Selection of Optical Cavity Surface Coatings for 1 μm Laser Based Missions

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Abstract—The particulate surface cleanliness level on several coatings for aluminum and beryllium substrates were examined for use in the optical cavities of high pulse energy Nd:YAG Q-switched, diode-pumped lasers for space flight applications. Because of the high intensity of the lasers, any contaminants in the laser beam path could damage optical coatings and limit the instrument mission objectives at the operating wavelength of 1 micron (μm). Our goal was to achieve an IEST-STD-CC1246D Level 100 particulate distribution or better to ensure particulate redistribution during launch would not adversely affect the performance objectives. Tapelifts were performed to quantify the amount of particles using in-house developed procedures. The primary candidate coatings included: chromate conversion coating aluminum (Al), uncoated Al, electroless Nickel (Ni) on Al, Ni-gold (Au) on Al, anodized Al, and gold (Au)/Ni on Beryllium (Be). The results indicate that there were advantages in Ni and Au coating applications for the two major substrates, Al and Be, when considering applications that need to meet launch environments.

TABLE OF CONTENTS

1. INTRODUCTION

Contamination control in spaceflight lasers is an important engineering and implementation function in maintaining the throughput performance requirements. Identifying the contamination critical surfaces within the laser system is the first step in understanding how to develop a contamination control plan to preclude any degradation. Cleanliness control issues with previous flight 1 μm lasers such as the Mars Observer Laser Altimeter (MOLA) launched in 1996 and Geoscience Laser Altimeter System (GLAS) launched in 2003 have focused attention on the need to go beyond traditional protocols for spacecraft processing. Areas that require a significant effort are: the ambient surface and airborne environment, vent design, and material & coating selection during fabrication, ground processing, launch, and on-orbit operations.

Many parameters for acceptability must be considered. At a minimum, considerations include mass, thermal properties, material survivability, and cleanliness aspects. Internal surface coatings are generally immune from space environmental effects, such as atomic oxygen erosion or external outgassing concerns. However, when considering the protection of surfaces for material survivability during ground processing and on-orbit operations, oxidation and corrosion must be examined. In addition, any particulate contribution from the inherent material during integration activities affects the derived surface cleanliness level necessary for laser spaceflight application.

In addition to the MOLA and GLAS laser instruments, the National Aeronautics and Astronautics Administration (NASA) Goddard Space Flight Center (GSFC) developed two flight contamination sensitive Q-switched laser instruments operating at 1 μm. The Vegetation Canopy Lidar (VCL) and the Mercury Laser Altimeter (MLA) are diode-pumped Nd:YAG with laser output of 15mJ, 10ns, and 20mJ, 4.8ns, respectively. The main focus of this paper is the potential particulate generation of surface coatings used for these two space flight related laser applications, and the considerations given to both in the cleanliness program for the selection of the optical cavity coatings.

2. VCL INSTRUMENT

Brief Spacecraft and Laser Unit Description

The VCL laser was an instrument on the Mulit-Beam Laser Altimeter (MBLA) spacecraft built by Orbital Sciences Corporation, Dullas, Virginia. VCL used an existing active Q-switch design that was adapted for a 400 km circular Earth orbit at an orbit inclination of 69°, and a non-operating survival range of -20° to +65 ° Celsius. The main mission goals were to map and condition of the forest ecosystems to provide an inventory of forest biomass and new measurements of the texture of the Earth's land cover. Figure 1 shows the VCL spacecraft configuration and the five laser units. The laser units maintained a pressurized environment using a Viton® seal. The solar arrays are not shown in the figure.

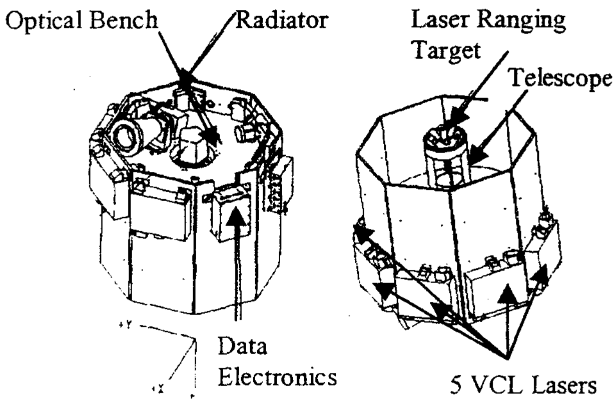

Figure 1 -MBLA Spacecraft Configuration

The instrument design was based upon airborne laser altimeters and a Space Shuttle altimeter. The operational design life for the lasers was 2 years with a 3-year goal. Because it was an existing design heritage, most materials and coatings were pre-selected. The housing was originally specified as Beryllium, but due to costs, it was changed to series 7075 Aluminum (Al). The laser bench and was fabricated using Alca Plus cast Al. Alca Plus is a Reynolds Al proprietary alloy, which was selected for its thermal characteristics and fabrication ease. Figure 2 shows the expanded laser unit with the two-cavity configuration and the bench removed from the housing. The bench, cover, and housing surface was Al with a chromate conversion coating. The conversion coating was applied according to the MIL-C-5541E specification. The conversion coating will prove to be a potential particulate generator as shown in later discussion and results.

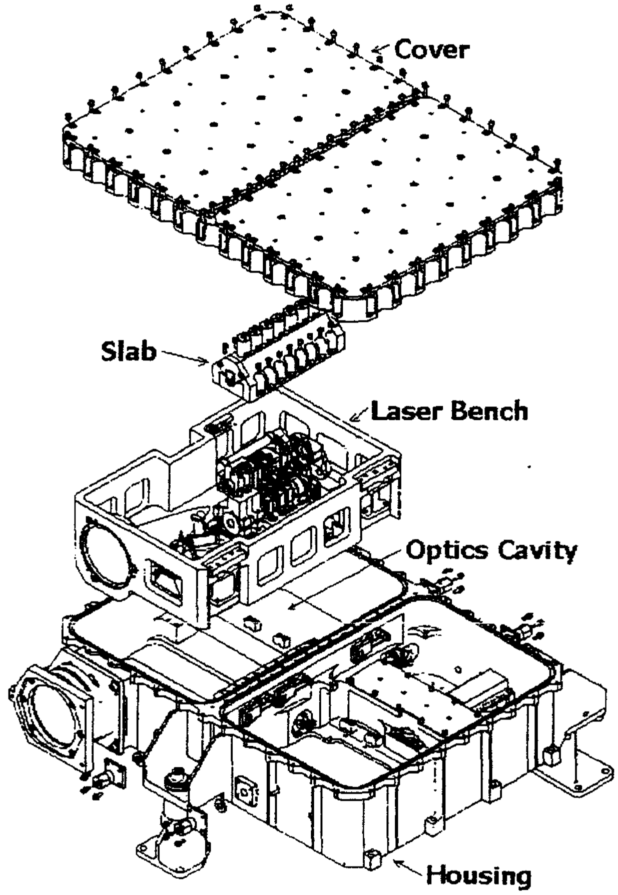

Figure 2 - VCL Laser Unit, Expanded View

Cleanliness Requirements for the Laser Cavity

The surface cleanliness requirements for the laser optics had the greatest potential for impacting the performance of the instrument. Because a correlation between performance degradation and surface cleanliness level was not established, particulate contamination requirements from other stringent flight programs were adopted. Molecular cleanliness was considered less stringent because it was believed the pressurized environment design of the laser cavity sufficiently lowered the molecular mean free path, and thus reduced, but not dismissed, outgassing concerns. However, high outgassing materials such as, silicones, were strictly prohibited. In addition, silicones have a tendency to creep on surfaces and polymerize with ultraviolet (UV) exposure, thus making them even more of a cleanliness threat. A surface cleanliness requirement of Level 300C per IEST-STD-CC1246D or a percent obscuration (% obs.) of 0.019 was established. A cleaning to Level 200C or 0.003 % obs. was applied to offer margin on the optics and account for particulate redistribution during launch, ascent, and on-orbit operation. The Level C (also approximately equivalent to 3.0 $\mu g/cm^2$) requirement represents the nonvolatile residue (NVR) molecular accumulation.

During the assembly of the laser optical cavity, an airborne cleanliness requirement of Class 10,000 or better environment per ISO 14644-1 was required along with full cleanroom attire. A relative humidity requirement between 30 to 50 percent was maintained. All piece part assemblies that went through a thorough precision cleaning according to material composition and a visual inspection with quantitative analysis to verify surface cleanliness levels. During the precision cleaning and verification process, the conversion coating applied to the bench and housing were discovered to be sloughing particles. Particle counts in excess of Level 500 were observed as shown in Table 1. It was later discovered that a contributing factor to the high particle distributions was that the conversion coating parts exceeded the recommended 60°C during post fabrication processing. The sloughing was believed to result from the coating dehydrating and the resulting insolubility of the chromates within the coating. Because of the sloughing particles, the surface cleanliness requirements were exceeded. Due to schedule constraints, fabrication of new parts was not possible. Another solution was to mechanically or chemically remove the conversion coating from the bench and housing. Before any modifications to flight hardware would occur, a mini-research effort was requested. A series of test coupons to mimic the substrate and proposed coatings were examined to provide data on acceptability from the cleanliness perspective.

Coupon Sample Testing

Several in-house test coupons were examined to replace the conversion coating coating. All sets of test coupons included one 7075 series Al and Alca Plus cast Al, both with similar machine finishes. The first coupons consisted of 0.000050in Gold (Au) outer layer over 0.0002in electroless Nickel (Ni). The second set was bare Al, the third was 0.0001in electroless Ni, and the fourth set was 0.0006in clear anodize. Each coupon was tapelift sampled to quantify the potential sloughing characteristics of the coating. The tapelift sampling is a method of detecting the particle distribution on a surface. In employs an in-house generated procedure that is acceptable for quantifying the IEST-STD-CC1246D surface cleanliness level.

Table 2 summarizes the results for the particle distribution for each of the four sets of coupons. The highlighted columns show those samples that exhibited low particle sloughing characteristics. Although Au/Ni showed no particles, Electroless Ni was eventually selected because it required one less step in the processing time.

Nickel Plating the Alca Plus Al Laser Benches

The next step after the coupon testing was to actually coat the Alca Plus Al flight benches. Because the conversion coating needed to be removed from the benches via a chemical (acid etch) removal process, a concern of etching the cast material was considered. Because Alca Plus is a cast material, it is porous in nature. The solution was to increase the Ni coating thickness to 0.0002in and later to 0.0004in to cover the substrate material surface. The 0.0004 thickness resulted in a sufficient coating that evenly plated the surface when visually inspected for particles.

Figure 3 shows the approximate 6in by 9in optical bench coated with 0.0004in Ni. Table 3 shows laser benches with serial numbers (S/N) 1,2, and 4 that were coated with 0.0002in Ni. S/N 1 showed a significant particle distribution compared to S/N 2 and 4. A visual difference in the coating appearance was also evident, which indicated an uneven Ni coating. As a result, S/N 1 had an additional 0.0002in Ni added. Table 4 shows the

Table 1. Conversion Coating on Aluminum and Alca Plus Cast Aluminum

Component	Particle Distribution Number	Range (μm)	Estimated Mil-Std 1246 Level	Comments
Laser Bench S/N 3	96 74 28 4 2 1	5-15 (μm) 15-24 (μm) 24-36 (μm) 36-70 (μm) 70-150 (μm) 150-300(μm)	500	MT 474, Particles mainly identified as Conversion Coating
Interior Housing	6 4 4 5 3 1	5-15 (μm) 15-24 (μm) 24-36 (μm) 36-70 (μm) 70-150 (μm) 150-300(μm)	750	MT 475 All Conversion Coating particles

Table 2. Summary of Coupon Tapelift Samples

Coupon	Particle Distribution		Estimated	Comments
	Number	Range (μm)	Mil-Std 1246 Level	
Au/Ni 7075 Al	None			Sample 515
Au/Ni Alca Plus	None			Sample 516
Bare 7075 Al	2 1	5-15 (μm) 24-36 (μm)	80	Sample 517, All Al particles
Bare Alca Plus	7 1 3	5-15 (μm) 15-24 (μm) 24-36 (μm)	108	Sample 518, All Al particles
Ni 7075 Al	[illegible] [illegible]	5-15 (μm) 15-24 (μm)	82	Sample 519, All Al particles
Ni Alca Plus	[illegible]	5-15 (μm)	57	Sample 520, All Al particles
Anodize 7075 Al	1	5-15 (μm)	57	Sample 521, All Al particles
Anodize Alca Plus	3 1 1	5-15 (μm) 24-36 (μm) 150 (μm)	196	Sample 522, All Al particles

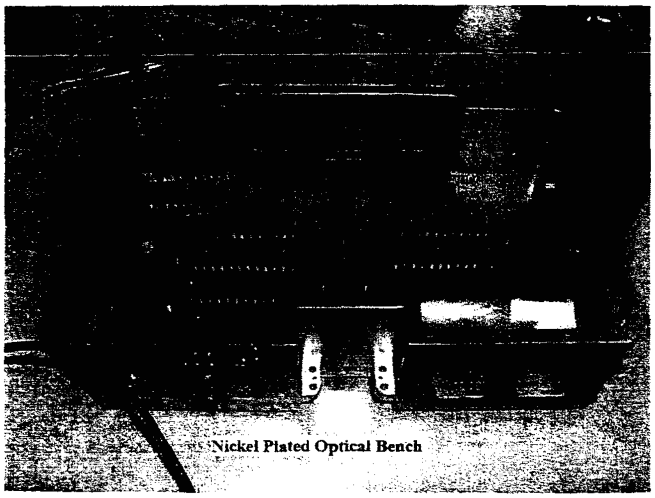

Figure 3 – Ni Plated (0.0004in) VCL Optical Bench

additional 0.0002in Ni to S/N 1 for a total thickness of 0.0004in Ni. S/N 6, the spare bench, and S/N 3 were coated with 0.0004in Ni. Comparison of S/N 1 in Table 3 and 4 show the improvement in particle count with the additional 0.0002in Ni. The results in Table 4 showed an acceptable cleanliness level for the Alca Plus laser benches.

Series 7075 Al Housing

Although the coupon testing for the 7075 Al was acceptable, the housing was not coated due to concerns with sealing surfaces. Figure 4 shows the housing divided into the optics and electronics cavity. The grooved surface identified as the o-ring sealing surface had a tolerance specifically sized for the given o-ring. Any additional thickness could invalidate the pressure testing previously performed on the seal integrity. Even though 0.0004in Ni in the groove was a small thickness addition, the main

Table 3. Electroless Ni (0.0002in) Laser Benches 1, 2 and 4

Laser Bench	Particle Distribution		Estimated	Comments
	Number	Range (μm)	Mil-Std 1246 Level	
S/N 1	23 2 2	5-15 (μm) 15-24 (μm) 24-36 (μm)	155	Sample 528, All Al particles
S/N 2	1	5-16 (μm)	57	Sample 526, All Al particles
S/N 4	1	5-15 (μm)	57	Sample 527, All Al particles

Table 4. Electroless Ni (0.0004in) Laser Benches 1, 3 and 6

Laser Bench	Particle Distribution		Estimated	Comments
	Number	Range (μm)	Mil-Std 1246 Level	
S/N 1	1	24-36 (μm)	94	Sample 535, Metal particle
S/N 3	1	36-70 (μm)	133	Sample 536
S/N 6	1	24-36 (μm)	94	Sample 537

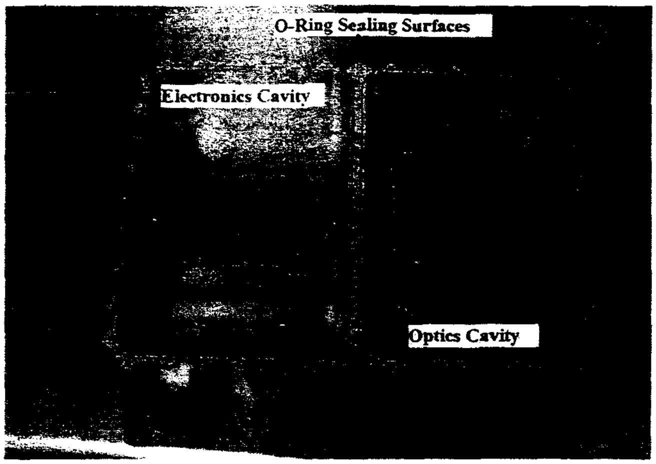

Figure 4 – VCL Housing

concern was the removal process of the conversion coating. This could potentially create more "peaks" and "valleys" in the substrate material. The acid etch used in the removal process could not be controlled accurately enough to alleviate concerns about the groove tolerance. If too much material in the groove was removed, the cavity seal may become invalidated, thus raising the optics cavity pressure and rendering the laser non-operational during its mission. Since the design and testing program was too schedule driven to warrant another series of pressure testing, the only solution was to create a method to capture the conversion coating on the housing to preclude particle redistribution. This was accomplished by masking the exposed surface area within the optics cavity with Y966 acrylic adhesive Kapton® tape. The vacuum baked acrylic tape was applied prior to installation of the laser bench into the optics cavity. The surface area was approximately 90% covered with the Kapton® tape, and eliminated a major concern with conversion coating sloughing during vibration ground testing and launch vibration/acoustics. Figure 5 shows how the Kapton® was applied to the internal housing surfaces. This approach to a particle containment fix was not considered ideal, but it alleviated the immediate concern with sloughing.

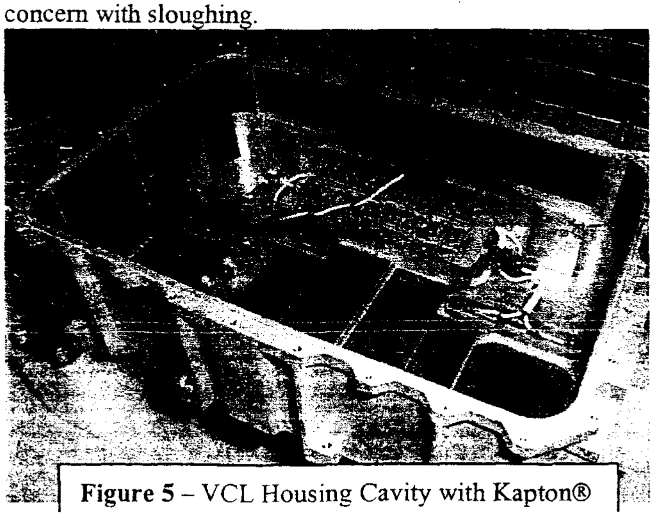

Figure 5 – VCL Housing Cavity with Kapton®

3. MLA Instrument

Brief Spacecraft and Laser Unit Description

MLA is an instrument on the Mercury MESSENGER mission conducted by the Johns Hopkins Applied Physics Laboratory (APL) in Laurel Maryland. The MLA instrument uses a passive Q-switch design that will measure the topography of Mercury. The key mission goal is to better understand how our own Earth formed by examining the elemental makeup of Mercury's crust, charged particles around Mercury's magnetosphere, measurement of atmospheric gases and minerals in surface materials, magnetic field, and surface imaging. Figure 6 shows the major components of the MLA instrument without the multilayer insulation thermal protection system.

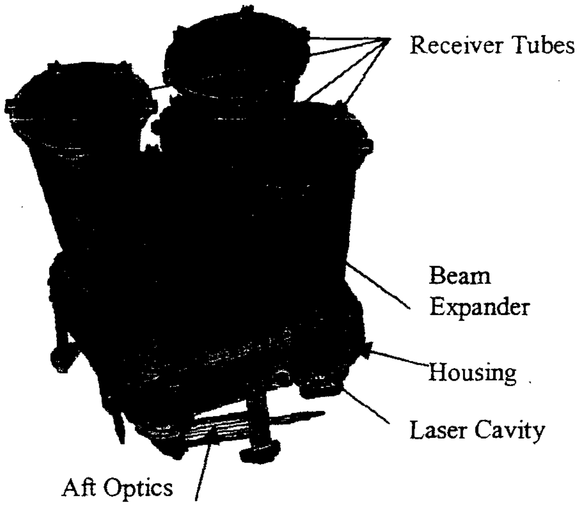

Figure 6 – MLA Instrument

MLA will orbit Mercury with a 12-hour elliptical orbit and a laser survival temperature of -20° to +40 ° Celsius. It will take 5 years to reach orbit with a one-year operational lifetime. Figure 7 shows the laser bench populated with optical components. The bench is approximately 3.5in by 5.5in, and is designed to operate in a vacuum environment.

The laser bench and housing material is machined instrument grade Beryllium (Be) I220H Grade 2 with a Ni and Au coating. The Ni was applied a thickness of 0.0002in with 0.00005 Au over the Ni. The receiver tubes and beam expander underwent a proprietary beryl-coat conversion coating process to preclude reaction of the beryllium with air during ground processing.

Cleanliness Requirements for MLA

Because MLA will operate in a vacuum environment, thus allowing for a more effect molecular transfer mechanism, a

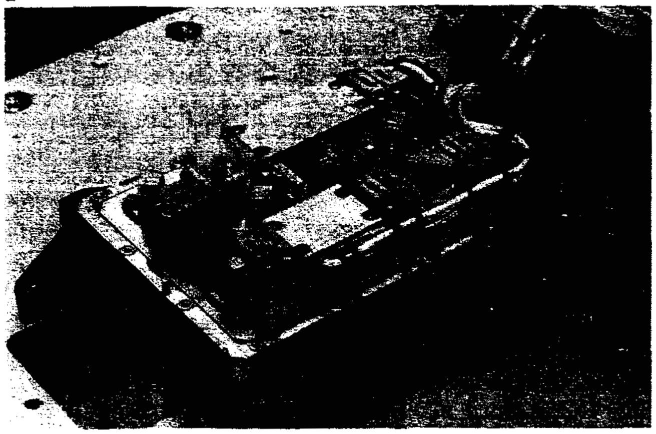

Figure 7 – MLA Laser Optical Bench

very stringent surface cleanliness verification and vacuum bakeout strategy was employed. The surface cleanliness requirements were Level 100, per IEST-STD-CC1246D, with a goal of Level 50 for particles, or %obs. of 0.0002. Molecular thickness was set at A/2 or the approximate equivalent of 0.5 $\mu g/cm^2$, e.g. 50Å, assuming a density of 1.0 g/cm^3. During the assembly of the laser optical cavity, an airborne cleanliness requirement of Class 10,000 or better was required. The data indicates that Class 100 during assembly and integration of optical components was achieved. A relative humidity requirement between 30 to 50 percent was maintained.

Another key was each part that went into the laser shown in Figure 7 was verified using hexane (Fisher Optima grade) rinse analysis with Fourier Transform Infrared Analysis (FTIR) to a criteria of: <1.0 percent unidentified peaks, <2.0 percent hydrocarbon peaks, and no trace silicone peaks. In some cases, gas chromatograph mass spectrometry (GC/MS) was performed to identify contaminants to ensure trace contaminants were low outgassing. The overall precision cleaning analysis required 625 individual analyses to complete one laser and spare components. In order to avoid a particle-sloughing event, such as with the VCL instrument, selected particulate verification was accomplished on the housing, receiver tubes, beam expander, and laser bench to ensure surface coatings adhered. Even though the beam expander and receiver tubes are external to the laser cavity, we did not want to contaminate the Shapphire windows on the receiver tubes and beam expander. The cleanliness requirements for these components were Level 400A, where A is approximately equivalent to 1.0 $\mu g/cm^2$.

Outgassing Requirement for MLA

In addition to surface cleanliness requirements, an outgassing requirement was applied to the entire laser interal cavity components as well as the beam expander and receiver tubes. Although the start of any flight program considers the American Society for Testing and Materials (ASTM) E595 outgassing screening test, many programs that are sensitive to contamination need to extend to actual in-situ outgassing measurements of components for at-use temperatures. The MLA specific outgassing requirement was satisfied through a number of vacuum bakeouts performed at the highest temperature predicted for survival or as the material of the component allowed. A temperature-controlled quartz crystal microbalance (TQCM) was used to certify acceptance for outgassing. A TQCM measurement was considered acceptable if a delta-delta frequency ($\Delta\Delta f$) of less than 5 Hertz per hour per hour (Hz/hr/hr) averaged over a 5 hour period was achieved. This measured the rate of change that a component was outgassing and indicates when the majority of the volatile material was diminished. Table 5 shows the outgassing achieved for both the rate (Δf) and the change in the rate ($\Delta\Delta f$).

The requirement was based upon general experience with other flight programs where molecular outgassing to stringent levels was a concern. The inherent risk associated with such similarity for outgassing requirements was the inability to understand the molecular transport for a specific geometry. A model of the internal laser cavity molecular transport could determine viewfactors of critical contamination sensitive surfaces and calculate expected depositions on those surfaces. However, budgetary constraints did not allow for such modeling.

Ni/Au and Beryl Coat on Beryllium

Table 6 lists the particle distributions we detected on the MLA components. These data helped to confirm that the surface coatings were inherently sufficient to meet established cleanliness requirements. The idea was to eliminate any concerns that surfaced on VCL with respect to the elevated temperatures of a conversion coating. Even though the coating processes are significantly different in application and chemistry, past concerns warranted a quick check on the cleanliness stability of the inherent coating. The results are well within the original cleanliness requirement of Level 100, but short of the Level 50 goal. An additional measurement was made to ensure that high temperature vacuum bakeouts were not acerbating any potential particle sloughing. The components identified as laser optical cavity-post vacuum bake and laser cavity- sealing surface, were sampled and met the Level 100 cleanliness.

Table 5. Summary of Outgassing for major MLA Components

Vacuum Baked Component	Outgassing Change in Rate ($\Delta\Delta f$)	Outgassing Rate (Δf)
Beam Expander Assembly	< 5 Hz/hr/hr	< 10 Hz/hr at 75° C
Filter Assembly	< 5 Hz/hr/hr	< 5 Hz/hr at 30° C
Housing	< 5 Hz/hr/hr	< 10 Hz/hr at 25° C
Aft Optics	< 5 Hz/hr/hr	< 10 Hz/hr at 40° C
Receiver Tubes	< 5 Hz/hr/hr	< 20 Hz/hr at 55° C
Laser Bench Assembly	< 5 Hz/hr/hr	< 80 Hz/hr at 48° C
Electronics Boards	< 5 Hz/hr/hr	< 27 Hz/hr at 40° C
Flight Harness	< 5 Hz/hr/hr	< 55 Hz/hr at 60° C

Table 6. Summary of MLA Components

Components	Particle Distribution		Estimated Mil-Std 1246 Level	Comments/(sample number)
	Number	Range (μm)		
Housing Exterior	24 3 1	<5 (μm) 5-15 (μm) 15-24 (μm)	95	Required level is 400, (SAI-281)
Laser Cavity	2 3	<5 (μm) 5-15 (μm)	80	(SAI-279)
Laser Optical Bench	1	5-15 (μm)	57	(SAI-276)
Laser Optical Bench- Post vacuum bake	None			No detectable particles, (SAI-322)
Laser Cavity- Sealing Surface	1 1	5-15 (μm) 15-24 (μm)	83	(SAI-324)
Receiver Tube	13 4 2	5-15 (μm) 15-24 (μm) 24-36 (μm)	150	S/N 1 tube, (MT-34)

4. CONCLUSION

Many considerations comprise a cleanliness program, and coating-type issues represent a fraction of the overall cleanliness program for this paper. Both laser instruments required a very stringent cleanliness program for surface particulate and molecular contaminants to avoid potential damage to optical components from such contaminants. Although MLA was the more contamination sensitive of the two lasers, there were coating concerns with both lasers because surface cleanliness is highly dependent upon the coating and substrate selection.

In the case of the VCL laser, we learned of the potential for conversion coating particulate contamination. We have traditionally used conversion coatings for spaceflight applications on aluminum. However, when lasers require stringent surface cleanliness levels along with high temperature vacuum bakeout, the conversion coating coating is limited to +60 °C as a maximum bakeout temperature. The chromate conversion coating also generated higher particle distributions as demonstrated by the data in Table 1 on the cast aluminum material, such as Alca Plus. Although the Ni on the Alca Plus improved the cleanliness level, it had a higher particle distribution than the Ni/Au coating on Be for MLA and the Ni on series 7075 Al for VCL. These data in Table 1 & 6 indicate that the two coatings consistently met the Level 100 cleanliness. In addition, there were no deleterious effects from the high temperature vacuum outgassing testing on the surface cleanliness level as noted in Table 6.

Although performance damage threshold requirements were not established for the given laser wavelength and energy, selecting an appropriate coating selection to preclude potential contamination is evident for the limited survey that was completed. A more in-depth survey of materials and coatings would probably yield a larger selection of acceptable candidates. In the cases presented in this paper, project schedules were the limiting factor for the coupons used.

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BIBLIOGRAPHY

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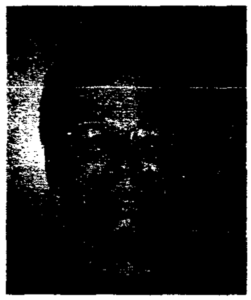

Joe Hammerbacher *has been working in the Contamination Control Field at NASA/GSFC for eighteen years. Since arriving in 1985 he has supported numerous projects including, Cosmic Background Explorer (COBE), Geostationary Operational Environmental Satellite (GOES), Tropical Rainfall Measuring Mission (TRMM), Earth Observing System Aqua Satellite and the Hubble Space Telescope (HST). His experience includes, precision cleaning of flight hardware, evaluating/ certifying cleanrooms, writing contamination procedures, FTIR and GC Analytical Analysis, microscopic evaluation of various materials and surfaces. During his time at GSFC he functioned as the Group Leader from 1995-1999 for the Mantech Contamination Control Group before joining Swales Aerospace in 1999.*

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