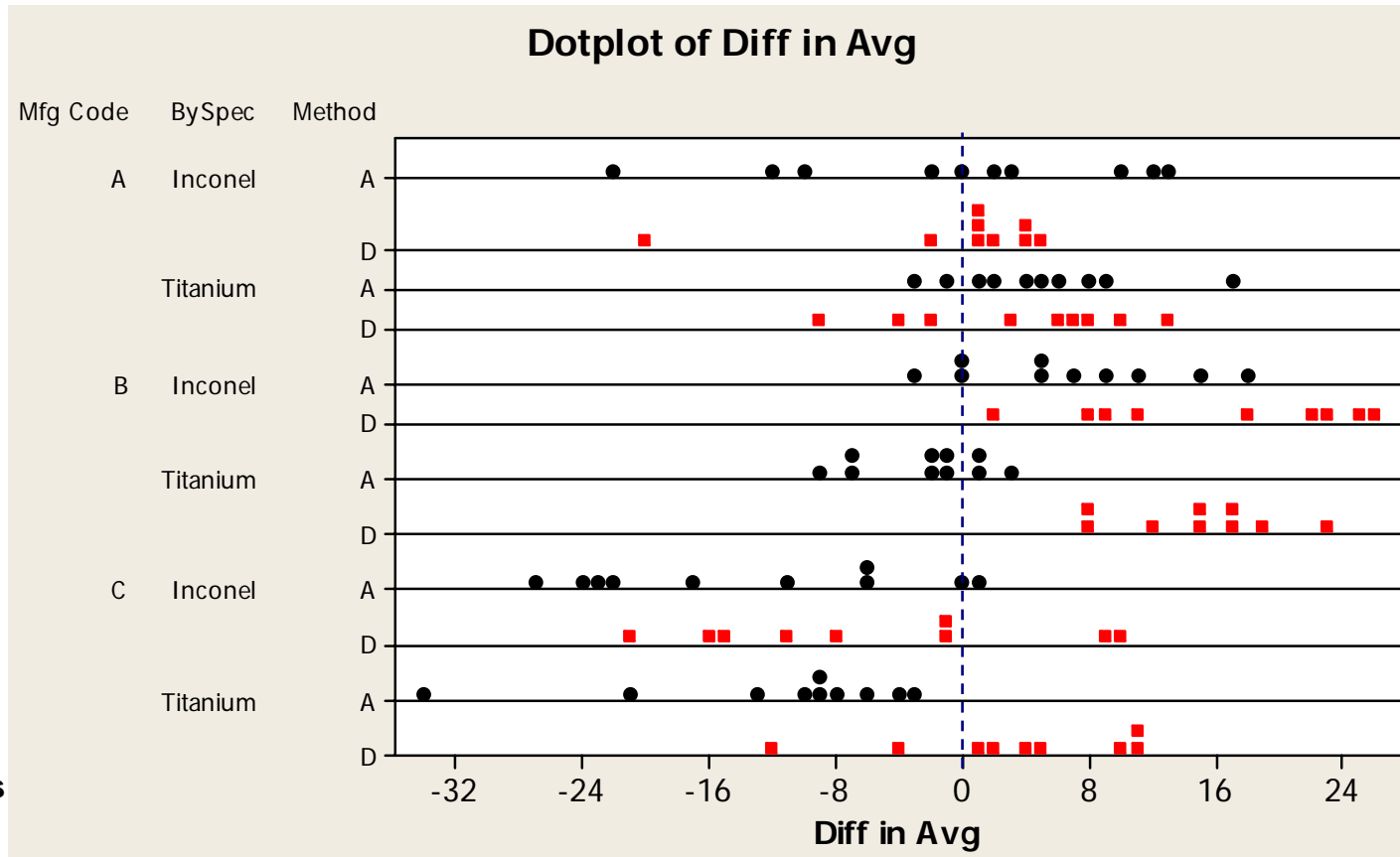


# Statistical Analysis

- The average readings (5 repeats) for each bar with the aged and the new penetrant were differenced (new minus aged) for each manufacturer - specimen combination
- The result was 54 differences (9 bars x 2 materials x 3 manufacturers) which are represented in the dot plot
- A positive difference average would mean “new” brighter than “aged”

Red Dots =  
Method “D” Data

Black Dots =  
Method “A” Data  
2007 Study  
(Previously Published)



# Statistical Analysis

Average of groups with estimate of pooled standard error of mean are given below (Standard error of mean uses pooled estimate of variance)

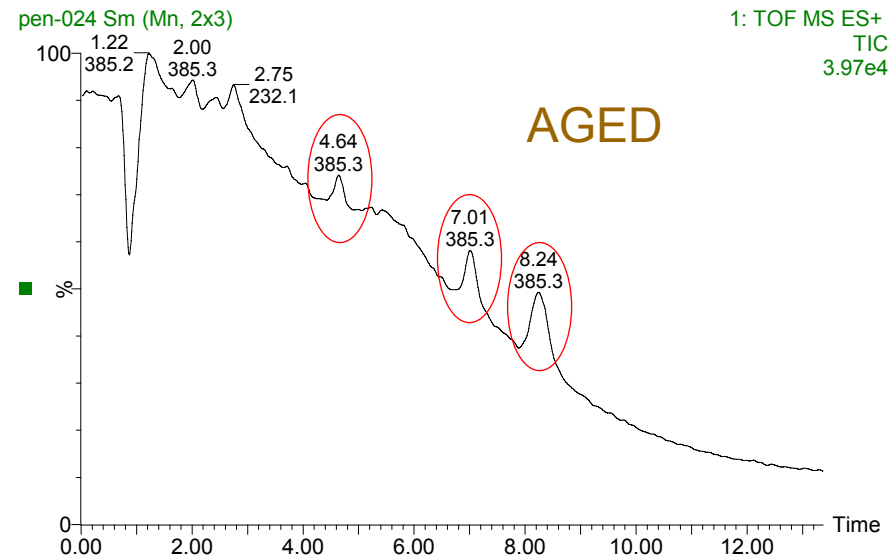
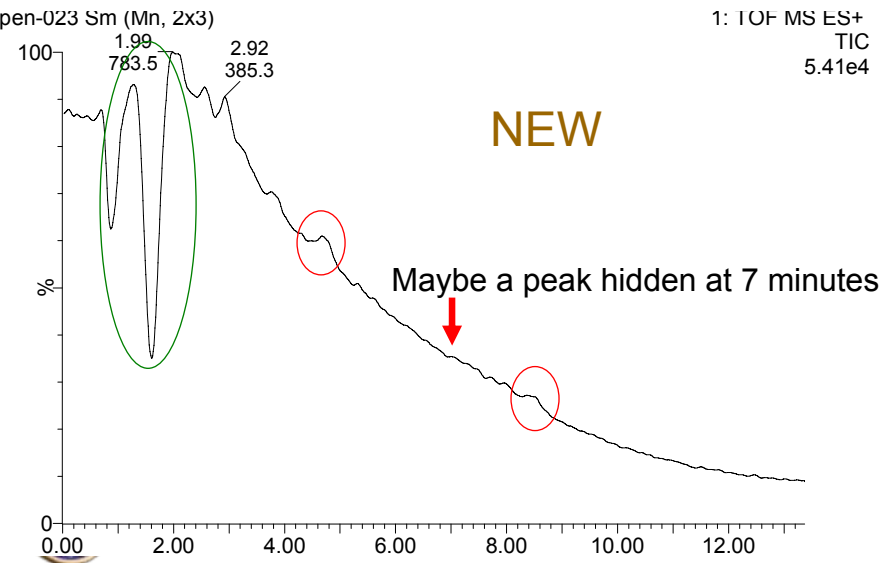
- The question of no difference between new and old is now a test that the mean difference is = 0
- The last column of the table gives the significance level associated with the hypothesis of mean difference = 0
  - The two instances of Manufacture B show a high significance level, as the significance level is less than 0.0005 (“new” brighter than “aged”)
  - The two instances of Manufacture C show a high significance level, as the significance level is less than 0.0005 (“aged” brighter than “new”)

Statistical Parameters for Method D Study				
Specimens	Manufacturer	Mean $\hat{\mu}$	SE Mean	Significance level $\mu = 0$
Inconel	A	-0.542	2.687	0.841
Titanium	A	3.527	2.687	0.196
Inconel	B	16.027	2.687	0.000
Titanium	B	14.727	2.687	0.000
Inconel	C	-6.049	2.687	0.029
Titanium	C	3.131	2.687	0.250

Statistical Parameters for Method A Study				
Specimens	Manufacturer	Mean $\hat{\mu}$	SE Mean	Significance level $\mu = 0$
Inconel	A	-0.6	2.656	0.822
Titanium	A	4.71	2.656	0.081
Inconel	B	6.77	2.656	0.013
Titanium	B	-2.57	2.656	0.337
Inconel	C	-13.49	2.656	0.000
Titanium	C	-11.77	2.656	0.000

# Chemical Analysis

- Ultra high pressure liquid chromatography revealed that there are at least 2 compounds that formed from the aging of the Manufacturer B's penetrant
- This has been determined by the appearance of noticeable peaks in the mass chromatograms
- It appears that there may be trace amounts of these compounds in the new sample as well (at 4.6 and 8.2 minutes)
- Further testing is needed to confirm that they are the same and to determine the identity of these extra compounds





# Conclusions

## Method D, Level IV, Penetrant Systems

- The oldest penetrant system tested in this study came from Manuf. B and significantly lower crack brightness levels (13-56% lower) were observed compared to crack brightness levels obtained using the new Manuf. B penetrant system
- A distinct visible color difference for “aged” Manuf. B penetrant vs. “new” Manuf. B penetrant was observed (photograph on slide 4)
- Mass chromatography showed that at least two compounds formed in the aged Manuf. B penetrant (these compounds were either not present or only present in trace amounts in the new Manuf. B penetrant)
- While the aged brightness levels were lower for Manuf. B, they were generally much higher when compared to Manuf. A or C for both aged and new Method D penetrant systems





# Conclusions

## Method A, Level IV, Penetrant Systems

- No strong evidence that the use of aged Method A, Level 4 penetrant systems would automatically lead to reduced likelihood of crack detection
- While aging was not observed we found that new Manuf. C penetrant gave significantly lower crack brightness levels compared to crack brightness levels obtained using aged Manuf. C penetrant systems
- Similar to the Method D study, we observed differences in average brightness levels “aged” and “new” when compared by manufacturer, and Manuf. A generally had the highest crack brightness levels for Method A penetrant systems
- Both studies do not account for possible batch-to-batch variation that existed at time of manufacture (old or new) – nor possible batch-to-batch differences in aging characteristics of old material. (In this experiment the old material was stored in a controlled environment and had never been opened)





# Future Work

- Determining the identity of the extra compounds in the aged penetrant may shed some light on the aging mechanism
- Testing of penetrant samples currently at OEMs
  - Some OEMs replenish or add penetrant to their tanks as the penetrant level gets low (tanks are not always drained), potentially leaving opened penetrant that can age
- The AANC could repeat this study in another 5-10 years using the aged penetrant materials tested in this effort

## References:

DiMambro, J., Koech, I.K., Moore, D.G., Spencer F.W., "Investigating the Performance of Aged Fluorescent Penetrant Materials," Materials Evaluation Volume 65 #8, August, 2007

## Acknowledgements:

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