

Cost-Effective Method for Measuring Contamination Effects

Chang-Keng Liu* and A.P.M. Glassford†

Lockheed Palo Alto Research Laboratories, Palo Alto, Calif.

A simple experimental method is demonstrated for determining the change in solar absorptance (α_s) of contaminated spacecraft surfaces as a function of deposit surface density. The method applies to contamination originating as material outgassing or vaporization. A quartz crystal microbalance is used to monitor the amount of contaminant condensing on a test thermal control surface in vacuum. Test surface spectral reflectance is measured in the laboratory environment before and after contamination using a standard laboratory spectrophotometer. Reflectance is integrated over the solar spectrum to obtain α_s . The data clearly show the relationship between α_s and contaminant mass per unit area. The observed data scatter appears to be assignable totally to the spectrophotometer measurements.

Introduction

THE increasing use of advanced spaceborne sensor systems and astronomical experiments, as well as the need for longer spacecraft lifetime, has required parallel development of the technology of contamination assessment and control for optical and thermal control surfaces under orbital conditions. In general, this development has sought to establish a more systematic and comprehensive approach to contamination control by systematically analyzing the controlling physical mechanisms. A specific example of this activity is the method for determination of allowable contamination levels. In the systematic method, maximum allowable changes in surface properties of thermal control surfaces and/or optical train for a given system component are determined from system analyses and the specified system performance tolerances. The maximum allowable amount of contamination in mass per unit area is then found from a general data base which relates change in surface properties to contaminant surface density, contaminant source, and environments.

At the present time few contaminant density/property change data are available mainly because of cost and measurement problems. To provide such data, projects are underway in many aerospace organizations to build comprehensive contamination effects simulation and measurement facilities. These facilities enable a test surface at arbitrary temperature to be contaminated to a known degree by any selected source and, if desired, exposed to solar radiation. Its optical properties can be measured without removal from the vacuum environment. These facilities are costly to build and operate, and difficulties have been reported in obtaining reliable data because of problems associated with performing the optical measurements *in situ*.¹⁻³ It may therefore be some time before a significant amount of data can be generated. However, while such facilities are ultimately necessary for generating a comprehensive data base, a considerable amount of reliable data can be obtained for a more restricted parameter range by using available proven apparatus and simple experimental

techniques. This paper describes such an experimental approach and presents some data to illustrate its effectiveness. In principle, a quartz crystal microbalance (QCM) is used to monitor the amount of contaminant collected by a test surface exposed to the outgassing flux from a source material in vacuum. The QCM and test surface are placed in a position of similar outgassing flux level and are controlled to the same temperature. It is then assumed that the deposition measured by the QCM is the same as that occurring on the test surface. After contamination the test surface is brought to 25°C and 1 atm pressure and removed to a laboratory spectrophotometer for optical property measurement. This technique is limited to contaminants which remain condensed at 25°C and 1 atm pressure, while the properties of the contaminated surface can only be measured at one temperature and pressure. However, all other simulation parameters can be varied.

The reliability of these measurements is influenced by the accuracy of the QCM and spectrophotometer and the validity of the assumption of equal deposit on both QCM and test surface. Both of these issues are addressed in more detail in Results and Discussion.

Apparatus

The apparatus used is shown in Fig. 1. It is located in a bell jar vacuum system capable of reaching pressures lower than 2×10^{-7} Torr. The principal components of the apparatus are a test surface holder, a QCM to record the mass deposition rate during the test, and a sample pot containing the outgassing source material. These components are linked thermally to a liquid nitrogen reservoir, and are completely surrounded by the cooled shrouding to reduce the background outgassing rate to undetectably low levels (less than 10^{-8} g/cm² per day). The QCM and test surface view the orifice through a hole in the shrouding with movable shutters. These shutters are controlled separately, permitting the incident flux to each surface to be interrupted when appropriate. The QCM and test surface holder are both placed at 7.7 cm from the sample pot orifice and are displaced 16 deg on either side of the normal line of sight of the orifice. Thus, they have the same view factor of the orifice and experience the same incident outgassing flux.

The QCM is a Celesco Model 700A unit, whose accuracy has been previously established.⁴ Its sensitivity is 4.43×10^{-9} g/cm² Hz, and its output is stable to better than ± 1 Hz over a period of days. The QCM test surface holder and outgassing source pot are all equipped with electrical resistance heaters and platinum resistance thermometers or thermocouples. Their temperatures can thus be adjusted and controlled over a wide range of values.

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*Research Scientist, Material Sciences Laboratory.

†Research Scientist, Material Sciences Laboratory. Member AIAA.

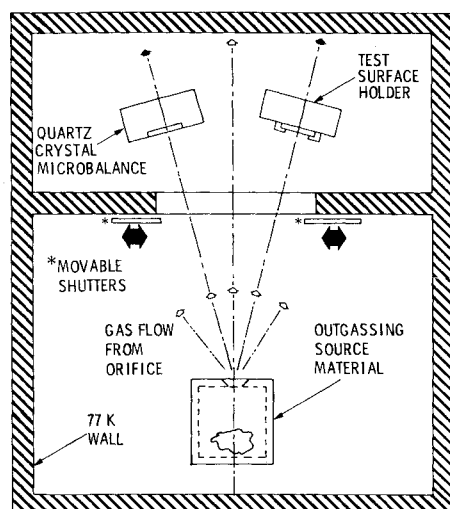


Fig. 1 Schematic of outgassing contamination study apparatus.

The surface properties were measured in a Cary Model 14 spectrophotometer, which measures the diffuse spectral reflectance and transmittance in the range of 0.27 to 1.8 μm . These properties are measured in air at room temperature, with a maximum uncertainty of ± 0.005 reflectance units. The solar reflectance can be calculated by integrating the spectral reflectance with respect to the solar spectral irradiance. The solar absorptance can then be calculated by subtracting the solar reflectance from unity.

The usefulness of the test approach was examined by making measurements of change in optical property as a function of magnitude of deposit for a range of outgassing source types, temperatures, and test surfaces.

Test Matrix

Two materials were selected as the outgassing source for the present study. These are described below.

RTV-560 is a methyl-phenyl room temperature vulcanizing (RTV) silicone rubber, manufactured by the General Electric Company, and used extensively in the Shuttle Orbiter's Thermal Protection System. The sample tested was prepared according to the manufacturer's instructions.

S-13 GLO is a low outgassing zinc oxide-silicate white thermal control paint for spacecraft hardware applications. It was supplied by the IIT Research Institute and mixed in accordance with the IITRI/NASA/MSFC specification No. 10M0183.

The surfaces tested were three optical solar reflectors (OSR's) used for thermal control. The OSR's were aluminized quartz (Al/OSR) and flexible OSR made from FEP Teflon, either silverized (Ag/FOSR) or aluminized (Al/FOSR). The combinations of outgassing source materials, temperatures, and test surfaces are shown in Table 1. The test surfaces were all maintained at 25°C.

Table 1 Test matrix

Test surface		Outgassing source	
Type	Clean property	Type	Temperature, °C
Al/OSR	$\alpha_s = 0.085$ to 0.113	RTV 560	125
Al/FOSR	$\alpha_s = 0.129$	RTV 560	125
		S - 1 3	
		GLO	125, 177
Ag/FOSR	$\alpha_s = 0.48$	RTV 560	125

Test Procedure

Before each test, the surface to be contaminated was cleaned and its solar absorptance was measured by the Cary Model 14 spectrophotometer. The clean test surface was then installed in the test surface holder.

A quantity of outgassing source material was weighed and placed in the sample pot. The amount of sample used was not a critical test parameter, but had to be sufficient to provide a measurable deposit on the QCM and the test surface. The apparatus was assembled, evacuated, and the liquid nitrogen shroud was cooled. The final pressure was 2×10^{-7} Torr or lower.

During evacuation, the sample pot was heated to a prescribed temperature, while the heat inputs to the QCM and test surface holder were adjusted to maintain their temperatures at 25°C as the rest of the apparatus cooled down. Steady-state conditions were obtained within about one hour after evacuation of the system. The shutters were then opened to allow the outgassing flux to impinge on the QCM and the test surface, both maintained at 25°C. The deposition rate was measured by the QCM reading, which was recorded as a function of time.

When the QCM had accumulated the desired amount of deposit, the shutters to both QCM and test surface were closed. The QCM reading was monitored to determine whether a significant amount of re-evaporation was occurring at 25°C. No such re-evaporation was observed for any of the materials tested. All heat inputs were then removed and inert gas was let into the system to bring the pressure up to 1 atm. When the system reached thermal equilibrium with laboratory air, the contaminated test surface was removed. Its solar absorptance was measured again and compared to that before the test. The QCM was exposed to the same pressure and temperature environment as the test surface throughout the postcontamination period. The amount of deposit on the test surface was thus assumed to be equal to the deposit mass per unit area on the QCM crystal at the time of solar absorptance measurement.

Results and Discussion

Figure 2 shows the change of solar absorptance as a function of contaminant deposit mass per unit area for the surface and contaminant combinations tested. The change of solar absorptance is given as a percentage of the clean value. This is the form of most use to thermal design engineers. For a uniform film deposit an exponential relationship between absorptance and deposit mass could be anticipated and in this case a log-linear plot would be appropriate from a phenomenological standpoint. It is known, however, that many outgassing product deposits from silicone-based materials can have the form of liquid droplets, which would cause departure from the exponential relationship. The log-log plot was therefore used, so as to achieve a compact graph.

The data for RTV-560 on all three surfaces show roughly linear form, with little scatter for Al/FOSR, more so for the Al/OSR and Ag/FOSR. The percentage change in α_s is about twice as much for Al/OSR and Ag/FOSR as for Al/FOSR.

For purpose of analyses, the data for RTV-560 products on Al/OSR and Ag/FOSR can be represented by a straight line. The observed data scatter about this line is due to a combination of uncertainties in measured optical properties and/or deposit mass densities. Because of the slope of the line the maximum possible uncertainty consistent with the observed scatter is only about 6% in optical measurement, but is as high as $\pm 50\%$ in mass measurement. The relative contributions of these two sources of uncertainty can be estimated by a more careful analysis of the measurement techniques and assumptions.

The Cary 14 spectrophotometer is a typical widely used laboratory instrument. A study of the accuracy and repeatability of a range of laboratory spectrophotometer is

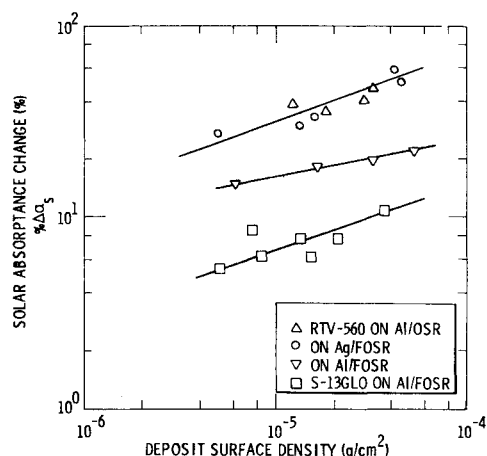


Fig. 2 Solar absorbance change of contaminated thermal control surfaces.

reported in Ref. 5. The repeatability observed on specific instruments for small groups of identical measurements lay in the range of ± 0.001 to ± 0.005 . For the specific Cary 14 used for these tests a conservative repeatability figure of ± 0.004 has been used, based on many years of experience with this instrument. Values of α_s for clean Al/OSR and Ag/FOSR lie in the ranges of 0.085 to 0.113, and 0.048, respectively. The percentage repeatability for the spectrophotometer used in these samples is thus in the range of 3.5 to 8.5%.

The determination of the mass on the test surface by assuming it to be identical to that condensed on the QCM is subject to a number of possible errors. The basic accuracy of the QCM in measuring the amount of material condensed on its surface is very high.⁴ Mass densities of the order of 10^{-8} g/cm² can be detected reliably. The outgassing flux impinging on the sample and QCM is known to be the same to within $\pm 1\%$. A prior calibration test was performed in which the test surface holder was replaced by another QCM, and both QCM's were cooled to liquid nitrogen temperature. With DC 704 silicone oil in the sample pot as a molecular source, the angular relationship of the sample pot to the two QCM's was adjusted during collection by remote control to equalize the flux condensing on each QCM.

The QCM surface and test surface are in general dissimilar and their temperatures may differ by $\pm 0.5^\circ\text{C}$ during a test. Neither of these situations will result in significantly different deposition rates on the two surfaces if the bulk re-evaporation rate of the impinging species at 25°C is negligible. However, if the re-evaporation rate of the contaminant species is of the same order as the impingement rate, significant differences in deposit magnitude may result. For example, a 1-deg change in temperature at 300 K could produce a 12% change in net deposition rate on similar surfaces, assuming a typical latent heat/activation energy of 20,000 cal/mol. The nature of the condensing surfaces can influence adsorption and nucleation processes, as well as the deposit morphology. These effects could also be significant when both the condensation and re-evaporation rates are finite. However, as noted in the previous section no net re-evaporation was observed in any of the present tests when the impinging flux was interrupted, and so the influence of small temperature and surface differences should be negligible.

Summarizing the preceding analysis, the observed uncertainty level for the overall measurement is within the range of that for the spectrophotometer alone. On the other hand, the error in mass measurement required to account for the scatter appears to be much too high. Hence, it is concluded that the observed scatter must be due mainly to the limitations of the spectrophotometer.

The data for S-13 GLO products on Al/FOSR show the linear behavior similar to that found for the RTV-560 products. In this case the maximum data errors are about

$\pm 2\%$ in the optical measurement, or about $\pm 70\%$ in mass measurement. For Al/FOSR with a clean value of α_s of 0.129 this corresponds to an error in absorbance measurement of ± 0.003 . Again, this error is very close to the limiting capability of the spectrophotometer, whereas the implied mass measurement error is unacceptably high. This tends to confirm the conclusion that the reproducibility of these measurements is limited by the performance of the spectrophotometer.

Using the same technique as that described in this paper, additional measurements have been made of the contamination effects due to the outgassing products of various spacecraft materials on selected thermal control surfaces and solar cell cover glasses.⁶ Although these measurements were not systematic, the magnitude of the observed scatter also seems to be attributable to the spectrophotometer.

Conclusions

The data clearly show the increase in solar absorbance resulting from an increase in surface contamination. There is some scatter to the data which appears to reflect the limit of resolution of the spectrophotometer, rather than errors in mass measurement or accuracy of simulation. This measurement approach is therefore concluded to be valid, for the conditions and materials tested, and achieves the best accuracy possible with standard laboratory equipment. This conclusion suggests an explanation for the difficulties that have been reported in obtaining reliable data from in situ contamination simulation facilities. In most cases such measurements are made using standard laboratory spectrophotometers modified with the integrating sphere placed in the vacuum chamber. This requires use of transfer optics and remote specimen manipulation, both of which would introduce further sources of experimental error in what appears to be the major source of experimental uncertainty.

In most space applications the thermal control surfaces would also be exposed to solar radiation, so contamination-induced degradation should also be measured after such exposure. The described technique is readily adaptable to making this measurement. Since the test surfaces must be removed from the contamination chamber for optical measurement they can be subjected to any treatment, including insertion in a standard radiation facility for any desired period.

Acknowledgments

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