JPL ANALYTICAL CHEMISTRY LABORATORY **S262**
Analytical Chemistry and Materials Development Group 3531

To: **Robert Taylor, Helena Armandula** **11/13/07**

From: Mark S. Anderson *(edited by Dennis Coyne to identify the parts and concerns)*

Subject: LIGO Molecular Contamination Analysis: *Large Aluminum Parts for the HAM-ISI System for Enhanced LIGO at LLO*

Purpose

Part surfaces were swab-sampled on site and submitted (11/12/07) for chemical analysis. This was to determine the level and identity of molecular (oily) contamination on the surface of parts.
Samples 1 through 4 were taken at AstroPak on cleaned large plates which had stains. The stains were created by prolonged exposure to the machining fluid/coolant after machining by the manufacturer, Zimmerman. AstroPak's standard wet cleaning process did not remove the stain. The FTIR sampling is to determine if the stain has a high hydrocarbon content (unlikely).
Sample 5 is a sample of the 3 SEI HAM-ISI parts that have been cleaned and air baked at Caltech (also destined to be parts of the HAM-ISI at LLO for Enhanced LIGO). These later parts are part of traveler # E070236-00, SEI HAM Parts, "First Half of Crate 1".

Method

The analytical swabs consisted of extracted fiber-free lens tissue using Freon-TF solvent. The low volatility residue (LVR) was analyzed using Diffuse Reflectance/ Fourier Transform Infrared (DRIFT/FTIR) spectroscopy. FTIR provides chemical functional group information for quantitative analysis and qualitative identification of materials (1). The analysis followed the ACL-120 procedure that complies with IEST-STD-CC1246D and is sensitive to the most stringent level (A/100).

Results and Discussion

The sample #4 had a moderate level of aliphatic hydrocarbon oil. The other samples had relatively low levels of oily residue (2). A level of 1 microgram per square centimeter (µg/cm^2) corresponds to an average film thickness of 100 angstroms (assuming a density of 1.0).

Sample	Chemical Functional Group	Amount * µg/cm^2
1	AHC	0.05
2	AHC	0.06
3	Trace AHC	~0.03
4	AHC, Trace Ester	**0.9**
5	Trace AHC	~0.02

AHC: Aliphatic hydrocarbon, base oil of common lubricants
Esters: commonly from plasticizers, fingerprints
µg/cm^2- micrograms per square centimeter
* Results based on 4in^2 (26cm^2)

References

1. M. S. Anderson et al "Analysis of Semi-Volatile Residues Using Diffuse Reflectance Infrared Fourier Transform Spectroscopy" in Optical System Contamination: Effects, Measurements, and Control VII; July 2002, edited by Phillip T. C. Chen and O. Manuel Lee; Proceedings of the SPIE, Vol. 4774, pp. 251-261, (2002).

2. The last mono-molecular layers are more complex to describe when cleaning or analyzing. Carbon/hydrocarbon based substances are known to rapidly (~1 hour) accumulate on most, if not all, freshly exposed surfaces. This "adventitious" carbon is well documented in clean rooms and vacuum systems and compositionally varies by environment. Adventitious carbon is a discontinuous layer of approximately ~0.2-1 nanometers thick or **~0.02** to 0.1 $\mu g/cm^2$ (for $\rho = 1$). The last mono-layer fractions may in some cases be strongly adsorbed to the surface as a "corrosion" layer. Therefore solvent based sampling methods may not remove these corrosion fractions. This is further complicated if the surface is porous. When specifying cleanliness level to less than level A/10 IEST-STD-CC1246D (0.1 $\mu g/cm^2$) these monolayer effects become more significant. See also: H. Piao and N. S. McIntyre, "Adventitious carbon growth on aluminum and gold–aluminum alloy surfaces", Surface and Interface Analysis, *Surf. Interface Anal.* 2002; 33: 591–594.

3. A typical solvent wipe has a detection limit of ~0.005 $\mu g/cm^2$ of removed residue from a $100cm^2$ sample. Note this limit is well below the adventitious carbon level. Lower limits are possible using modified methods. The wipe blanks are at levels less than 10% the amount removed from the sample and this is subtracted from the reported sample amount. High blanks (greater than 10%) are noted in the report.

16 November 2007, Interpretation and direction from Dennis Coyne:
The AstroPak FTIR results for the set of samples 1 through 4 (large plates cleaned by AstroPak), especially sample 4, are poor. However, FTIR results (prior to air baking) for parts cleaned by AstroPak have also been marginal in the past as well. I suspect (but can not prove) that it is their cleaning process and not the stains per se. The stains are likely an oxide. I have asked Ken Mason (SEI Lead) to have AstroPak perform a phosphoric acid etch, which they claim will clean the stain off the parts. This will also have the parts go through additional cleaning.

Regarding Sample 5: The FTIR result shows a clean sample, but a single sample for 3 large parts to represent a large bake load is under-sampling too much. These loads are approved (in traveler E070236-00) but more samples should be used in future loads.

Dennis Coyne

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