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REDUCTION OF CALCOFLUOR IN SOLITHANE CONFORMAL COATINGS OF PRINTED WIRING BOARDS

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ABSTRACT

An investigation on the outgassing of a pigment employed as a fluorescent medium in conformal coatings has been performed. The conformal coatings in question are used to protect printed wiring boards from environmental hazards such as dust and moisture. The pigment is included in the coating at low concentration to allow visual inspection of the conformal coating for flaw detection. Calcofluor, the fluorescent pigment has been found to be a significant outgasser under vacuum conditions and a potential source of contamination to flight hardware. A minimum acceptable concentration of Calcofluor for flaw detection is desirable. Tests have been carried out using a series of Solithane™ conformal coating samples, with progressively lower Calcofluor concentrations, to determine the minimum required concentration of Calcofluor. It was found that the concentration of Calcofluor could be reduced from 0.115% to 0.0135% without significant loss in the ability to detect flaws, while at the same time significant reductions in Calcofluor outgassing and possible contamination of systems could be realized.

INTRODUCTION

Calcofluor[1] is mixed into Solithane[2] to provide detection of flaws when the Solithane-Calcofluor conformal coating is exposed to UV light. It has been found that the use of Calcofluor in Solithane conformal coatings for circuit boards may be a large source of contamination. Because of this, interest has been expressed in developing a specification for an acceptable minimum percentage Calcofluor mixed with Solithane which is still adequate to detect flaws in the conformal coatings under UV. The normal amount of Calcofluor used in a Solithane compound has been 100g of part A(Solithane 113), 74g of part B(Solithane 113-300), and 0.2g Calcofluor. These weights are referred to as a 0.2 pbw (parts by weight) Calcofluor mixture. Likewise the same mixture of Solithane components with 0.1g Calcofluor would correspond to the 0.1 pbw mixture. In order to determine the amount of Calcofluor required to detect defects in the Solithane conformal coatings, a set of 4 mil thick coatings composed of different percentages of Calcofluor were applied to aluminum test plates. The plates were visually inspected for defects and fluorescence. The set of plates included the following amounts of Calcofluor mixed with 20.0 g of Solithane 113 and 14.8 g of Solithane 113-300:

Plate Number	Calcofluor (g)	Calcofluor (pbw)*	%Calcofluor
1,2	0.0	0.0	0.0
3,4	0.04	0.2	0.115
5,6	0.02	0.1	0.0574
7,8	0.01	0.05	0.0287
9,10	0.01	0.05	0.0287
11,12	0.005	0.025	0.0144
13,14	0.0027	0.0135	0.0078

* The units pbw are a common designation used locally within GSFC, they refer to the weight in grams used per 100g of the A component of Solithane and 74g of the B component.

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METHOD

The study consisted of mixtures including 0.2, 0.1, 0.05, 0.025, and 0.01 pbw. The goal of the study was to determine the minimum amount of Calcofluor required for effective flaw detection in conformal coatings of circuit boards prior to exposure to vacuum conditions. The second step was to determine if this reduction in the quantity of Calcofluor would actually reduce the level of contaminant present under vacuum conditions. To determine if this was so a series of tests was carried out in a Vacuum Industries test chamber at pressures on the order of 10^{-7} and 10^{-8} torr. The 0.2, 0.1, 0.025 and 0.013 pbw test plates as well as the pure Solithane test plates were exposed to vacuum for a period of 24 hours at a temperature of 75°C. During the test, condensates were collected on a cold plate at a temperature of -25°C. After completion of the 24 hours cycle the cold plate was rinsed and condensed material was weighed to determine the collected volatile condensible mass (CVCM). Then the residue was analyzed by both Infrared and Gas Chromatograph-Quadrupole Mass Spectometry. The IR and GS/MS analysis was used to obtain the recommendation on the level of Calcofluor to be used.

A diagram of the instrumentation is shown in the appendix. The Vacuum Industries chamber includes a two stage pumping system with a mechanical backing pump and a diffusion pump which can achieve pressures down to approximately 10^{-8} torr. Backstreaming from the diffusion pump was controlled using liquid nitrogen cooled cold trap and a liquid nitrogen controller.

The chamber itself is an 18 inch diameter bell jar which contains a hot plate, cold plate and thermocouples. All of the hardware within the bell jar was chosen with the fewest number of outgassants possible, so as to hold contamination to a minimum. The hot plate rested on an adjustable scissors jack and was raised so that there was approximately 1 inch spacing between the sample and the cold plate. The hot plate itself was a simple resistance coil hot plate controlled by an external controller. The controller ran through a high limit controller which detected the hot plate temperature and would open an alarm condition switch if the temperature went above a preset value. The controller signal was sent to a 110 volt, 10 ampere solid state relay which would open and close the switch from the power source to the hot plate depending on the temperature of the hot plate. Temperatures were measured on the hot plate, the cold plate and the sample itself using a series of type T thermocouples.

The 8x6 inch cold plate consisted of a simple aluminum plate with copper tubing coiled at 1 inch intervals across the top surface. These coils carried ethylene glycol based antifreeze which was cooled by a Neslab chiller to temperatures of -25°C. This plate was covered with an aluminum foil wrap which had been cleaned using a series of isopropyl alcohol and chloroform rinses. The foil served as a surface for condensation which provided and easy method to collect and rinse the contaminant

Once the series of plates were made the first step in analysis was visual inspection of the samples for defects. Upon visual inspection of the conformal coatings under UV illumination it was apparent that as little as 0.0135 parts by weight was sufficient for detection of flaws in the conformal coating. Each coating was prepared with included flaws (nylon fibers and brush fibers) to simulate possible defects. The nylon filament was 2 mil in diameter and cut into lengths of approximately 1-2 mm. The brush hair was approximately 1 mil in diameter and was cut to lengths of less than 1 mm. In addition a certain amount of defects were created during the preparation technique. It was found that although the level of fluorescence was reduced somewhat in both the 0.025 and 0.0135 pbw Calcofluor, the fluorescence was sufficient to detect both the included flaws as well as naturally occurring defects in the coating. This demonstrated that the level Calcofluor could be reduced to as little as 0.0078% rather than the 0.115% currently used.

The following observations were made during visual inspection of the test plates under UV:

0.2 pbw Calcofluor: The entire plate demonstrated signs of fluorescence. All the included flaws (nylon fibers and brush hair) could be seen with relative ease. The plates also exhibited numerous spots which demonstrated fluorescence. These were attributed to areas of higher Calcoflouor concentrations.

0.1 pbw Calcofouor: The same observations were seen as in the 0.2 pbw Calcofluor plates. In additon a dark, pin sized hole was observed penetrating the entire thickness of the coat in plate #6. However, when the edge of a probe was used to scratch the surface of the plate, an increase in fluorescence at the scratch was observed.

0.05 pbw Calcofluor: All the included flaws could be seen with relative ease. In additon bright spots of Calcofluor were visible.

0.025 pbw Calcofluor: The bright spots due to undissolved Calcofluor were less visible, and the fluorescence was slightly less intense, but the flaws were clearly visible.

0.0135 pbw Calcofluor: The level of fluorescence was similar in intensity to the 0.025 pbw. All flaws were visible.

0.0 pbw Calcofluor: Demonstrated no signs of fluorescence from the Solithane itself. Nylon fibers could be seen because of their own fluorescence.

Once the visual inspection was completed each plate was placed in the vacuum chamber and heated to a temperature of 75°C for 24 hours at pressures ranging from 10^{-6} to 10^{-8} torr. The actual pressure was somewhat time dependent and also depended to some degree on the amount of material outgassing from the Solithane sample. Condensables were collected on the aluminum wrapped cold finger at -25°C. These deposits were rinsed from the aluminum after the test was completed with alcohol and chloroform. The collected material was weighed and then analyzed by both IR and GC/MS analysis. The results of this analysis are discussed in detail in the Results section.

RESULTS

The following data on the collected deposits and the related CVCM (mass collected/initial mass of Calco + Solithane) were obtained.

Sample Plate Initial Conditions and Results

% Calcofluor	Wt. Solithane(g)	Wt. Calcofluor(mg)	Collected Wt.(mg)	%CVCM
0.115	3.34	3.834	7.0	2.10E-03
0.057	1.18	0.678	3.7	3.14E-03
0.014	1.13	0.162	2.8	2.48E-03
0.008	2.34	0.182	4.8	2.05E-03
0.0	0.00	0.000	3.8	1.05E-03

pbw Calcofluor	% Calcofluor
0.2	0.115
0.1	0.057
0.025	0.014
0.0135	0.008

0.0 0.0

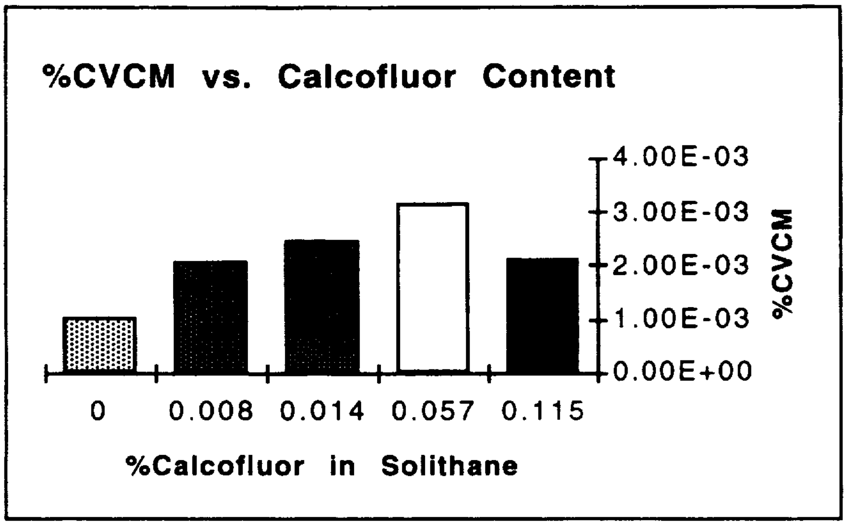

Figure 1

This results indicate that the highest concentration of Calcofluor produces by far the greatest accumulation of contamination. However there was not a linear trend towards less contamination as the concentration of Calcofluor was decreased. One possible explanation was that below a certain percentage of Calcofluor, the contamination was mainly produced by the Solithane, and that variations in the thickness of the Solithane coating itself lead to variations in the weight of the outgassants collected.

The information illustrated in Figure 1 indicates that as the Calcofluor content is increased the level of contamination also increases. However the highest Calcofluor concentration did not follow this trend. An explanation may be that the conformal coating was somewhat thicker on the sample plate for the 0.2 pbw(0.115%) sample. The thicker coating could limit the diffusion of Calcofluor out of the material leading to lower collected weights. Additional analyses were carried out to provide correlation between increased Calcofluor and CVCM.

The contaminant was analyzed by IR and GC/MS to determine the composition of the released products and quantify the amount of those materials present. Upon completion of the 24 hour test the collected deposits were rinsed into an evaporation dish and weighed once dry. The residue was then analyzed by FTIR and GC/MS. FTIR analysis primarily focused on detection of absorption peaks at 1711 and 1612 cm^{-1}. These wavelengths are indicative of the presence of Calcofluor, but 1711 cm^{-1} proved to be less useful because the several common esters cause absorption at that frequency as well. By comparing the height of the Calcofluor peaks at 1612 cm^{-1} relative to the peaks caused by silicones in the same spectra, the following charts (fig. 2) were obtained.

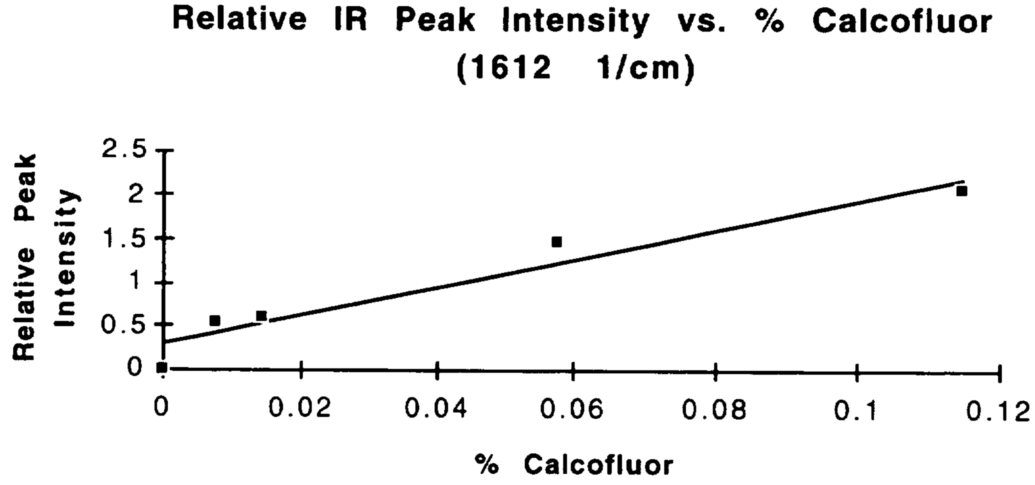

Figure 2

Figure 2 displays the Calcofluor peak at 1612 cm^{-1} relative to the peak caused by silicone pump oil at 1216 cm^{-1} as a function of the concentration of Calcofluor in the conformal coating. A second plot Figure 3 was produced by comparing the peaks due to Calcofluor relative to a peak at 1177 cm^{-1} from silicone pump oil, as a function of the Calcofluor concentration. This diagram is shown below.

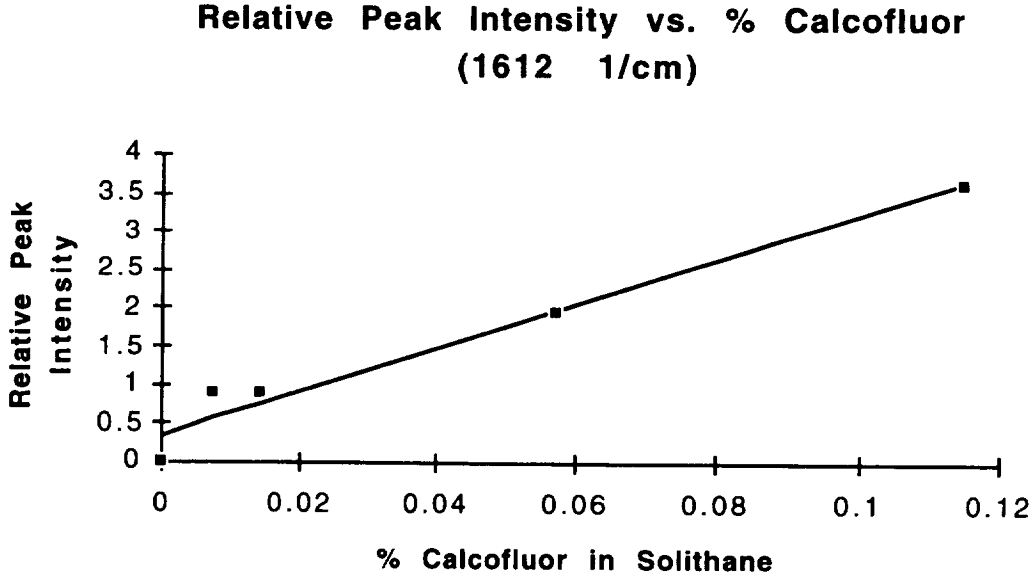

Figure 3

GC/MS analysis of the residue showed similar results. Each sample was normalized to a silicone peak caused by backstreamed diffusion pump oil present in each sample. The silicone peaks were assumed to be relatively constant and thus could be used to compare with the peak areas for Calcofluor. This resulted in the following curves (fig. 4 and 5):

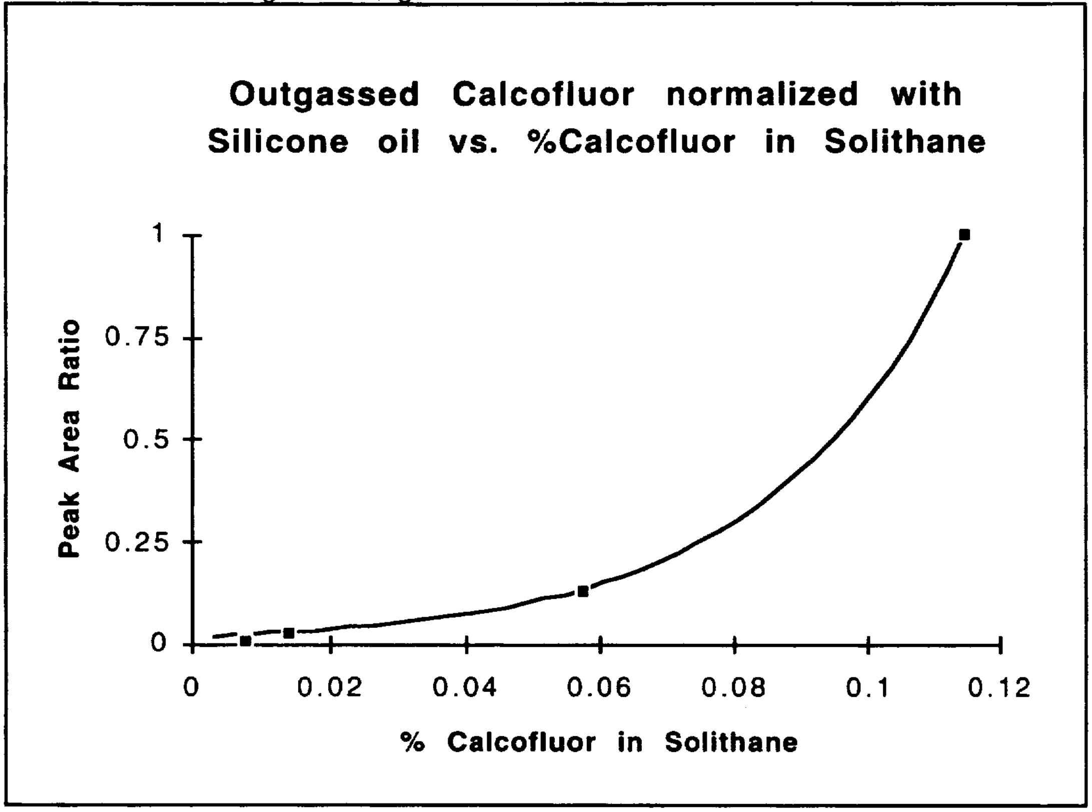

Figure 4

Calcofluor peak area normalized to diffusion pump oil

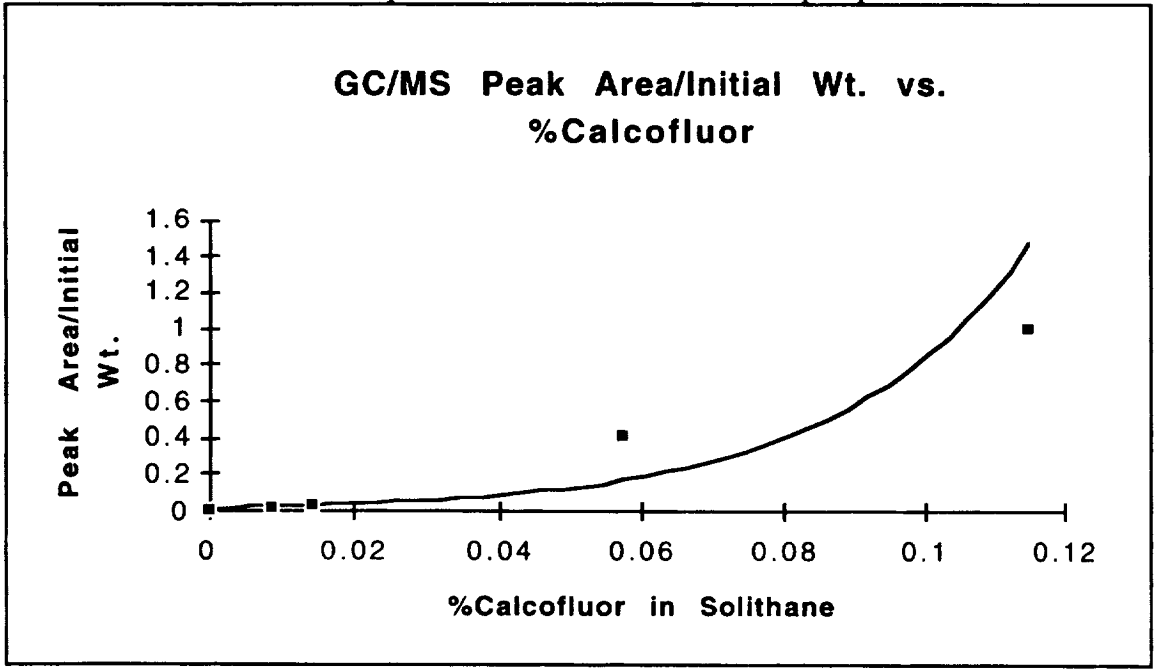

Figure 5

Calcofluor peak area normalized to initial weight

This method of analyzing the contamination is perhaps the most accurate and yet shows the most dramatic increase in the level of contamination as the concentration of Calcofluor was increased.

CONCLUSIONS

After investigating the fluorescence of different concentrations of Calcofluor in Solithane conformal coatings, as well as the contamination caused by outgassing Calcofluor in these coatings, one can recommend that the concentration of Calcofluor be reduced from 0.115% Calcofluor (0.2pbw) to 0.0078% Calcofluor (0.0135pbw). Using only 0.0135 pbw Calcofluor significantly reduced the contamination caused by the fluorescent pigment yet proved to be sufficient for flaw detection in Solithane conformal coatings.

The investigation has also indicated the methods to be followed in evaluating outgassing originating from compounds. The peak area indications of GC/MS with respect to peak areas of some reference compound can be helpful in establishing the amount of a specific contaminant over a series of initial sample concentrations.

REFERENCE

[1.] Calcofluor is a trade name of American Cyanamid Co. for a fluorescent powder consisting of 4-methyl-7-diethyl amino coumarin (MDAC).

[2.] Solithane is a trade name of Thiokol Chemical Corp. for a polyurethane coating.

APPENDIX

VACUUM CHAMBER & TEST SET-UP