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Feasibility Study of OSEE Inspection for Flux Residue on Electronic Assemblies  
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No Clean Flux Residue Detection: Optimization of Extraction/Ion Chromatography  
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How Clean is Clean: Effect of No-Clean Flux Residues and Environmental Testing Conditions on Surface Insulation Resistance  
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ABSTRACT
The technique of Optically Stimulated Electron Emission (OSEE) is considered as a method of inspecting printed circuit based electronics assemblies for flux residue immediately following production soldering. The technique has been used for several years by NASA and its contractors in the refurbishment of solid rocket motors for the Space Shuttle. The application to copper substrates and soldered copper substrates has shown sensitivity to small amounts of residues of some solder fluxes. The technique was extended for inspection of insulating substrates used in printed wiring board (PWB) construction by altering the measurement procedure to include charge replacement, thereby attaining measurement reproducibility. The results indicate that OSEE inspection of electronic assemblies for flux residues is feasible. An inspection based on this technology subjects the inspected object only to photons of ultraviolet light and immersion in an inert gas, such as argon. It is potentially rapid enough to provide 100% inspection of boards processed on a production line, and it has potential spatial resolution of less than 1 micron.

INTRODUCTION
With a new generation of fluxing, soldering and cleaning technologies in the electronic industry, the entire assembly process needs to be reevaluated from a yield, quality and reliability perspective. From the standpoint of long-term reliability, it is important to distinguish between innocent chemical residues and "dangerous" ones. Cleanliness detection methods, of course, change with the use of new solder fluxes and pastes. Several on-line detection methods popular in the rosin flux/CPC cleaning era have become questionable in the emerging technologies using "no-clean"; or low solids flux soldering.

In 1992, a team was formed under the umbrella of the National Center for Manufacturing Sciences (NCMS) to establish a correlation between the type and quantity of residues left on a printed circuit board (PCB) and the electrical performance of a circuit The goal was to perform analyses of specific chemical residues and establish the impact of these chemical residues on electrical function. The guiding principle used is that contamination induced failure occurs when a chemical concentration exceeds a critical value.

In order to maintain consistency in the study, two important issues were addressed. First, model fluxes were used to avoid variables (such as extraneous additives) associated with using commercial fluxes. Second, contamination-by-design was used, so dose-response curves were generated by using specimens that were contaminated with "controlled" amounts of specific chemicals. Theoretically, analysis of the "type" and "quantity" of the contaminants can be done in one of two ways: extraction based analysis and direct analysis.

Extraction based Analysis
The most common and universally accepted analytical methods for residue detection on electronic assemblies are all based on extraction. These methods depend on optimizing an extraction method for removing the residue from the PCB substrate, followed by analysis of the extract. All extraction measurement based methods provide an average over the entire area of the circuit board. So far, two extraction based detection methods were investigated by the group: Ion Chromatography (IC) and Solvent Extract Conductivity (SEC). An optimum method of extracting and detecting low solids flux residues was developed by this team through extensive round robin testing 1.

Direct Analysis Of Residue On Substrate
This is the "ideal" way of measuring cleanliness. An electronic assembly operation almost always results in uneven distribution of flux/solder residues over the PCB cleaning area. In general areas near or underneath large components are harder to clean. Concentration of "harmful" residues across conductor lines will be harmful to the electrical long-term performance of the circuit board. Therefore, the group considered it necessary to investigate available techniques for direct analyses of residues on PCB's. Among the methods considered are some which have proven to be of great utility in surface science investigations. Methods considered include Fourier Transform Infrared Microscopy (FTIR), Optical Techniques with Automatic Image Analysis, Optically Stimulated Electron Emission (OSEE), X-Ray Photoelectric Spectroscopy (XPS), Auger Electron Spectroscopy (AES) and black light inspection 2.

One of the techniques, Optically Stimulated Electron Emission (OSEE) is presented in this report. In particular, its detection of contamination with a Rosin Mildly Activated (RMA) flux
was studied. The work presented here includes results on copper substrates, soldered copper substrates, and an extension of the technique to permit OSEE measurements on circuit board insulating substrate material. While the results so far look promising, the studies are preliminary, and development continues.

OPTICALLY STIMULATED ELECTRON EMISSION (OSEE)

OSEE is a technique used for inspecting surfaces for contamination. It has been of restricted use in some specialty areas, in which it has generally been successful owing to its ease of application, rapidity of response and simplicity of interpretation. It has not had wider use because in some instances it is highly variable and difficult to interpret. Its uses have been mostly as a quality control indicator in production environments where the sources of contamination are few and reproducible and the inspected surfaces are primarily metals. The work reported here represents an extension in the application of OSEE.

Descriptive Introduction To OSEE

OSEE is a measurement technique which is based on the photoelectric effect, in which a photon of light with a sufficiently short wavelength, or high energy, interacts with the material constituents of a solid surface it strikes to eject an electron \(^3\),\(^4\). These so-called photoelectrons can be detected with a sufficiently sensitive electrometer if they are collected on a positively charged anode. A schematic of OSEE as used for inspection of Shuttle Solid Rocket Motor casings is shown in Figure 1\(^5\). The essential elements in the OSEE measurement process are shown as a light source, a positively biased electrode connected through an electrometer, and a conductive return path to return charge to the sample equivalent to that removed.

In previous work \(^6\),\(^7\), it has been shown that the field strength at the sample surface is an important determinant of OSEE current, although currents are generally low enough that field modification by the photoelectrons, the space charge effect, is not substantial. With a low-pressure mercury discharge lamp, it has been shown that the very short (185 nm) component in the illumination is responsible for the majority (~95%) of the OSEE current observed. This wavelength is known to interact with atmospheric oxygen to produce ozone, which is frequently detectable by its odor near an OSEE measurement apparatus. It is also known to interact with water vapor. The resulting reaction products are themselves highly reactive, and they engage readily in surface chemistry with the sample, changing surface characteristics which affect OSEE current generation capacity. These tendencies lead to variability in the OSEE current from the sample which is not related to the initial surface condition. This variability sometimes obscures the interpretation of OSEE data. Previous work has shown this variability to be largely suppressed by purging the illuminated volume of an OSEE measurement with argon gas, which is non-reactive. The previous work also established that the intensity of short wave radiation emitted from a low-pressure mercury arc lamp is dependent on lamp current and bulb temperature. Once these interferences and sources of variability are taken into account, the OSEE measurement of a given surface gains both reproducibility and stability. Figure 2 indicates the stability and reproducibility, which has been achieved in two successive OSEE measurement cycles on a cleaned copper circuit board surface. The two runs are the second and third in a three run set. The rise and fall in the OSEE current are caused by a opening and closing a shutter, which admits light to the sample between 50 s and 450 s following the start of a run.
The OSEE measurement from a given substrate has been shown to be sensitive to very small amounts of some field contaminants in particular oils and greases. Previous work has shown that part of the sensitivity is due to absorption of the incident light by the contaminant. Many organic compounds have a very high absorptivity to light at 185 nm. Some other compounds and mixtures become photo conductors under ultraviolet radiation. For these, an alteration of the work function of the surface occurs, increasing the sensitivity of OSEE to contamination at very low levels. In summary, previous work on metallic substrates has shown OSEE to be a sensitive indicator of small amounts of contamination on metal substrates. In any particular case, pending a general theory of OSEE response, it is necessary to develop a dose-response curve for each new contamination-substrate combination for which contamination monitoring is desired.

Application Of OSEE to Circuit Board Inspection
To apply OSEE to printed circuit board inspection, several issues must be considered. First, the substrate consists of two types of surfaces, a soldered copper surface and an insulating surface. Second, the contaminant should be a known substance. In the case of circuit boards, the contaminant is solder flux, a known substance in each particular production system. The response of OSEE on soldered copper is shown in Figure 3, which depicts three successive measurement runs on each of two samples in a laboratory environment. The data suggest that the state of a clean surface is indicated by an OSEE current in a range of 10% of the reading on the initial exposure to illumination. This provides the first indication of measurement reproducibility needed to produce a viable inspection on soldered surfaces. The stability of the initial indication, obtained in 5 seconds with the limited bandwidth equipment used, suggests that an OSEE inspection system can be made to produce rapid inspections on a production line and so be a practical inspection tool.

Extension Of OSEE Method To Include Insulating Surfaces
Virtually all previously reported work on OSEE response is associated with metallic surfaces, for which a reference state of electrical potential can be established with a simple return wire from the sample to the instrument, completing the electrical circuit. With an insulator, no such reference is readily available, yet casual observation with the commercially available OSEE instrument often indicates that OSEE currents are generated when the probe is moved into proximity with an insulating surface. If there is a current produced, there is some hope of producing a viable contamination measurement.

Starting with the knowledge that casual positioning of an OSEE probe next to an insulating surface produces a response, an apparatus was constructed to mimic the metal substrate with a surface made of circuit board insulator by mounting it in an aluminum frame backed with conductive foam. The frame with its circuit board sample was mounted in the same position as a metallic sample. The result of this modification is to expose the insulating surface only to the ultraviolet light while maintaining a uniform electric field in the measurement region. With this apparatus, the OSEE current from the insulator was shown to be unsteady, decreasing from an initial value over time. It was also demonstrated that original current was not the same from time to time. It was shown that this current had some properties in common with metal substrates. The current ceases on closing the shutter, indicating that it is a photo current. It consists predominantly if not entirely of negative charges, as little current is emitted and collected when the sample is positively charged. The current is produced primarily by the 185 nm line of the mercury lamp rather than the lower energy spectral components, a result obtained by placing appropriate filters in the light path. These results are consistent with currents originating from the release of relatively loosely bound electrons under the action of the ultraviolet light. The difference between the metal and insulating case is that the electrons, once removed, are not again replaced, and so the current decreases with time. Presumably, the surface becomes positively charged from the locations previously occupied by electrons.

Figure 4a shows the results of three successive OSEE measurements on FR-4, an insulating substrate in common use in printed circuit boards. Not surprisingly, the OSEE current decreases in each run with time. This can be attributed to a decrease in the population of accessible electrons in the illuminated region as those available are removed from the system. The surface also is left with a net positive charge, decreasing the field in the measurement region. With a good
insulator, the charge partly remains on the surface between runs, so that the measured OSEE current depends on the surface charge at the beginning of a measurement. This lack of reproducibility affects the credibility of the entire measurement, rendering OSEE as generally practiced, an impractical contamination measurement tool on insulators.

Examining the OSEE process, the only physical process which seems to be missing on insulating surfaces is the return of charges which have been removed by the measurement. In a metal, these charges are replaced from the pool of conduction electrons, which is itself replenished from the conducting return connection (Figure 1). Accordingly, for the insulating case of the circuit board substrate, a substitute method was used to replace the charges. The method, which is termed charge replacement, consists of measuring the total charge which passes through the electrometer during the measurement, and reversing the bias field following the measurement. This field reversal generally produces some current in the opposite direction.

One source of this current is stray light impinging on the collection grid, which becomes a source of electrons rather than a collector with the field reversed. The current is permitted to flow until an amount of charge has passed the electrometer equal to that which passed during the measurement. The measurement may then be repeated. The presumption is that the charges passing the electrometer during the reversal find the correct places on the surface by the attraction of the residual positive charge centers following the original removal of the charges. By employing charge replacement, reproducible OSEE runs were attained, as shown in Fig. 4b.

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**Figure 4.** a. Three successive OSEE runs on insulating circuit board material with simple repetition, showing non-reproducible current histories. b. For the same material, three successive OSEE runs using the charge replacement protocol between runs.
OSEE Dose Response Curve For Rosin Flux On An Insulator

With reproducibility established on a clean surface, the next step is to examine the effect of flux contamination on the insulating surface. This is done in Figure 5, which shows three successive measurements using charge replacement on FR-4 substrates with three levels of contamination by rosin flux, the three levels being characterized as none, light and heavy contamination. The runs with charge replacement all show high reproducibility. The initial value of OSEE response decreases as contamination is increased, and the slope of the curve with time also decreases, the highly contaminated case actually responding like a conductor, with essentially no decrease over time. Either the initial value or the slope in time seems to be a good indicator of contamination in these three cases. Because rapidity of response is desirable in a measurement, the initial response level is chosen as a contamination indicator. Figure 6 shows an experimentally determined dose-response curve for rosin flux on FR-4 with

Figure 6. Dose-response curve for Rosin Mildly Activated (RMA) Flux on insulating circuit board substrate material using OSEE.
the initial reading being used as the response indicator. The general character of the curve with a rapid decrease in the vicinity of 0-15 μg/cm² and a flat non-zero response increasing slightly at large contamination levels is similar to the dose-response curve seen on solid rocket motor inspections. It shows that OSEE is an effective contamination monitoring tool for this substrate/contaminant pair in the region of 0-15 μg/cm².

OSEE Inspection Status
The work to date has demonstrated that OSEE is reproducible on soldered copper substrates. It has extended the applicability of OSEE by virtue of establishing reproducibility of successive measurements to the non-conducting FR-4 substrate. It has also identified a contamination level range over which OSEE discriminates contamination amounts, while at larger contamination amounts, it simply indicates heavy contamination. The next steps in producing a viable inspection tool are to extend the sensitivity determination of OSEE to fluxes and other contaminants in addition to RMA, and to develop a method to examine a surface composed of soldered copper traces on an FR-4 substrate.

CONCLUSION
The feasibility of OSEE for inspection of electronic assemblies has been demonstrated. The detection limits associated with each method were established. In addition, the pros and cons of these methods as routine quality control inspection tools were discussed. OSEE was demonstrated to be a sensitive technique for detection of low levels of flux circuit residues on insulating substances. However future work including development of rugged OSEE instrumentation will determine whether the electronics assembly industry can accept this technique in a production environment.

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