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Cleaning Practices for the National Ignition Facility (NIF)

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ABSTRACT

We describe the cleaning processes, treatment methods, facilities, and cleanliness verification techniques developed to achieve and maintain the demanding cleanliness requirements for both hardware and optics used in the National Ignition Facility (NIF).

Keywords: Cleaning, cleanliness, contamination, particles, VOCs, AMCs, damage, clean room, optics, residue, NVRs

1. INTRODUCTION

Rigorous cleanliness on the National Ignition Facility (NIF) is essential to assure that 99.5% optical efficiency is maintained on each of its 192 beam lines by minimizing obscuration and contamination-induced laser damage . A major challenge for the NIF was the precision cleaning of large stainless steel and aluminum parts and structures, which included vessels as large as freight cargo containers. In addition, thousands of optics, some with sensitive sol gel coatings, had to be cleaned. In order to meet the stringent IEST-STD-CC1246D[1] Level 83 A/10 cleanliness requirements and even more rigorous requirements for optics, specialized cleaning procedures and large scale cleaning facilities were developed, as were post-installation cleaning processes and protocols. These include large volume surfactant sprays, ultrasonic baths and wiping techniques, with and without solvents, for metal and other non-optic components. In addition, in order to minimize outgassing of organic substances that can contaminate sensitive sol-gel anti-reflective coatings and appreciably reduce their transmission, many components required baking in customized vacuum ovens. To assure that requirements were met, specialized test methods were developed for verification of the cleanliness of non-optical parts. For optics, cleaning included traditional solvent wiping (isopropyl alcohol [IPA], ethanol and acetone) and complex detergent baths and surface preparation processes, including toluene sprays. Now that the NIF is in operation, cleanliness of the optics must be maintained by in-situ cleaning, including the use of gas knives on mirrors. In this paper we discuss the types of contamination that are of concern and the specialized cleaning facilities, treatments, processes and equipment that are necessary to remove the contamination.

2. CONTAMINATION TYPES AND THEIR IMPACT

2.1 General

Contamination of concern for optics, and hence for related enclosures and mechanical structures, can be divided into two general categories, organic compounds and particles. Organic compounds ("organics", Table 1) are non-metal and non-mineral substances that deposit on surfaces and may be incorporated in flexible plastics, and typically include hydrocarbons (e.g. lubricants), plasticizers (e.g. phthalates), and siloxane compounds (precursors of silicone rubbers). Organics may be left on surfaces because of inadequate cleaning or they may be transported to otherwise clean surfaces in the vapor phase as a result of outgassing from materials that have not been properly treated to eliminate them. Particles (Table 2) include solids of all types, but generally consist of minerals, metals, and plastics. This category also includes fibers.

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[1] "Product Cleanliness Levels and Contamination Control Program," Institute of Environmental Sciences and Technology, Arlington Place One, 2340 S. Arlington Heights Road, Suite 100, Arlington Heights, IL 60005-4516, December 2002

TABLE 1. ORGANICS

Contaminate	Impact	Mitigation (Paragraph)
Organics are non-mineral and non-metallic compounds of low volatility, NIF is typically concerned with those of a molecular weight $\geq$150 AMU • Most common sources are plasticizers (e.g. phthalates), siloxanes (constituent of silicone rubbers) and hydrocarbons (lubricants) • The organic requirements for the NIF are more restrictive than those for the aerospace and microelectronics industries and they posed a unique and difficult challenge • Differentiated as to gaseous, and liquid or solid phases.		
Organics in gaseous phase are called vapor-phase organic compounds (VOCs) or, in air environments, airborne molecular compounds (AMCs) • Originate from outgassing of organic compounds, such as lubricants, adhesives, electronics and insulations • Exemplified by the outgassing of organic compounds, such as phthalates which produce a haze the inside of automobile windshields	• Can be adsorbed into the pores in sol gel anti-reflective coatings on transmissive optics (e.g. conversion crystals and lenses) thereby degrading the coating effectiveness • Can form an obstructive haze on optics	6.0 7.2
Organics in a liquid or solid phases on surfaces are called non-volatile residues (NVRs) • Originate from a number of sources such as lubricants, transfer of vapor-phase organic compounds (VOCs or AMCs in air environments), fingerprints, machining fluids and mold releases; often inherent in manufacturing operations	• Can convert to VOCs or AMCs • Can convert to particle contamination if contacted by laser light and transported to optics	3.2 b. & d. 3.3 b. & d. 4.2 a. 4.3 b. 5.2 a., c. & d. 5.3 7.2 a.

TABLE 2. PARTICLES

Contaminate	Impact	Mitigation (Paragraph)
Airborne (typically ≤10microns) • Commonly referred to as "dust" and sources include ground source minerals, air pollution and human skin • Readily transported by air flow and commonly mitigated by the isolation and fan filter units (FFUs) of clean rooms	Individual particles can result in micro-pits, which alone are not necessarily a problem , but an large number can cause obscuration	3.2 a. 5.1
"Smut" (1-5 micron particles commonly on aluminum surfaces) • The adhesion of smut particles is dominated by Van der Waals, electrostatic, and capillary forces that inhibit the removal of smut particles during ordinary aqueous detergent cleaning. • Nevertheless, smut particles, which are abundant on untreated aluminum, are readily transferred between surfaces by light rubbing (as with a gloved hand) so handling may result in contamination of otherwise clean items	As with landed airborne particles, individual particles may result in micro-pits	3.2 c. 3.3 c.
"Rouge" on stainless steel surfaces • Micro surface oxidation products from free iron, typically from interaction with moisture	Similar to smut	3.2 c. 3.3 c. 5.2 b.
General gross debris (10 micron+ particles) • Introduced from clean room or beam path breaches or protocol deviations; not detectable by particle counters	Can be a source of significant local optic damage owing to interaction with of the particles with laser light	3.2 b. & d. 3.3 b. & d. 4.2 a. 4.3 a. & b. 5.2 a., c. & d. 5.3 7.3
Abrasion products (10 micron+ particles) • Introduced into clean room or beam path because of physical contact interaction of solids and aggravated by the restrictions on hydrocarbons (contact of "dry" surfaces) • Common causes: removal of fasteners, electromechanical devices, vibration, stray light/ghosts • Often includes metal chips	• Can be a source of significant local optic damage owing to interaction with of the particles with laser light • Studies have shown that metal abrasion products, particularly ≥30 microns, to cause profound surface pits, and often with a propensity to grow when exposed to further laser light[2]	5.2 a., c. & d. 5.3 7.3

3. THE CLEANING OF METALS

3.1 General

As discussed above, NIF was faced with an enormous challenge to provide metal structures and enclosures of various shapes, materials and sizes cleaned to a requirement of IEST-STD-CC1246D Level 83 A/10 ("precision clean").

[2] SPIE Boulder Damage Symposium 2005: Impact of contaminates on the laser damage threshold of 1ω HR coatings [5991-53] M. Norton, C. Stolz, E. Donohue, W. Hollingsworth, K. Listiyo, J. Pryatel, R. Hackel

Some of the traditional cleaning process involved methylene chloride, Freon™ and other solvents unfriendly to the users and the environment, particularly at the volumes that would be needed for a project the size of NIF. Before fabrication was begun on NIF, LLNL was actively involved with other organizations in investigating and testing alternative aqueous solutions. Detergents produced by Brulin[3] were among the successful candidates, and were selected for basic cleaning for the NIF. In addition, NIF developed or refined acid treatments for mitigating the surface fines of smut on aluminum alloys and rouge on stainless steels.

3.2 Large Metal Assemblies

a. To precision clean metal assemblies and enclosures, some the size of cargo containers, a facility that was set-up under contract and dedicated to cleaning parts for the NIF. It included a 5000 ft^2 Class 100/ISO Class 5 cleanroom (Figure 1), and specialized process and filtering systems (Figure 2). In addition, specialized cleaning equipment was deployed, such as the high pressure spray swivel heads for cleaning the inside of long beam tubes (Figure 3).

Figure 1. 5,000 foot^2 Class 100 (ISO Class 5) developed for NIF cleaning

[3] Brulin, 2920 Dr. Andrew J. Brown Avenue, Indianapolis, Indiana, 46205

Figure 2. Large storage tanks and process systems for detergent/aqueous solutions (left) and deionize filter system for 18 MegaOhm-cm water supply (right).

Figure 3. Specialized cleaning equipment deployed in NIF's cleaning facilities. Large NIF beam tubes (top) with internal spray swivel head (top and lower right) were cleaned in a specialized enclosure (lower left).

b. Initially the items received a "gross" cleaning in a "clean area," which included a high pressure spray (1,000-2,500 psi) of hot (43 – 54^{o}C) aqueous detergent solutions of Brulin™ 1990 GD (Figure 4) followed by spray rinsing with DI water.
c. Subsequent to the initial gross cleaning, acid gel solutions were applied (Figure 5), phosphoric acid for aluminum to facilitate the removal of smut, and ASTM recommended acid aqueous passivation solutions for stainless steel. After acid treatments, the gross cleaning process was repeated.
d. After gross cleaning, precision cleaning was performed using similar detergent-based cleaning methods, which were conducted in a clean room.

After precision cleaning and thorough drying the product was wrapped in three layers of 6-mil thick ultra-low outgassing (ULO) polyethylene sheeting, which had been tested and approved by NIF as free of additives that might be transferred onto the otherwise clean surfaces (Figure 6).

Figure 4. High pressure spray application of detergent solution

Figure 5. Application of an acid gel solution using a roller


Figure 6. Precision cleaned product triple wrapped with ULO polyethylene sheeting

3.3 Medium and Small Assemblies and Parts

a. Generally the same detergent cleaning solutions and acid treatments that were used for cleaning large assemblies were applied to medium and small items. A major difference was that the size of these items allowed for the use of tanks, as opposed to sprays and gels.
b. Ultrasonic gross and precision cleaning was applied at various cleaning facilities, including in the optics assembly building (OAB) located on the NIF site (Figure 7). One difference in the treatments was that a foaming version of the detergent was used instead of the non-foaming version used for high pressure sprays to enhance the cavitation process at the surface being cleaned.
c. Dip tanks were typically utilized for applying acid solutions (Figures 8 and 9).
d. Cleaning processes must often be modified to accommodate unique items. Examples are shown in Figures 10 through 12. Modifications can also include baking to drive off organics or moisture (Section 6.0).
e. As with large items, cleaned medium and small items were triple wrapped in ULO polyethylene sheeting or bags (Figure 13).

Figure 7. Four 2,500 gallon ultrasonic precision cleaning tanks in the NIF OAB.

Figure 8. An 8,000 gallon acid dip tank at cleaning contractors facility

Figure 9. Aluminum parts being etched in a phosphoric acid solution

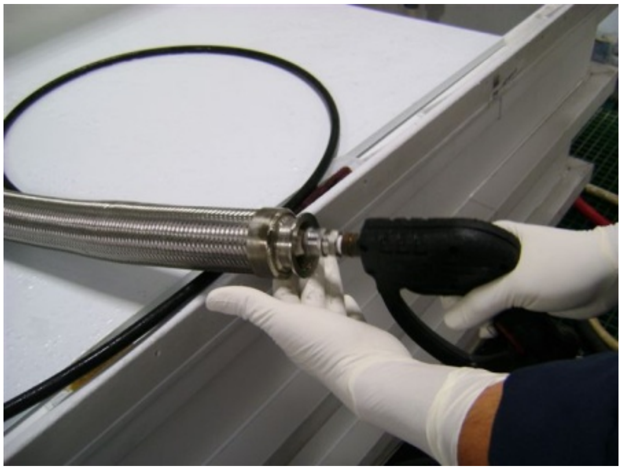

Figure 10. A flexible metal hose is flushed with detergent and DI water rinse solutions

Figure 11. A gas bottle with a single opening was cleaned by repeated blown clean with ultra-pure nitrogen after a detergent solution soak and DI water rinse

Figure 12. Items incompatible with aqueous/detergent solutions are cleaned by floating a pan of organic solvent in an ultrasonic tank

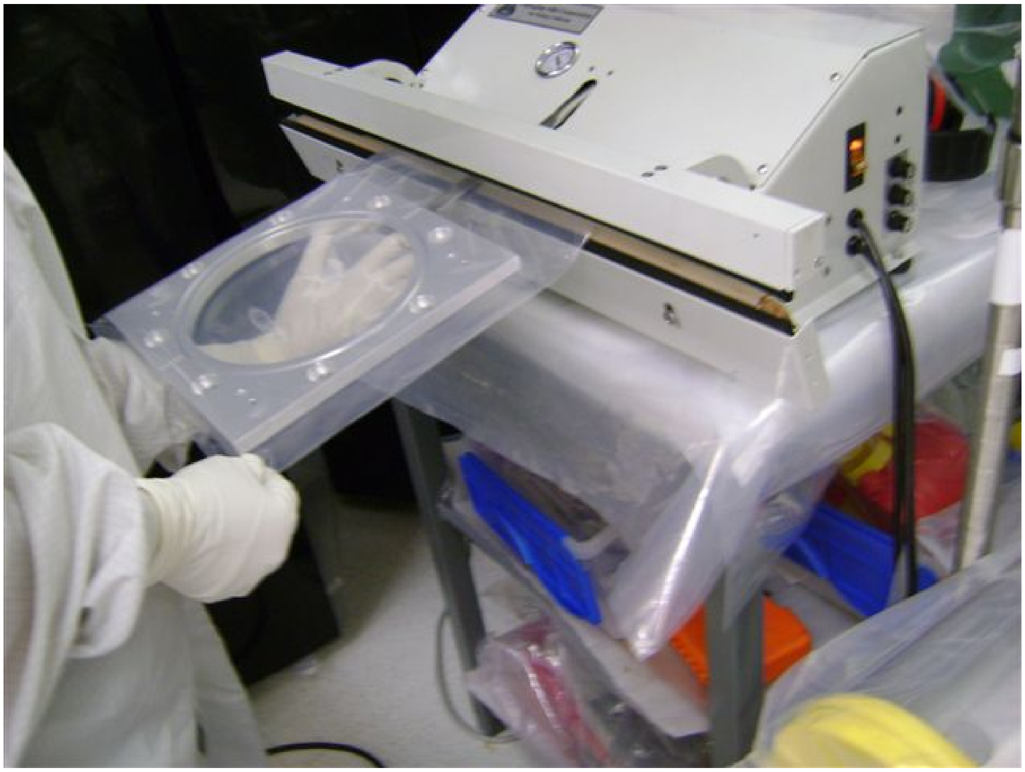

Figure 13. A precision cleaned item being heat-sealed in 3 layers of ULO polyethylene sheet.

4. THE CLEANING OF OPTICS

4.1 General

NIF relied on a number of processes for cleaning optics, some of which used detergent/aqueous solutions, and some of which used organic solvents for small optics or remedial cleaning (Section 5.3). One advantage is that cleanliness was often inherent in the coating processes for optics coated by vendors.

4.2 Large Optics

a. There are several cleaning processes used by NIF's Optical Production Facility to prepare large optics for coating or installation, including the following:

- Before application of sol gel anti-reflection coating, conversion crystals are processed with toluene (the crystals are incompatible with water), first by ultrasonic followed by spraying (Figure 14).
- Neodymium-doped amplifier slabs are hand- and spray-cleaned with DI water (Figure 15) to remove stray particles. Owing to good vendor cleaning practices, residues are not a concern here.
- Mirrors previously coated by vendors are hand-washed with detergent, followed by several DI water rinses (Figure 16).
- Before application of sol gel anti-reflection coating, fused silica optics receive automated ultrasonic cleaning using sodium hydroxide followed by a detergent solution and several DI water rinse cycles (Figure 17)

Figure 14. A conversion crystals being processed with toluene


Figure 15. A neodymium doped amplifier slab hand and spray cleaned with DI water

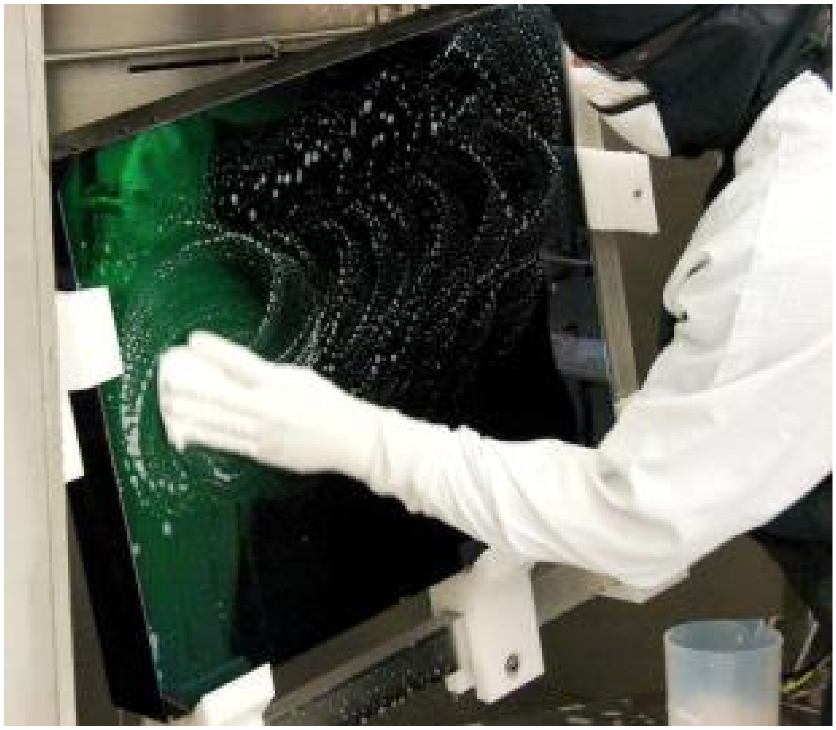

Figure 16. A coated mirror hand cleaned with detergent

Figure 17. Fused silica optics automated ultrasonic cleaning using sodium hydroxide followed by a detergent solution and several DI water rinses

4.3 Small Optics

a. Small optics for the NIF are coated by vendors, and typically require only a blow-off with ultra-pure air or nitrogen for stray particles before installation (Figure 18.), often aided by an ionizer.
b. In the case of stubborn particles or the occasional residue, optics are wiped with ultra-pure organic solvents (IPA, ethanol, acetone or a combination thereof, but never dry) using polyester or polyester/cellulose optic wipers (Figure 19).

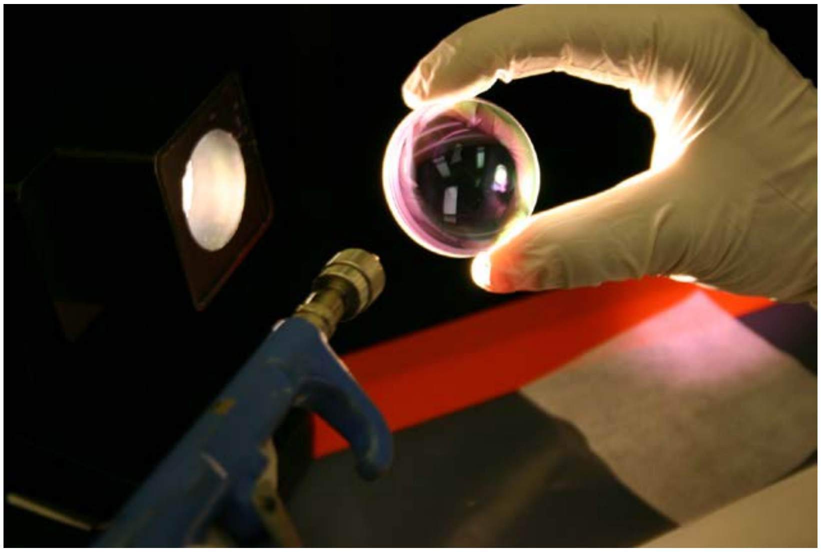

Figure 18. Blowing off particles with ultra-pure nitrogen

Figure 19. Wiping an optic with an organic solvent

4.4 Packaging of Optics

Both large and small cleaned optics are protected in ultra-low out gassing hard PET-G (polyethylene terephthalate – glycol modified) containers (Figure 20), often purged with N_2 for the large optics, then sealed with clean room tape.

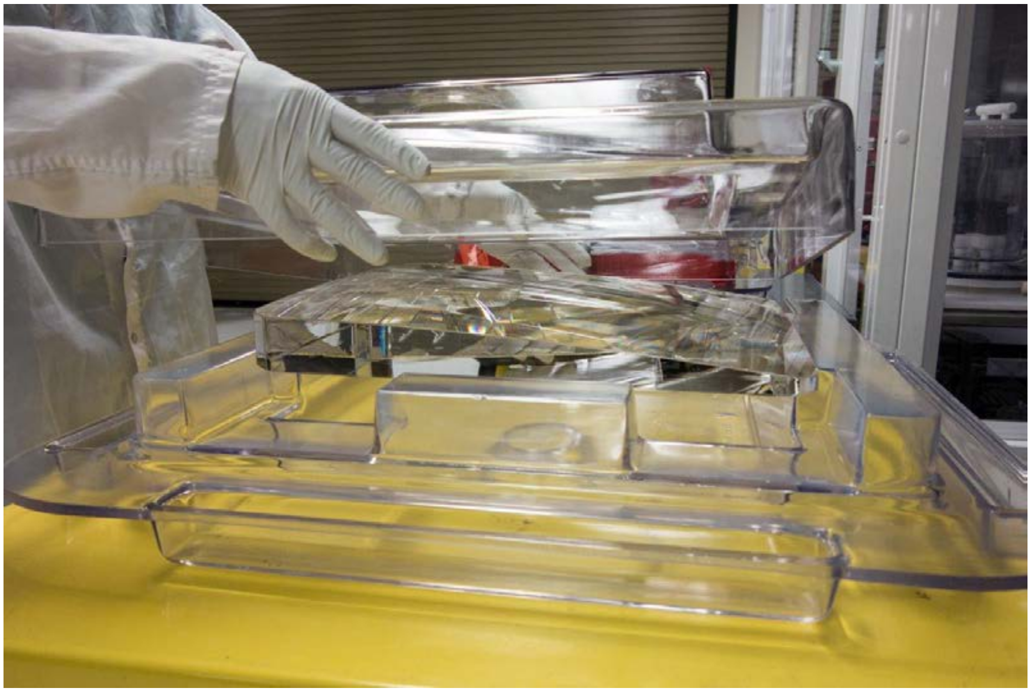

Figure 20. A clean wedge focus lens being packaged in a PET-G container

5. *IN-SITU* AND REMEDIAL CLEANING

5.1 General

There are a number of situations where mechanical enclosures and structures, and optics need to be cleaned outside of cleaning or optics processing facilities. This includes *in-situ* cleaning of very large enclosures installed in place (Figure 21) and remedial cleaning of items that were re-contaminated after installation because of off-normal conditions or operational debris. Cleaning in these situations is done by hand and methods include dry and solvent wiping, as well as vacuuming and blowing using oil-free HEPA-filtered compressed gas (air or nitrogen). *In-situ* cleaning can involve building local portable cleanrooms or converting an enclosed space into a "clean room" using forced HEPA-filtered ventilation air and strict cleanliness protocols.

5.2 Metal Enclosures and Assemblies

a. Owing to the size of several enclosures (e.g the roving mirror diagnostic enclosure [RMDE], Figure 21, and the "Periscopes") it was necessary to gross and precision clean them *in-situ*. Using hot detergent sprays was not practical so gross cleaning was performed by wiping with organic solvents, typically IPA supplemented with acetone, followed by dry wiping for particles. Special oversized cleanroom-processed 3M™ wipers were obtained for the dry wiping.
b. Subsequent to the initial gross cleaning, the stainless steel surface was passivated with acid gel solutions, similar to those used on the large enclosures discussed in 3.2. A special heated DI water spray vacuum rinse and recovery system was developed (Figure 22) to remove the acid gel.
c. After the acid treatment the surfaces received a second solvent and dry wiping gross cleaning, followed by setting up a clean room environment in the enclosure and performing a dry wiping precision cleaning.

Figure 21. The Roving Mirror Diagnostic Enclosure (RMDE) built inside the NIF

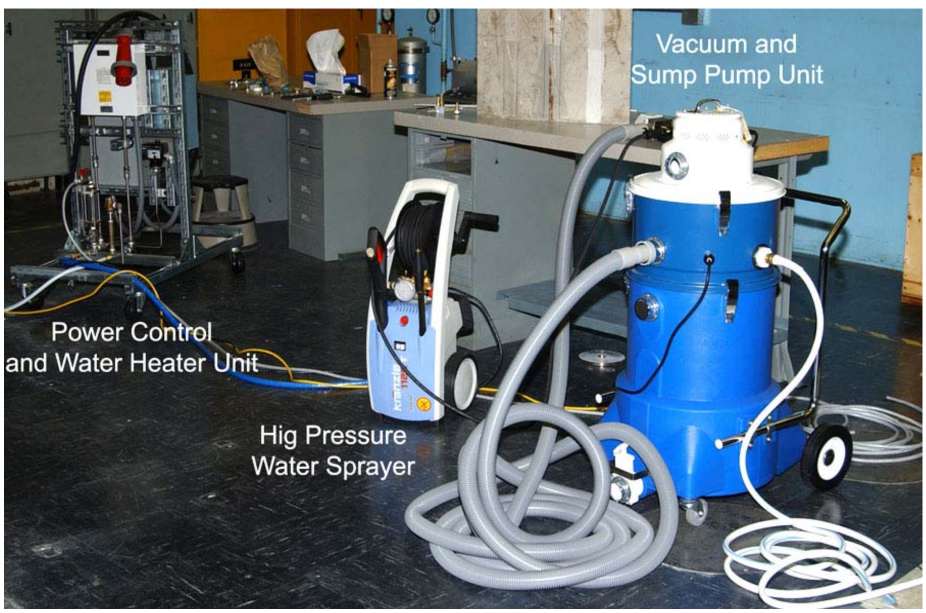

Figure 22. A spray-vacuum rinse and recovery system removed the acid gel solutions

d. Remedial cleaning of spot contamination was done by wiping (Figure 23a), often supplemented by vacuuming, often with special attachments and extensions (Figure 23b).

Figure 23a. In-situ dry wiping

Figure 23 b. *In-situ* vacuuming

5.3 Optics

As with new small optics, the primary methods for *in-situ* and remedial cleaning of optics are wiping with organic solvents (not to be used for sol gel coated optics) and blowing off with ultra clean gas. In addition, all upward-facing NIF transport mirrors are equipped with "gas knives" (Figure 24) which blow near-supersonic jets of ultra-clean argon or air across the mirror surfaces to remove any accumulated particles[4]. Prior to every NIF system shot to Target Chamber Center, the gas knives are pulsed at least once on all upward facing mirrors that will participate in the shot.

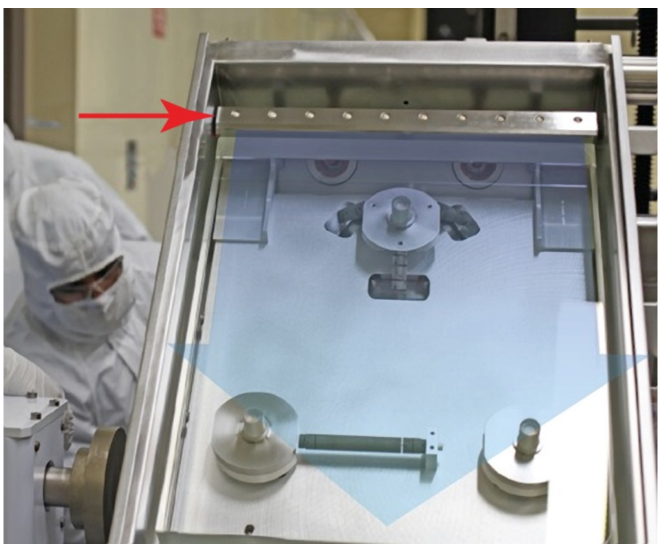

Figure 24. A gas knife installed on a NIF upward facing mirror

6. BAKING OF COMPONENTS TO REMOVE ORGANICS

Unacceptable levels of organics in components (see verification by witness optics, Section 7.2 b) can often be mitigated by baking. Vacuum ovens are used (Figure 25) with periodic nitrogen pulse-purge, which is essential to

[4] SPIE Boulder Damage Symposium 2004: "*In situ* surface debris inspection and removal system for upward-facing transport mirrors of the National Ignition Facility" [5647-9] W. Gourdin, E. Dzenitis, D. Martin, K. Listiyo, G. Sherman, W. Kent, R. Butlin, C. Stolz, J. Pryatel

remove organics in many materials.[5] Temperatures range from 80 to 200 ^{o}C and durations from eight hours to several days depending on the material and the amount and type of organic present.

Figures 25. Pulse purge vacuum baking ovens

7. CLEANLINESS VERIFICATIONS

7.1 General

To assure that the rigorous cleanliness requirements of the NIF are consistently met, cleanliness must be regularly verified. For NIF this verification is performed for both organics and particles on mechanical assemblies and parts, as well as on optics.

7.2 Organics

a. Non-volatile residue (NVRs) are measured by rinsing a surface with an organic solvent (Figure 26). The rinsate is evaporated and the remaining dry residue is weighed. The NVR precision clean requirement for NIF is 1mg per 1 m^2 ($\leq$ 0.1mg/ft^2, A/10 per IEST-STD-CC1246D).
b. Vapor-phase organic compounds (VOCs) are measured by exposing a witness optic coated on both faces with porous silica sol-gel to the environment. Organic compounds are readily adsorbed by the sol-gel coatings and reduce the transmission through the optic. Measurements are performed under environmental conditions comparable to those in which items will be used (i.e. vacuum or 1 atm air or argon) and can also be performed *in-situ*, as in a laser enclosure. The acceptance criterion is ≤0.1% transmission loss through the optic.
c. Airborne molecular contaminates (AMCs) can also be measured using adsorption tubes, in which air from the environment is drawn over a granulated adsorbing medium for an extended period and then subsequently desorbed and analyzed. This method is less sensitive than the measurement of VOCs with witness optics.

[5] SPIE Boulder Damage Symposium 2006: Qualification of materials for applications in high fluence lasers [6403-82] J. Pryatel, W. Gourdin, G. Hampton, D. Behne, R. Meissner

Figure 26. Performing an NVR sample

Figure 27. A sol gel coated witness optic

7.3 Particles

a. The primary tool for verifying particle cleanliness on non-optical surfaces is a particle swipe, a type of “white glove test” for collecting particles (Figure 28), which are then counted by an automated electro-optical instrument. The precision clean requirement is ≤ Level 83 per IEST-STD-CC1246D.

Figure 29. Performing a particle swipe

b. The primary tool verifying particle cleanliness of optical surfaces is “bright light” inspection (Figure 29), with which particles 10μm or larger typically can be seen, as well as residues. The precision clean requirement for optics is ≤Level 50 per IEST-STD-CC1246D (5 visible particles per ft^2), and no residues. Bright light inspections are also used to supplement particle swipes on non-optical surfaces (Figure 30), and are particularly effective in detecting abrasion products.

Figure 29. Bright light inspection of optic

Figure 30. Bright light inspection of a surface

8. CONCLUSION

We have described the cleaning processes, treatment methods, facilities, and cleanliness verification techniques used to process materials and components for the National Ignition Facility. Rigorous application of these protocols is essential to meet and maintain the demanding cleanliness requirements of large laser systems.

ACKNOWLEDGEMENT

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