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TECHNIQUES FOR REMOVING CONTAMINANTS FROM OPTICAL SURFACES*

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The advent of very high power solid state lasers for fusion research has created a need for extremely clean large optical elements. Cleanliness is vital in laser systems because the direct heating of contaminants by the laser beam or xenon flashlamps is sufficient to cause anti-reflection coatings, high-reflectance coatings, and uncoated optics to become damaged. The contaminants of concern are particles or agglomerations of particles exceeding $1\text{ }\mu\text{m}$ diameter. The primary concern is the elimination of these particulates from optical surfaces before the application of evaporated or sputtered coatings, before the optic is installed into the laser system, and during periodic maintenance.

Cleaning methods are needed which are extremely effective on small and large surface areas, relatively rapid, and easily monitored. In general detailed knowledge of the composition of contaminants is of little importance except as is necessary to aid in determining their source. Since inadvertent recontamination is inevitable, only cleaning methods applicable to all types of contaminants are appropriately considered.

Particle removal procedures such as plasma cleaning, ultrasonic agitation of solvents, detergents, solvent wiping, mild abrasives, vapor degreasing, high pressure solvent spraying and others have been evaluated and the results

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are reported here and elsewhere^{4,5,11}. Wiping with a lens tissue wetted with an organic solvent and high pressure fluid spraying are the only methods by which particles as small as 5 μm can be effectively removed. All of the other methods tested were found to be at least two orders of magnitude less effective at removing small insoluble particles. An additional and as yet unresolved problem is the development of a reliable method for evaluating particulate surface cleanliness. Without such a reproducible monitoring technique, the large diversity of cleaning methods currently available cannot be quantitatively evaluated.

INTRODUCTION

Several ultra-high power laser systems have been constructed by the University of California at the Lawrence Livermore Laboratory and the Los Alamos Scientific Laboratory to prove the feasibility of driving thermonuclear fusion reactions by the inertial confinement of a plasma. The Nd:glass laser system, Shiva¹, at the Lawrence Livermore Laboratory is constructed of twenty individual beams arranged to impinge on the fusion target simultaneously. The Shiva laser system contains 1600 coated and uncoated optics and Nova², the next generation laser system, will contain approximately 5000 optics. To extract maximum power and energy from the laser, the beam intensity is maintained only slightly below the damage threshold of the coated and uncoated optical surfaces. Contaminants on these surfaces therefore act as sites for the initiation of damage to the bare optics or the anti-reflection coatings. Optically absorbing films and nontransparent particulate contami-

nants attenuate the beam and add intensity variations to the beam profile. Both of these effects reduce the amount of focusable energy reaching the target³ and therefore must be avoided. A very significant problem therefore is the cleaning and maintenance of literally thousands of components in the laser system^{4,5}.

A contaminant distributed as a uniform film on an optical surface can be tolerated if its net beam attenuation does not exceed 0.01% per surface. This is derived from a desire to keep the cumulative effect of films on the approximately 100 surfaces in each beam from exceeding 1% attenuation. A typical size distribution of contaminants at a concentration of only 15 particles/cm² >10 μ m diameter obscures .01% of an optical surface. We are primarily concerned with reducing the surface particle concentration below this level. This is a very clean surface and is very difficult to verify in practice.

Because of our need to remove particulate contaminants from optics, we have investigated many surface cleaning procedures to quantify their cleaning speed, particulate removal efficiency, and the cleanliness levels that are ultimately attainable with each procedure. A search of the literature indicates that quantitative cleaning evaluations of this type have not been performed and in general particle removal mechanisms do not appear to be well understood. Although measurements of the binding forces between particles and surfaces have been reported^{6,7,8}, the work has not been extended to include the application of this information to overcoming the binding forces. Small particles are strongly bonded to surfaces by Van der Waals and electrostatic forces far exceeding the particle's weight, and binding forces exceeding 10,000 times the particles weight⁸ have been measured.

EXPERIMENTAL PROCEDURE

Because of a general lack of information by which cleaning methods using vastly different particle removal mechanisms could be directly compared, the authors undertook such a research program. Briefly, an artificially contaminated surface was created by uniformly distributing $5\text{ }\mu\text{m}$ Al_2O_3 particles onto both flat optical surfaces and polished metal surfaces¹¹. The contamination concentration was determined by microscopic counting both before and after cleaning. The contaminated samples were then cleaned by the seven methods listed in Table I and the efficiency of cleaning ((initial - final)/ initial concentration) and lowest achieved particle concentration tabulated.

Aluminum oxide was chosen as the contaminant because it closely resembles airborne contaminants in that it 1) is insoluble in common solvents used for cleaning optics, 2) has a reproducible size distribution for testing uniformity, and 3) should be as difficult to remove as any naturally occurring mineral such as silica. The basic size of $5\text{ }\mu\text{m}$ represents a size which is relatively easy to count microscopically and represents the size range which is by far the most difficult to remove. Particles exceeding $200\text{ }\mu\text{m}$ are relatively easy to remove by all the methods listed in Table I and therefore are not of practical interest.

The choice of a solvent for a cleaning procedure may have a profound effect on the results. This is particularly true if the contaminant composition is known, then a solvent should be selected in which the contaminant is soluble. Solvent extraction will not be discussed further because with mixed

EFFICIENCY OF REMOVING PARTICLES FROM OPTICAL SURFACES

		Removal Efficiency for Particles $> 5 \mu\text{m}$	Lowest Achieved Particle Concentration/cm ² $> 5 \mu\text{m}$
1. Scrubbing and dragging with lens tissue using ethanol and acetone		99.6 – 99.93%	2 – 40
2. Spraying with liquid solvent, 5-30 s duration			
Trichlorotrifluoroethane (Freon TF*)	345 kPa (50 psi)	97%	1500
		3% for $> 1 \mu\text{m}$ particles	
"	"		
	6.9 MPa (1000 psi)	99.7 – 99.9%	10 – 35
		81% for $> 1 \mu\text{m}$ particles	
	Water		
	17 MPa (2500 psi)	98 – 99.5%	2 – 60
3. Adhesive strippable coating			
Scotch 2253†		95 – 98%	500
4. Ultrasonic agitation of Freon TF, 1–2 min duration			
		24 – 92%	9000 – 70000
		1% for $> 1 \mu\text{m}$ particles	
5. Sequential cleaning operation			
Ultrasonic agitated TWD-602*/followed by Freon TF low pressure spray/vapor degrease/immersion in boiling solvent/ultrasonic agitation in rinse tank		92%	4000
6. Compressed gas jet for 10 s duration			
Micro-duster** 690 kPa (100 psi)		50 – 61%	5000 – 5800
7. Vapor degreasing in Freon TF		11 – 28%	65000 – 80000

* E. I. duPont de Nemours & Co. Inc., Wilmington, DE

† 3M Co., St. Paul, MN

** Texwipe Co., Inc., Hillsdale, NJ

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contaminants the extraction process will only aid in the removal of soluble contaminants leaving behind the insoluble species. The choice of a solvent also influences the cleaning efficiency based on its evaporation rate. Adequate flushing action is difficult to achieve with rapidly evaporating solvents unless large quantities of the solvent are applied to the surface. Rapid evaporation has the effect of redepositing those suspended or soluble contaminants as small concentrated spots of contaminants when an isolated drop evaporates. *These concentrated contaminant spots are particularly noticeable on optical surfaces.*

EXPERIMENTAL RESULTS

Vapor Degreasing

Vapor degreasing creates a gentle flushing action on an optical surface when immersed in the solvent vapors. Soluble organics can be expected to be effectively flushed from the surface but small insoluble particles are simply redistributed and very few are ultimately removed as shown in Table I. The flushing time is also comparatively short in duration because of the relatively rapid heating of the glass surface.

We have observed the reentrainment of particles from the liquid solvent to the vapor and their deposition onto clean surfaces in a vapor degreaser. This occurred in a vapor degreaser with a high boiling rate created by electric immersion heaters. This effect can be reduced by preventing rolling boiling and by continuous filtration of the degreaser solvent.

In general, vapor degreasing is not needed nor recommended for optics cleaning unless it can be experimentally shown that the process indeed removes something not removed by one of the procedures to be discussed below.

Ultrasonic Cleaning

The ultrasonic agitation of solvents has long been used as a means of improving the effectiveness of the simple immersion cleaning and solvent extraction process. Simple solvent immersion is completely ineffective for removing 5 μ m particles, but when aided by ultrasonic agitation can approach 60% removal as shown in Table I. The length of immersion time or the choice of solvent appear to have little effect on improving these experimental results¹¹. In fact, long immersion times (30-60 minutes) appear to reverse the cleaning effect and surfaces generally show more contaminants than after 15 seconds cleaning.

Ultrasonic cleaners which the authors have encountered are totally inadequate in their filtration rate. Their pumps are generally sized to circulate solvent once per hour or less. Since very little filtered solvent comes into contact with the surface during immersion, it can be expected that particles are removed and then redeposited because of the lack of flushing action. All ultrasonic cleaners we have investigated also extract their fluid only from the bottom of the tank thereby accumulating buoyant contaminants on the solvent surface. The cleaned part must then be extracted through this surface layer and will again become contaminated. Ultrasonic cleaning therefore can only be recommended for gross cleaning and is not recommended for the precision cleaning of optics.

Strippable Adhesive Coatings

The use of a strippable adhesive coating to remove particles is a somewhat unconventional cleaning procedure but has proven to be quite effective at removing even small particles. The coating may be applied to the surface with a brush or by dipping. The coating is removed by peeling it from the surface after thorough drying. Coatings such as collodion (cellulose tetranitrate in ether and alcohol) and commercial coatings such as Scotch 2253* Strip-Coat have been evaluated with results shown in Table I. A drawback to these adhesive coatings is that a thin organic film remains on the surface after the thick plastic coating is removed. This film may be subsequently removed by using an organic solvent, as in a vapor degreaser. If the organic film is composed of a low molecular weight constituent of the coating formulation, it may be possible to reformulate the strippable coating to minimize this undesirable effect.

A clean strippable coating could prove to be very helpful for cleaning mounted optics. Substantial effort is expended in large laser systems re-cleaning large optics such as turning mirrors exposed to airborne contaminants. Disassembly of most components is time consuming and exposes the optic to unnecessary physical abuse. In-place cleaning is particularly difficult inside inherently long and complex hardware such as disk amplifiers and polarizer-Faraday rotator assemblies. A strippable coating that could be

*Minnesota Mining and Manufacturing, St. Paul, Minnesota.

applied by any laser technician and easily removed when dry would greatly improve system reliability and overall system cleanliness.

Cleaning by Viscous Shear Forces

The shear force exerted by a moving fluid on a small particle on a surface has been reported^{7,10} in the literature. If the shear force exceeds the adhesion force holding the particle to the surface, the particle will be removed (reentrained) and suspended in the turbulent fluid. This has the dual advantage of both dislodging small particles and applying a flushing action which carries the particle away from the vicinity of the surface. For a given free stream fluid velocity, the more viscous and dense the fluid, the more momentum will be imparted to the attached particle. This is verified by comparing the cleaning efficiency of compressed fluorocarbon "dusters" with liquid solvent spraying. Table I indicates that liquid spraying at 345 kPa (50 psi) is ten times more effective than a gaseous spray at a similar pressure. Increasing the pressure, and corresponding fluid velocity, results in an increase in removal efficiency. Using a narrow fan-spray nozzle, the nozzle-to-surface distance should not exceed one hundred nozzle diameters to retain the quoted removal efficiencies.

An unfortunate result of high pressure spraying with trichlorotrifluoroethane is the cooling of the solvent that results from its rapid expansion and the subsequent chilling of the glass surface. If the surrounding atmosphere contains water vapor, the surface can be cooled below the dew point causing water condensation. The evaporation of the cold water droplets can leave the

surface spotted as described earlier. Ongoing experiments will determine if surrounding the surface being cleaned with dry nitrogen or saturated solvent vapors will prevent the observed water spotting.

An alternate method for removing water droplets that results in spotless drying is the use of centrifugal forces generated by spinning the optic while it is being sprayed. A commercially available integrated circuit silicon substrate cleaner was recently evaluated for particle removal efficiency. The cleaner uses a water spray at 17 MPa (2500 psi). The results are similar to those obtained using the high pressure organic solvent spray and did not result in water spotting. This process is the most viable alternative to the currently used lens tissue scrubbing procedure described below.

Cleaning by Scrubbing and Swiping

Rubbing an optical surface with a solvent saturated lens tissue is currently our standard cleaning procedure. It takes advantage of solvent extraction and imparts a high liquid shear force to attached particles when the fibers in the tissue pass over the surface. Final cleanliness is sensitive to the presence of contaminants in the solvent and in the lens tissue. Recontamination is avoided by discarding each tissue after one pass over the surface. Although this method achieves the surface cleanliness levels necessary for laser systems, it has the following limitations: 1) slow laborious process, and 2) results are subject to technician skill and attention to detail. Because of these inherent limitations, faster and more operator independent techniques such as the high pressure solvent spraying technique are being developed. Such techniques will be needed to efficiently maintain glass laser systems of the future.

CONCLUSION

Large glass laser systems have created a need to clean and maintain clean thousands of optical surfaces in an ultra clean condition for periods of time exceeding a year.

Measurements of the particle removal effectiveness of seven cleaning processes has shown that scrubbing with solvent saturated lens tissues is the most effective particle removal process leaving only 2-40 particles/cm² (>5 μ m) followed closely by spraying with high velocity liquid jets 2-60/cm² and strippable adhesive coatings 500/cm². Either of the new techniques greatly exceeds the cleaning effectiveness of the more conventional production cleaning techniques of either ultrasonic agitation of a solvent or vapor degreasing.

High pressure liquid spraying using an organic solvent or water may evolve into a very rapid and efficient cleaning procedure for unmounted optics when problems of spotless drying are overcome. Strippable adhesive coatings hold great promise for the cleaning of mounted optics. Their single drawback, that of leaving an organic film when stripped from the optical surface, may be overcome by advances in strip-coating formulations.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

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TABLES

- I. Table of cleaning methods showing cleaning efficiency $((\text{initial}-\text{final})/\text{initial concentration})$ and lowest particle concentration achieved with artificially contaminated glass and metal surfaces.