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Comparative Mirror Cleaning Study

"A Study on Removing Particulate Contamination"

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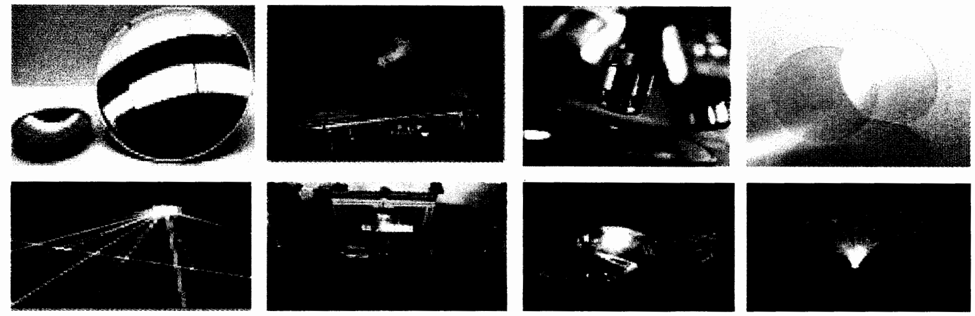
Outline

- **Introduction**
  - Optical Devices
  - Contamination Effects of Optical Surfaces
- **Experimental Design**
  - Test Plan
  - Cleaning Procedures
  - Verification Instrument
- **Design of Experiments (DOE) Software**
  - Jump/Statistical Analytical Software (JMP/SAS)
- **Results**
- **Conclusions**
- **Recommendations**
- **Future Work**

Introduction

Optical Devices

- Mirrors and telescopes
- Microscope and lenses
- Lasers and interferometers
- Prisms and optical filters



Optical Industry

The cleanliness of optical surfaces is recognized as an industry wide-concern for performance of optical devices:

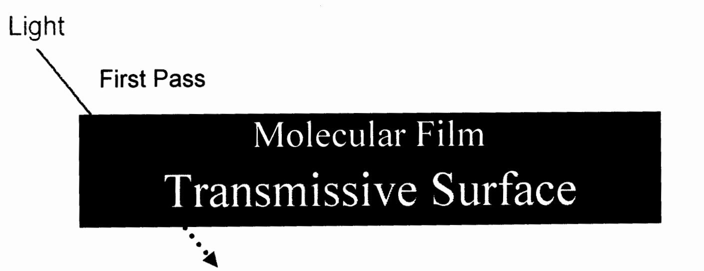
- No established standard for optical cleaning
- No standard definition of a "clean" optical element

Advantages of Experimental Study…

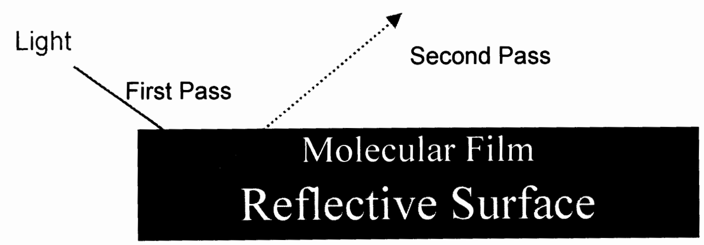
- It evaluates the effectiveness of commonly used optical cleaning techniques based on wafer configuration, contamination levels, and the number and size of removed particles
- The results can help ensure mission success to flight projects developed for the NASA Origins Program (JWST, SAFIR, etc.)

Contamination Effects

1. Molecular Contamination: Accumulation of submicron particles (i.e. Water, hydrocarbons, and silicones)



a. Absorptive Effects (Transmissive Surface)



b. Absorptive Effects (Reflective Surface)

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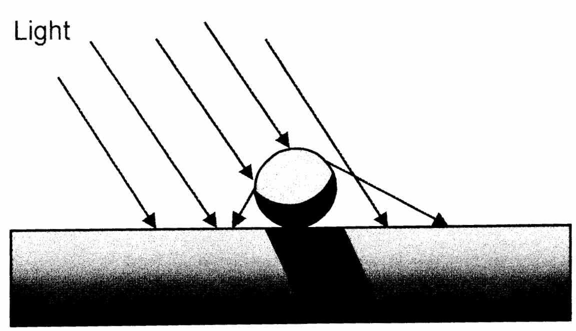
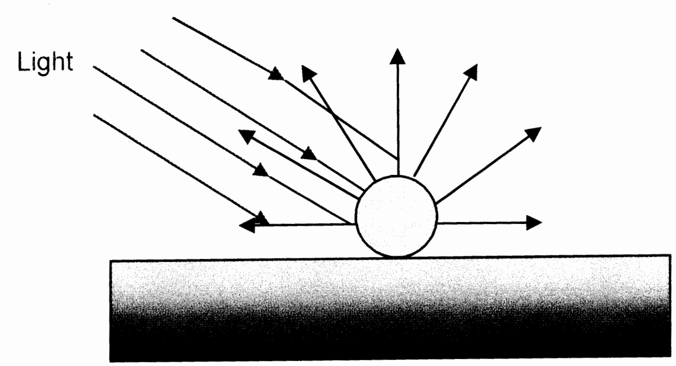
2. Particulate Contamination: Conglomerate of visible sized particles (e.g. Dust)



a. Obscuration Effects



b. Scattering Effects

Test Plan

Experimental Objective:

To compare the effectiveness in removing particulate contamination from coated and uncoated silicon wafers with commonly used optical cleaning methods

Technical Objectives:

- Determine the cleaning ability of each method based on the number and size of removed particles
- Assess the risk of surface damage for each cleaning procedure
- Evaluate each method as a function of its initial contamination level ("fairly clean", "dirty, "very dirty")

Experimental Process:

- Contaminate wafers
- Characterize surface (Measure and count number of particles)
- Clean wafers
- Characterize surface

Experimental Design

Cleaning Methods

- Detergent Bath
- Solvent Rinse
- CO_2 Snow Cleaning

Wafer Configuration

- (12) - Silicon (Si)
- (12) - Gold coated silicon wafer (Si+Au)
- (12) - Gold coated silicon wafer with a silicon oxide coating (Si+Au+SiOx)

Table 1: Wafer Specifications

Wafer Properties	
Thickness	500 µm
Diameter	4″
Coating Thickness	
Gold Coating	2000 Å
Silicon Oxide Coating	1000 Å

Exposure Times

- 1 day, 3 days, and 5 days (Building 7 Highbay)

Design of Experiments (DOE) Software

Sample Size (Total Sample Size = 36 wafers)

- Calculated by specifying a 95% Upper Confidence Level
  - 13 DOF: Number of values in final calculation that are free to vary

Randomization Table

- Randomly paired cleaning methods with wafer configurations and exposure time
- Divided into 9 blocks
  - Each block has 4 wafers

Table 2: Example of Block Format

Cleaning Method	Wafer Configuration	Exposure Time	Y (Response)
Rinse	Si+Au+SiOx	3	•
CO2	Si+Au	5	•
Bath	Si+Au+SiOx	1	•
Bath	Si	5	•

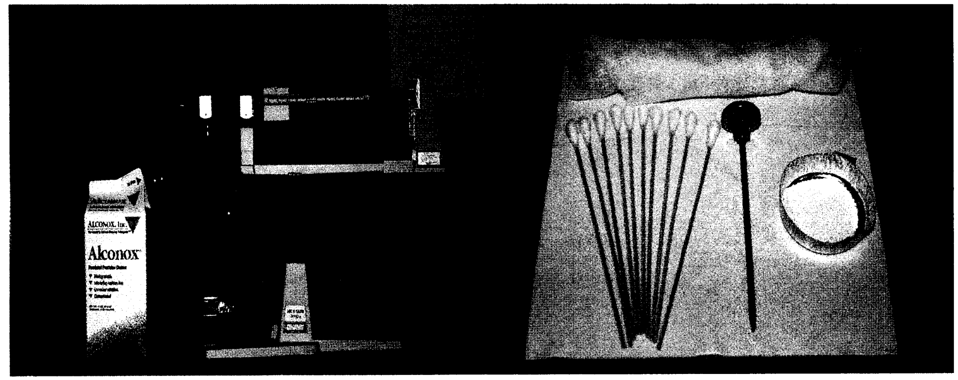
JMP/Statistical Analytical Software

- Simultaneously compares input variables

Mirror Cleaning Procedure (1)

Detergent Bath

Direct contact method that uses an aqueous based, nonionic detergent to remove contamination

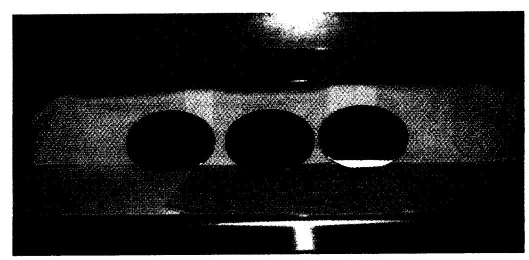


Pre-Clean Preparation[1]:

1. Alconox Solution
   a. 5 grams of Alconox detergent
   b. 4 cups of distilled water
2. Rinse Solution - 2 cups of distilled water at 120 °F

Cleaning Process:

1. Wafer submerged in Alconox Solution
2. Q-tip placed directly on wafer at a 20°; Surfaces cleaned using a multi-directional wiping technique for 1 minute
3. Wafer rinsed 10x's in distilled water
4. Water vertically positioned (at a 10-15° angle) for drying (~15 min)



[1] NASA/GSFC Optical Component Cleaning (551-WI-8072.1.7B)

Mirror Cleaning Procedure (2)

Solvent Rinse

Direct contact method that uses an aqueous based, nonionic detergent and an acetone rinse to remove contamination

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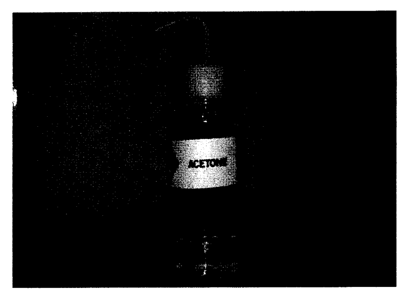

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Cleaning Process:

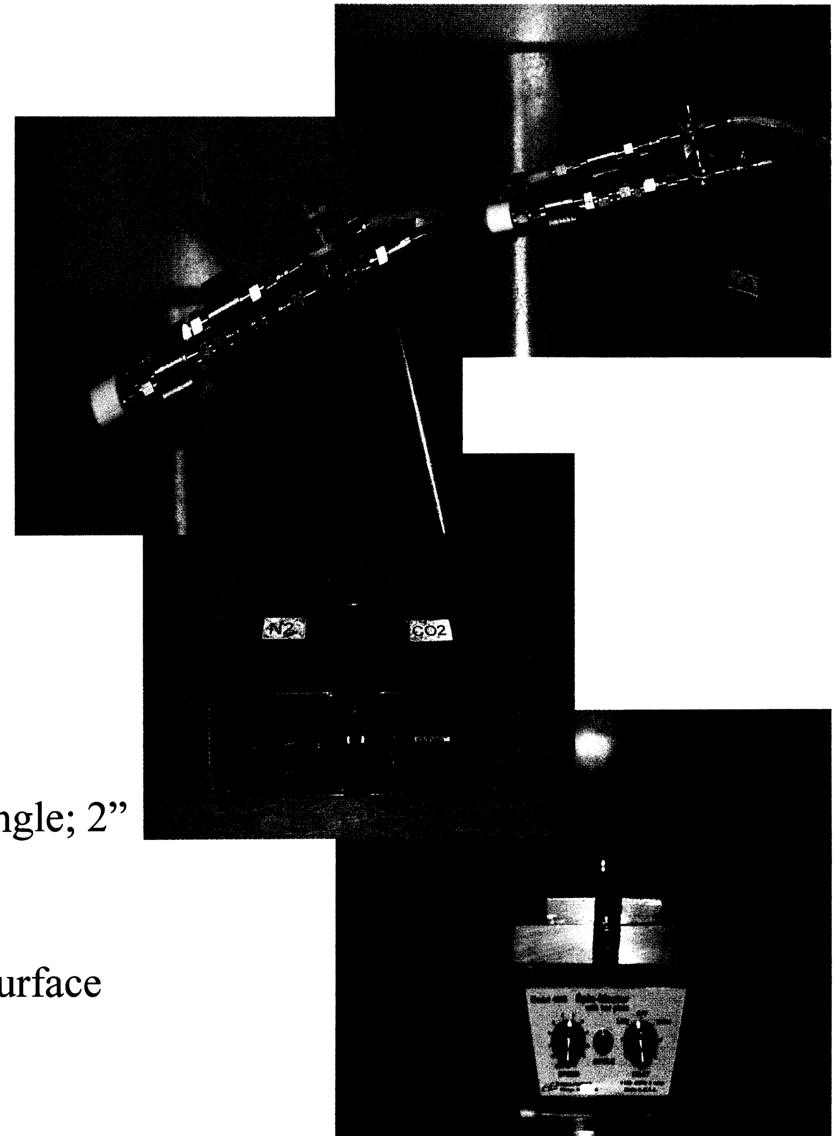
1. Wafer submerged in Alconox Solution
2. Q-tip placed directly on wafer at a 20° angle; Surfaces cleaned using a multidirectional wiping technique for 1 minute
3. Wafer rinsed 10x's in distilled water
4. Water vertically positioned (at a 10-15°angle) for drying
5. Wafer rinsed with 4fl. oz of IPA grade acetone in a top-bottom, left-right pattern

[1] NASA/GSFC Optical Component Cleaning (551-WI-8072.1.7B)

Mirror Cleaning Procedure (3)

CO_2 Snow Cleaning

Non-contact method that uses a high velocity stream of CO_2 gas and snow pellets to remove contamination



Pre-Clean Preparation:

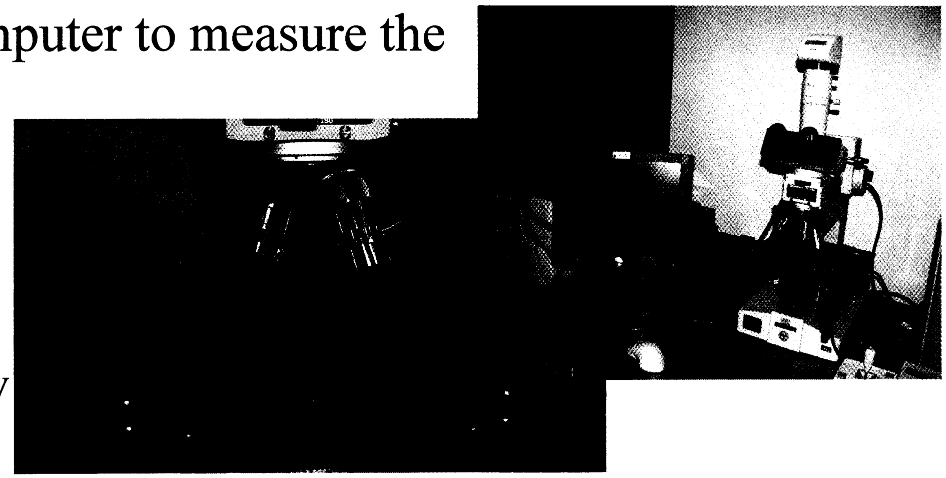
1. Place vacuum chuck on hot plate; turn hotplate on "High" setting (120 °F). Let warm-up for 15-20 minutes

Cleaning Process:

1. Place wafer on vacuum chuck; power on motor
2. Open pressure valve on CO_2 cylinder tank
3. Position nozzle at the upper right hand corner (30° angle; 2" from surface)
4. Open CO_2 circuit using footswitch
5. Clean entire wafer surface using 7 vertical strokes; surface cleaned in a top-bottom, right-left pattern

Verification - Image Analysis

Image Analysis (IA) is a verification instrument that incorporates the use of a microscope, camera, and computer to measure the size, shape, and number of particles.



IA Specifications:

- Leica camera/CCD
- Olympus microscope (5X Obj; 50 Mag)
  - Detects 0.3μm particles at 95% certainty
- Robotic stage

Facility Specifications:

- Building 84 Cleanroon
- Class 10,000
- Avg. RH: 44%
- Average Temp: 69 °F
- Laminar Flow: 135-150 ft/min

$$PAC = \frac{\text{Total Area of Particles}}{\text{Total Surface Area}} \times 100$$

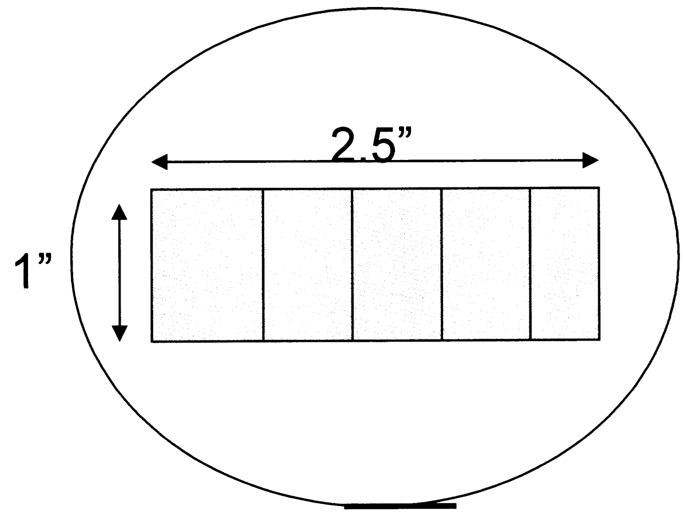


Figure 1: IA Reading Area

Results – Detergent Bath

Table 3: PAC Removal Percentage of Detergent Bath Samples

Wafer Configuration	Exposure Time	PAC (Before Cleaning)	PAC (After Cleaning)	Efficiency Removal %
Si	5	0.23924	0.08072	66
Si+Au	3	0.09553	0.01502	84
Si+Au+SiOx	5	0.09312	0.01560	83
Si+Au+SiOx	1	0.08290	0.01974	76
Si	5	0.07863	0.03506	55
Si+Au	3	0.06028	0.02268	62
Si	3	0.04832	0.04448	8
Si+Au+SiOx	3	0.04033	0.05241	----
Si+Au+SiOx	1	0.02070	0.01017	51
Si	1	0.01020	0.01460	----
Si+Au	1	0.00454	0.01077	----
Average Removal %				**61**

Particle Count – Detergent Bath

Table 4: Particle Count for Sample B5_23_Si.Au_3D (67% PAC Reduction)

Range of Particle Sizes (microns)	Total # of Particles (Before Cleaning)	Total # of Particles (After Cleaning)	Removal Efficiency (%)
$0 \leq x \leq 1$	0	0	0
$1 \leq x \leq 5$	54	1770	-
$5 \leq x \leq 10$	482	782	-
$10 \leq x \leq 25$	656	326	50
$25 \leq x \leq 50$	542	231	57
$50 \leq x \leq 100$	420	29	93
$100 \leq x \leq 150$	211	2	99
$150 \leq x \leq 250$	235	0	100
$250 \leq x \leq 500$	245	0	100
$500 \leq x \leq 750$	125	0	100
≥ 750	146	1	99
Total No. of Particles	**3116**	**3141**	-
PAC (Cleanliness Lvl)	**0.06 (375)**	**0.02 (300)**	**67**

Particle Distribution – Detergent Bath

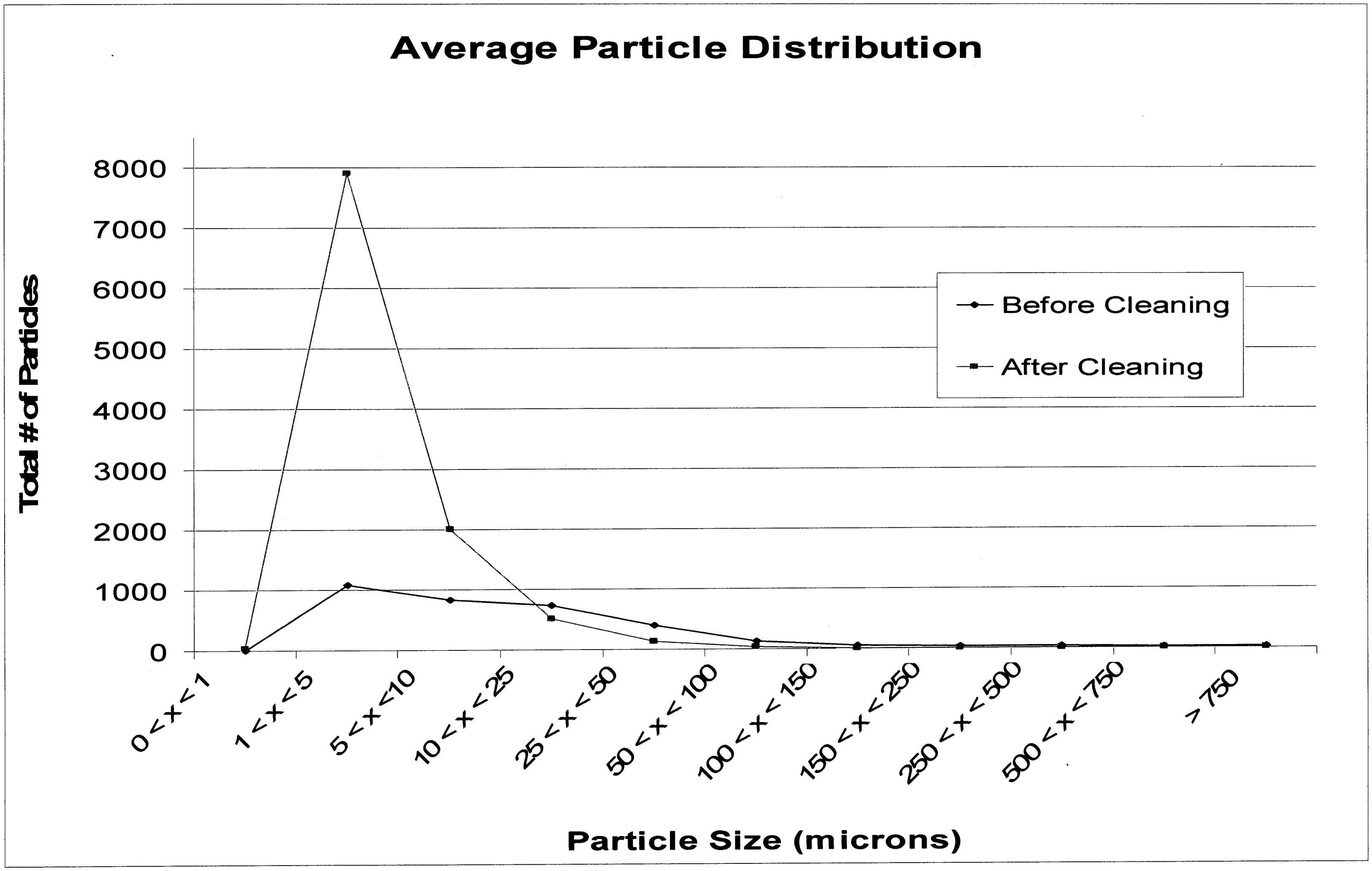


Figure 2: Average Particle Distribution for the Detergent Bath Samples

Results – Solvent Rinse

Table 5: PAC Removal Percentage of Solvent Rinse Samples

Wafer Configuration	Exposure Time	PAC (Before Cleaning)	PAC (After Cleaning)	Efficiency Removal %
Si+Au	5	0.12063	0.00815	93
Si+Au+SiOx	3	0.08629	0.02234	74
Si+Au	5	0.07477	0.01800	76
Si	5	0.06177	0.00013	100
Si	3	0.06071	0.00718	88
Si+Au	3	0.04662	0.01021	78
Si+Au+SiOx	5	0.04414	0.01301	71
Si+Au+SiOx	5	0.04271	0.00171	96
Si	1	0.03862	0.03458	10
Si+Au	3	0.03226	0.00582	82
Si+Au+SiOx	1	0.02965	0.01254	58
Si+Au+SiOx	1	0.01209	0.00497	59
Average Removal %				**74**

Particle Count – Solvent Rinse

Table 6: Particle Count for Sample B1_5_Si.Au.SiOx_3D (74% PAC Reduction)

Range of Particle Sizes (microns)	Total # of Particles (Before Cleaning)	Total # of Particles (After Cleaning)	Removal Efficiency (%)
0 ≤ x ≤ 1	0	0	0
1 ≤ x < 5	2026	2596	-
5 ≤ x ≤ 10	1409	660	53
10 ≤ x ≤ 25	995	278	72
25 ≤ x ≤ 50	418	58	86
50 ≤ x ≤ 100	119	33	72
100 ≤ x ≤ 150	13	11	15
150 ≤ x ≤ 250	12	11	8
250 ≤ x ≤ 500	14	12	14
500 ≤ x ≤ 750	6	2	67
≥ 750	8	4	50
Total No. of Particles	**5020**	**3665**	-
PAC (Cleanliness Lvl)	**0.086 (415)**	**0.022 (310)**	**74**

Particle Distribution – Solvent Rinse

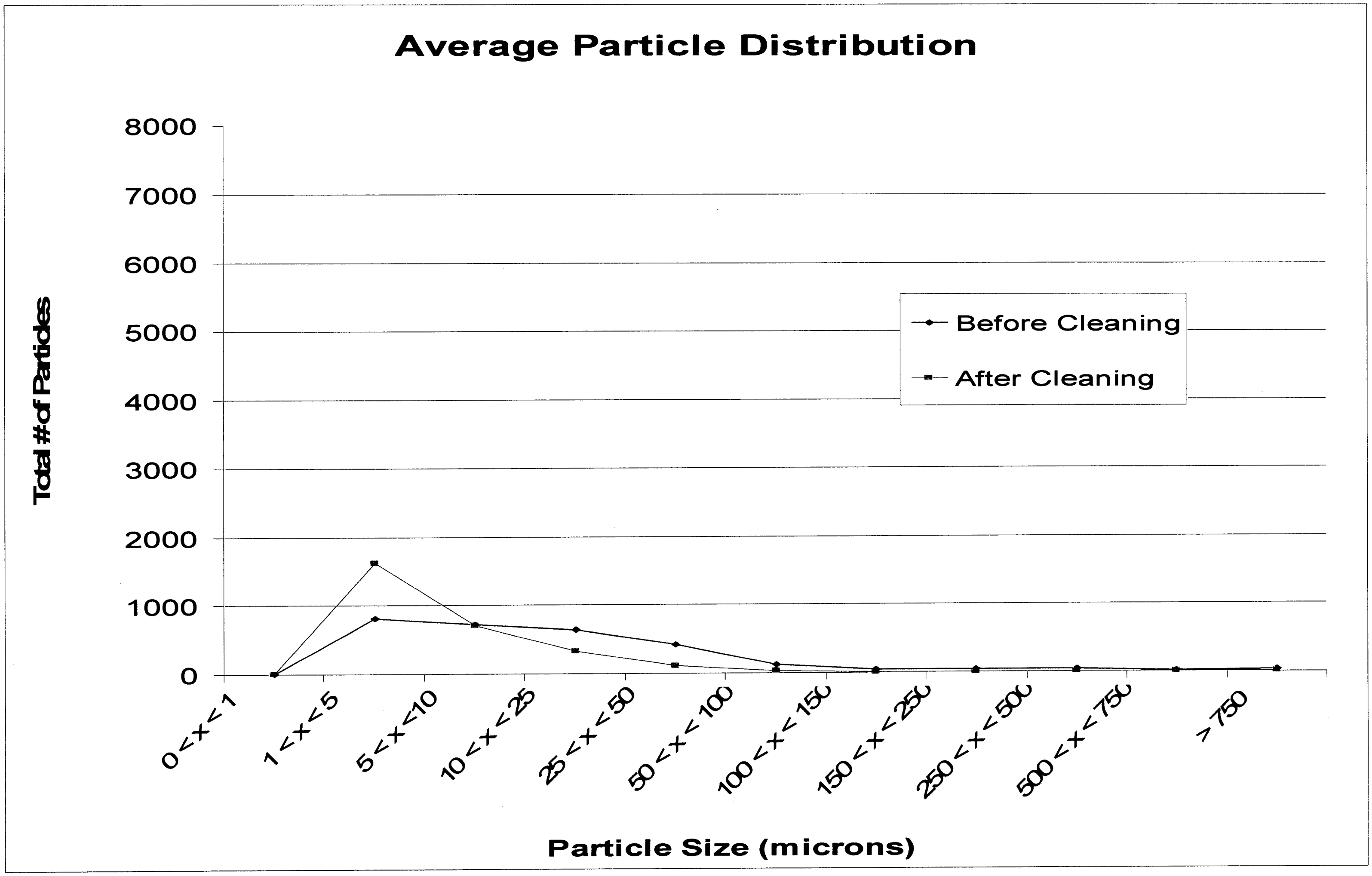


Figure 3: Average Particle Distribution for the Solvent Rinse Samples

Results – CO_2 Snow Cleaning

Table 7: PAC Removal Percentage of CO_2 Snow Cleaning Samples

Wafer Configuration	Exposure Time	PAC (Before Cleaning)	PAC (After Cleaning)	Efficiency Removal %
Si+Au	5	0.19466	0.00716	74
Si+Au+SiOx	5	0.07471	0.00555	93
Si	3	0.06550	0.00261	96
Si+Au	1	0.06086	0.00414	93
Si+Au+SiOx	1	0.03851	-	-
Si+Au	5	0.03823	0.00554	86
Si+Au+SiOx	3	0.03606	-	-
Si	5	0.03522	-	-
Si+Au+SiOx	3	0.03380	-	-
Si+Au	1	0.01034	0.00284	73
Si	3	0.01139	-	-
Si	1	0.00317	-	-
Average Removal %				**86**

Particle Count – CO_2 Snow Cleaning

Table 8: Particle Count for Sample B4_19_Si.Au_5D (86% PAC Reduction)

Range of Particle Sizes (microns)	Total # of Particles (Before Cleaning)	Total # of Particles (After Cleaning)	Removal Efficiency (%)
$0 \leq x \leq 1$	0	0	0
$1 \leq x \leq 5$	1184	1126	5
$5 \leq x \leq 10$	997	1028	-
$10 \leq x \leq 25$	739	942	-
$25 \leq x \leq 50$	277	901	-
$50 \leq x \leq 100$	46	7	85
$100 \leq x \leq 150$	10	0	100
$150 \leq x \leq 250$	10	0	100
$250 \leq x \leq 500$	5	0	100
$500 \leq x \leq 750$	2	0	0
≥ 750	2	0	0
Total No. of Particles	3272	4004	-
PAC (Cleanliness Lvl)	0.038 (335)	0.006 (225)	84

Particle Distribution – CO_2 Snow Cleaning

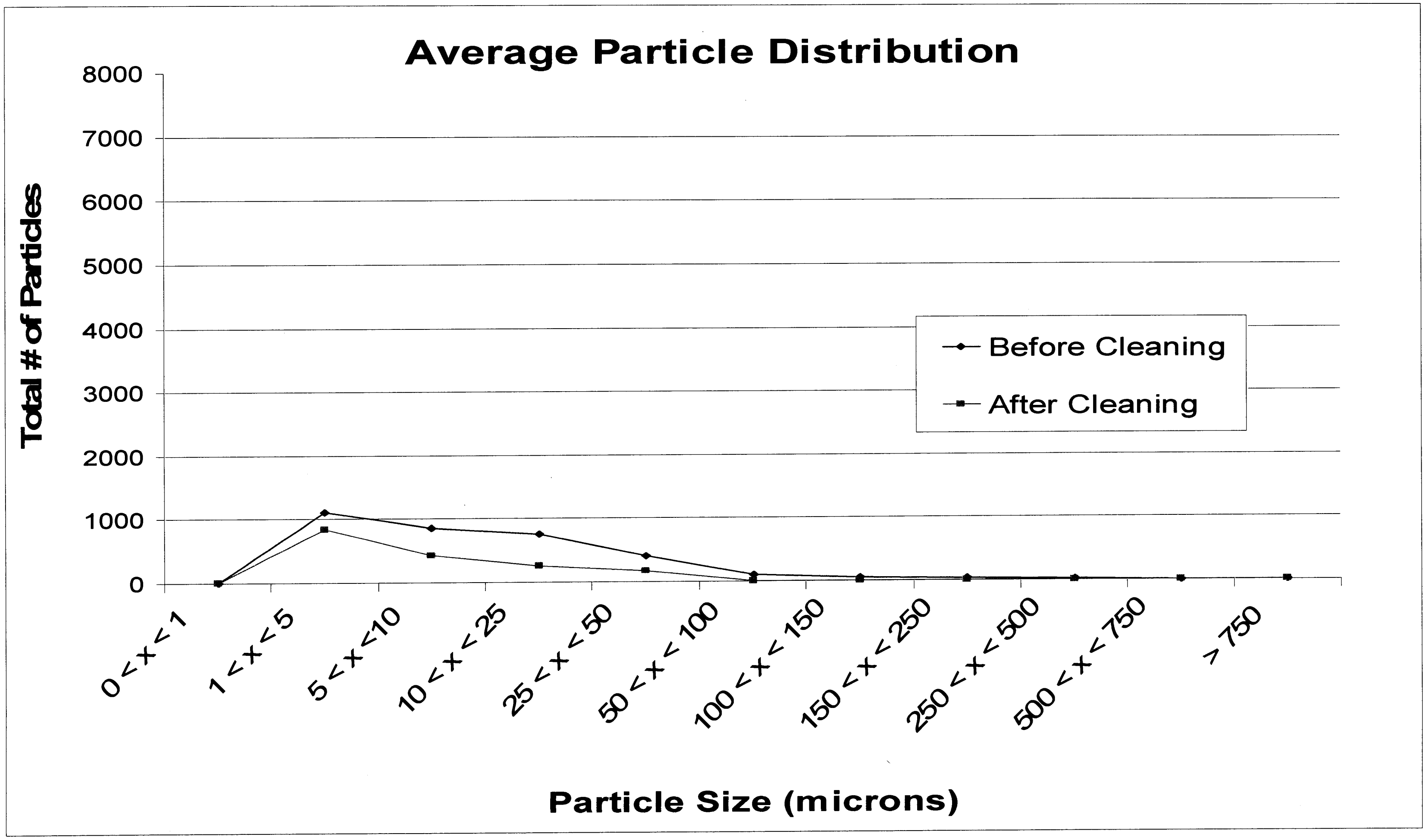


Figure 4: Average Particle Distribution for CO2 Snow Cleaning Samples

Control Wafers

Environmental Controls

Monitors particulate fallout from surrounding air during image analysis reading

Table 9: PAC Values for Environmental Controls

Sample ID	IA Reading Time (Hrs:Min)	PAC Value
Block 2	4:47	0.006615
Block 5	4:43	0.006966
Block 3	5:10	0.009732
Block 4	5:43	0.009523

Each Block includes 4 wafers

$$PAC = \frac{\text{Total Area of Particles}}{\text{Total Surface Area}} \times 100$$

Cleaning Controls

Determines amount of introduced contamination from the cleaning materials

Table 10: PAC Values for Cleaning Controls

Cleaning Process	PAC_{Before}	PAC_{After}
Detergent Bath	0.000	0.011494
Solvent Rinse	0.000	0.009231
CO_2 Snow Cleaning	----	----

Comparative Results

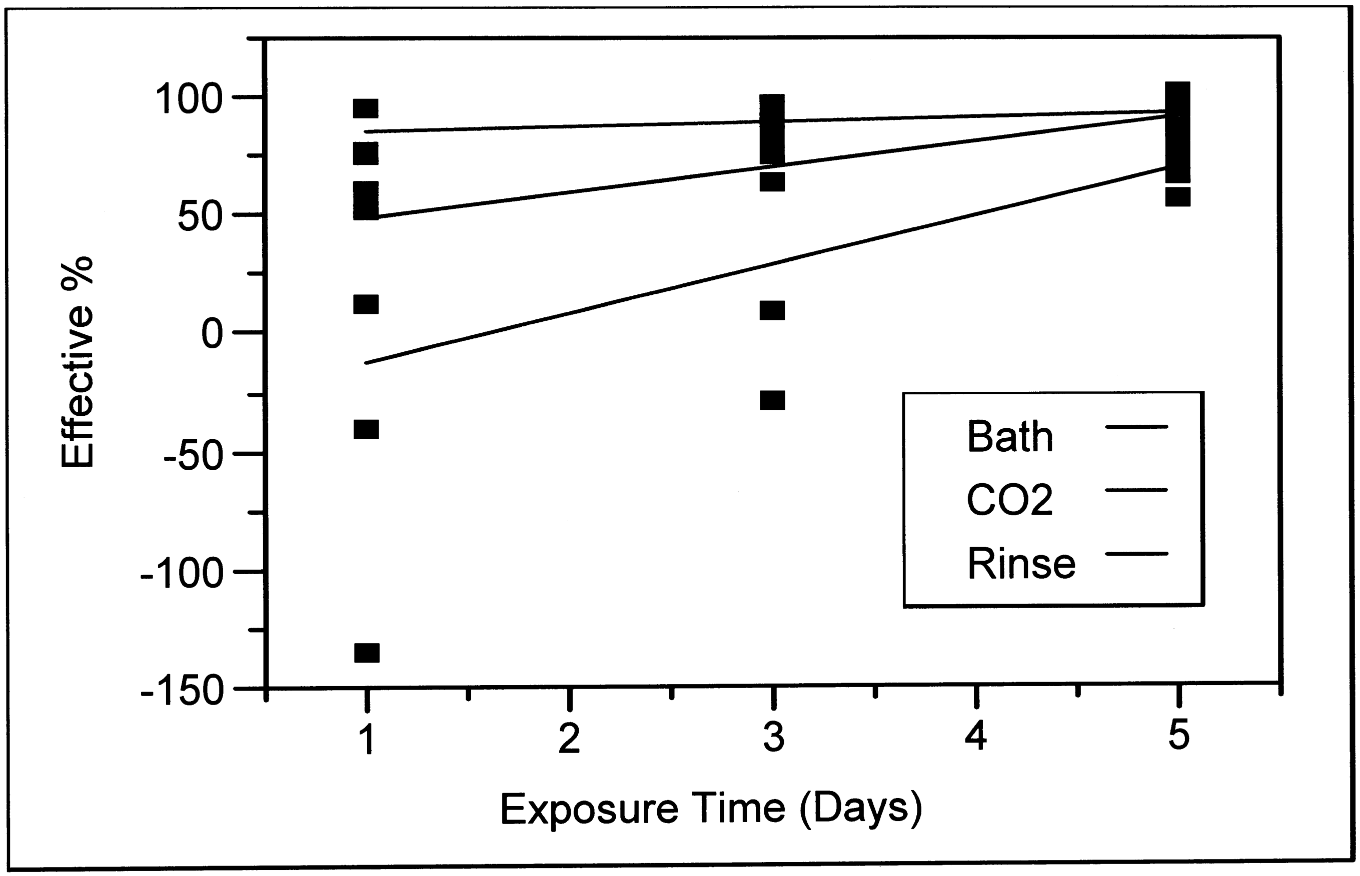
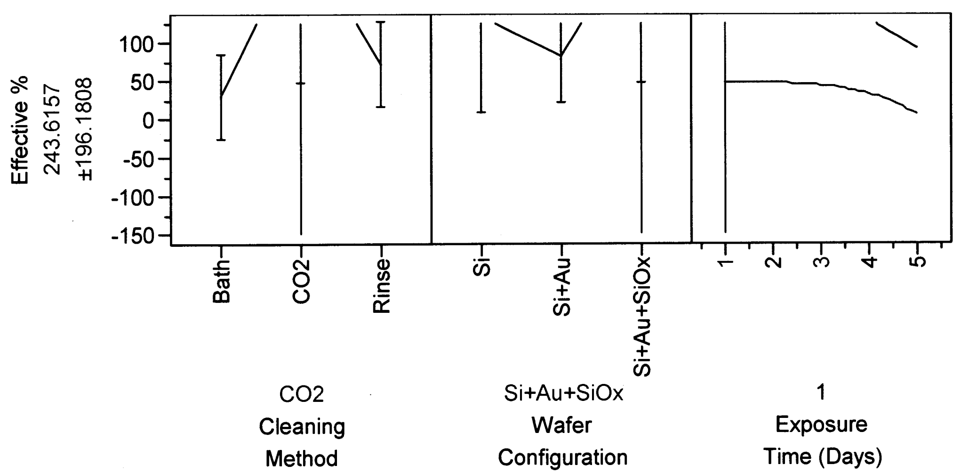


Figure 5: Regression Plot of Effective Removal Percentage

Conclusions

- Cleaning method and exposure time plays a significant factor in obtaining a high removal percentage.
  - The detergent bath and solvent rinse method displayed an increase in effective removal percentage as the contamination exposure increased.
  - CO_2 snow cleaning showed a relatively consistent cleaning effectiveness.

- For optimal removal of particulate contamination, the following settings should be used:
  - CO_2 Snow Cleaning
  - Si+Au+SiOx
  - 1 Day Exposure Time



Conclusions –cont–

Table 11: Advantages and Disadvantages of Optical Cleaning Methods

Cleaning Method	Description	Advantages	Disadvantages
Detergent Bath	Direct contact method that uses an aqueous based, nonionic detergent to loosen contaminants from the surface.	▪ "Free Rinsing" capability ▪ Reduces "creep" contamination	▪ N/A to large/complex optics ▪ Excessive handling ▪ Direct contact increases risk of surface damage ▪ Cleaning materials could introduce contamination
Solvent Rinse	Direct contact method that uses an aqueous based, nonionic detergent <u>and solvent rinse</u> to loosen contaminants from the surface.	▪ "Free Rinsing" capability ▪ Reduces "creep" contamination ▪ Rapid/spot free drying ▪ Removes some molecular contamination	▪ N/A to large or complex optics ▪ Excessive handling ▪ Direct contact increases risk of surface damage ▪ Cleaning materials could introduce contamination ▪ Excessive use of solvent could create water spots
CO_2 Snow Cleaning	Non-contact method that uses a high velocity stream of CO_2 solid and gas; removing contamination through momentum transfer.	▪ Reduced risk of surface damage ▪ Removes fingerprints ▪ No waste ▪ Quick cleaning process	▪ Requires controlled environ. ▪ Electrostatic charge ▪ Introduction of gas constituents

Recommendations

Detergent Bath

- Select low or non-particulating cleaning materials
- Use a nitrogen purge gas during drying process

Solvent Rinse

- Select low or non-particulating cleaning materials
- Use a nitrogen purge gas during drying
- Use filtered solvents
- Use a certified clean storage method for solvent

CO_2 Snow Cleaning

- Perform cleaning in a dry box or with a nitrogen purge
- Develop a working instruction for the CO_2 cleaning procedures

Future Work

- SPIE Optics and Photonics Conference in San Diego, CA (August 2006)
- Perform a repetitive mirror cleaning study
- Develop a cleaning procedure for JWST's Optical Telescope Element

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- Jeff Gum, Optics Engineer/Code
- George Harris, Coatings Engineer/Code 546

THANK YOU!

References

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[2] Kubacki, Emily. “The Dirt on Cleaning Optics.” CVI Laser LLC, Albuquerque, N.M. Photonics Spectra, March 2006

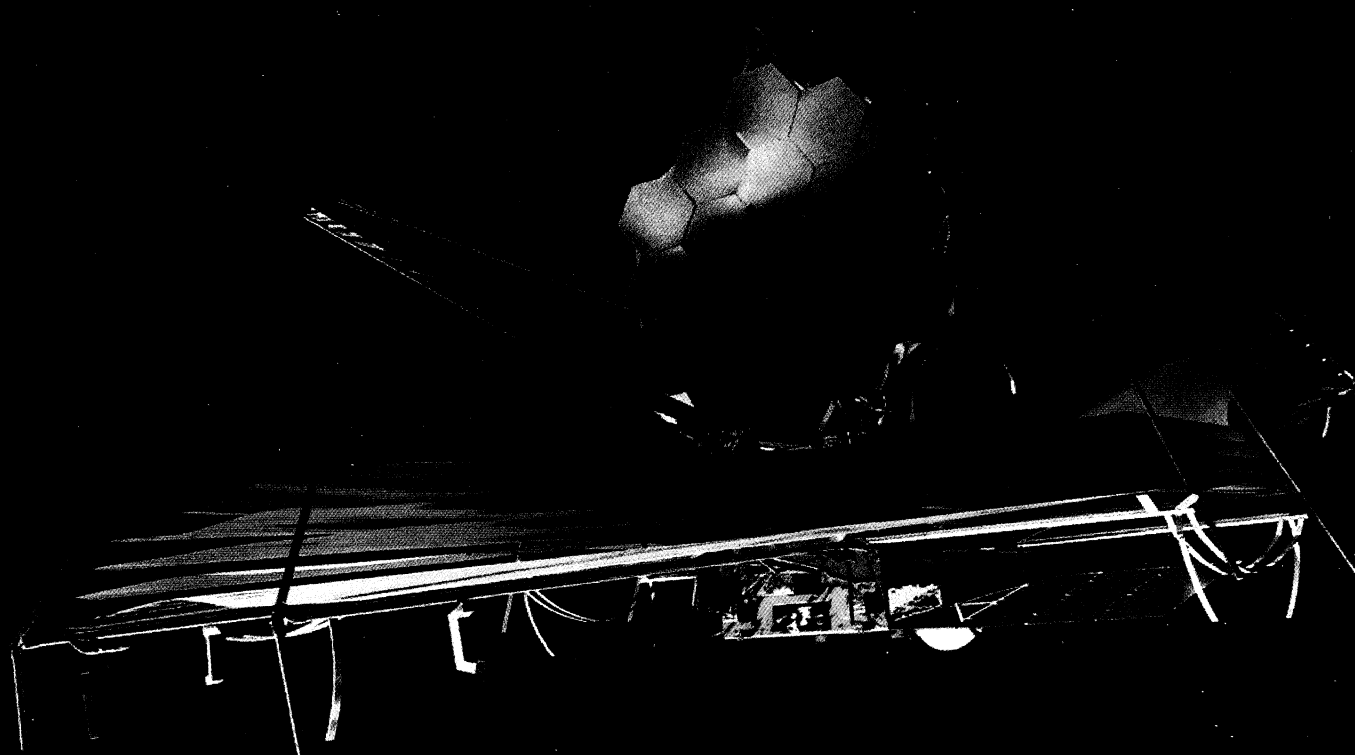
[3] Maymon, Peter. “Optical Component Cleaning.” NASA/GSFC, 551-WI-8072.1.7B, June 2004.

[4] Sherman, Robert, John Grob and Walter Whitlock. “Dry Surface Cleaning using CO2 Snow.” BOC Group, Murray Hill, New Jersey 07974. March 1991.

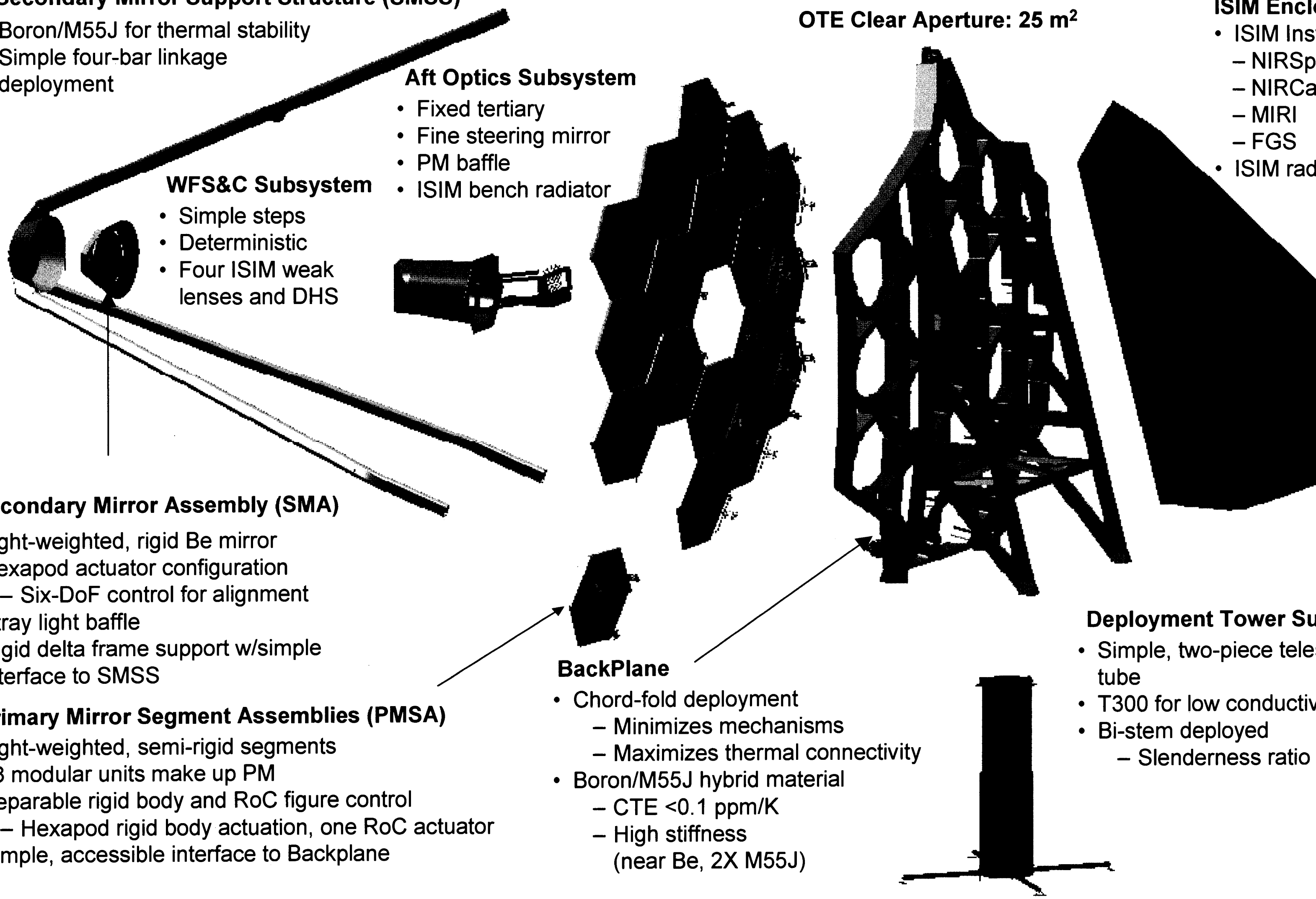
[5] Willford, John F. “The Advantages and Disadvantages, and History of CO2 Snow Cleaning.” www.cleantechcentral.com/Magazine/Past Issues/jun1998/1.asp.

Back-Up Slides

James Webb Space Telescope



Optical Telescope Element



Secondary Mirror Support Structure (SMSS)
- Boron/M55J for thermal stability
- Simple four-bar linkage deployment

WFS&C Subsystem
- Simple steps
- Deterministic
- Four ISIM weak lenses and DHS

Aft Optics Subsystem
- Fixed tertiary
- Fine steering mirror
- PM baffle
- ISIM bench radiator

OTE Clear Aperture: 25 m^2

ISIM Enclosure
- ISIM Instrument Suite
  - NIRSpec
  - NIRCam
  - MIRI
  - FGS
- ISIM radiators

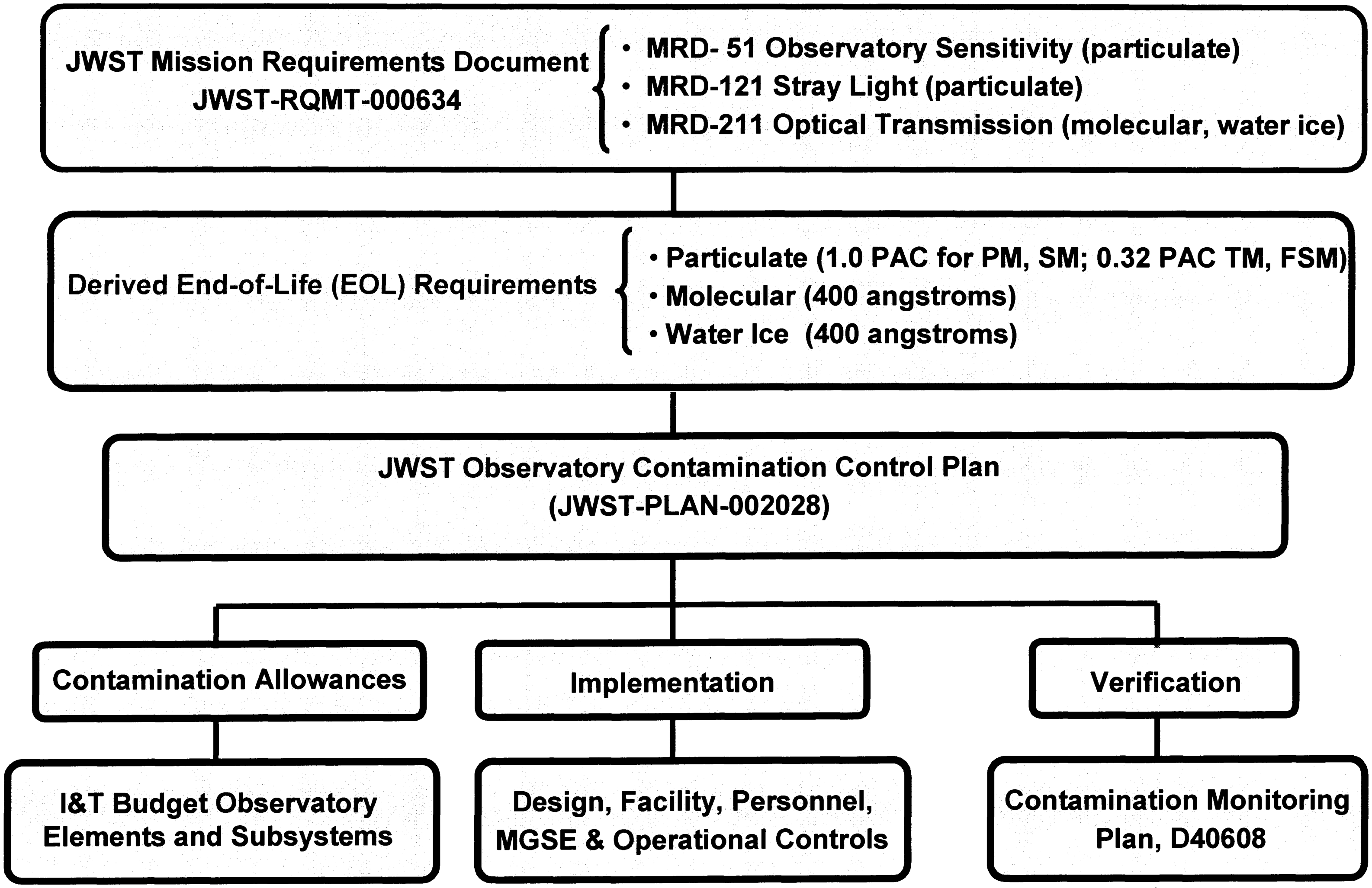
Secondary Mirror Assembly (SMA)
- Light-weighted, rigid Be mirror
- Hexapod actuator configuration
  - Six-DoF control for alignment
- Stray light baffle
- Rigid delta frame support w/simple interface to SMSS

Primary Mirror Segment Assemblies (PMSA)
- Light-weighted, semi-rigid segments
- 18 modular units make up PM
- Separable rigid body and RoC figure control
  - Hexapod rigid body actuation, one RoC actuator
- Simple, accessible interface to Backplane

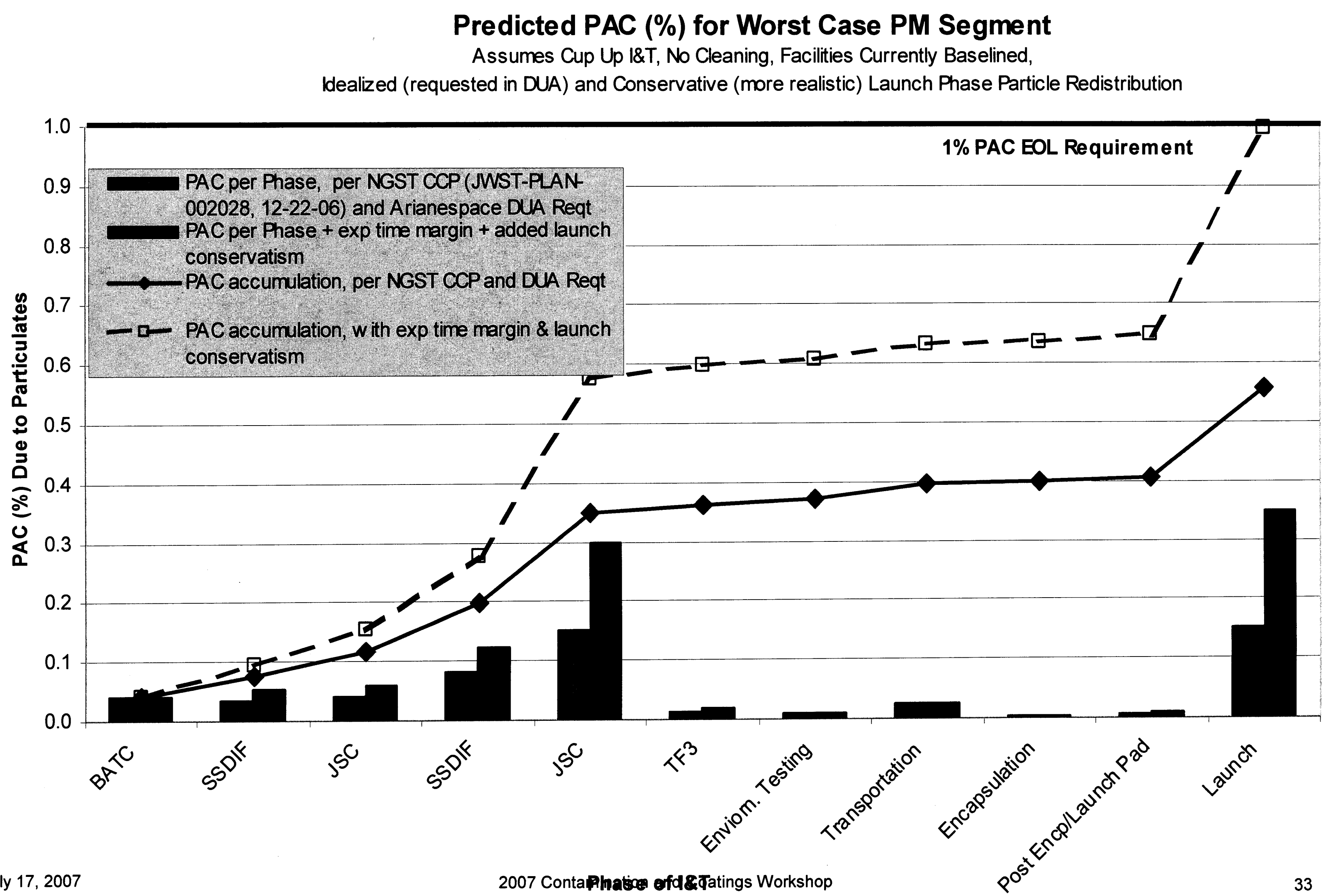
BackPlane
- Chord-fold deployment
  - Minimizes mechanisms
  - Maximizes thermal connectivity
- Boron/M55J hybrid material
  - CTE <0.1 ppm/K
  - High stiffness (near Be, 2X M55J)

Deployment Tower Subsystem
- Simple, two-piece telescoping tube
- T300 for low conductivity
- Bi-stem deployed
  - Slenderness ratio <60

Contamination Requirements



I&T Particulate Budget



I&T Budget –cont-

Predicted PAC (%) for Worst Case PM Segment

Assumes Cup Up I&T, No Cleaning, Facilities Currently Baselined,
Idealized (requested in DUA) and Conservative (more realistic) Launch Phase Particle Redistribution