OPTICALLY STIMULATED ELECTRON EMISSION (OSEE): A NON-INVASIVE TECHNIQUE FOR CONTAMINATION DETECTION

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Abstract

Optically Stimulated Electron Emission (OSEE) is a unique technique for non-invasive inspection of surfaces for molecular contamination. This technique utilizes a tool which utilizes ultraviolet radiation to create electron emission from a surface, resulting in a small current detected by the tool. Electron emission is dependent on the substrate's surface chemistry; hence the electron emission characteristics will change with the presence of a contaminant on the surface, generally by attenuating the signal. An Aerospace Sponsored Research program was conducted to explore potential applications of the OSEE technique. These and other applications are described, including surface inspection/verification prior to adhesive bonding of thermal insulation in rocket motor cases (Space Shuttle solid-rocket boosters); development of pre-weld cleaning/surface preparation; inspection of polished beryllium mirror surface for fingerprints; inspection of graphite/epoxy skins on aluminum-core honeycomb sandwich panels; detection of corrosive electrolyte on battery surface and development of battery cleaning procedure; and verification of plasma-etch treatment of an elastomeric material. Limitations and other considerations are also discussed.

OSEE FUNDAMENTALS

The release of electrons from surface exposed to various frequencies of light was observed as early as 100 years ago. In 1887, a clean zinc plate was noted to develop a positive charge when exposed to ult~"violet light. It was in 1890 that J. J. Thompson and P. Lenard explained this phenomenon as splitting out of negatively charged particles. They were able to show that the velocity of the emitted electrons was directly related to the frequency of incident light, while the number of charged electrons was related to the electron availability (a characteristic of the substrate).

In 1905, Albert Einstein further explained this phenomenon by postulating that light has some of the characteristics of a particle (photon), and that photon energy is inversely proportional to the wavelength. Therefore, shorter wavelengths of light impart higher energy to electron on a surface. The remaining factor is the work (energy) required to release the electron from the surface (work function). This "Photoelectric Effect" is represented mathematically as:

$$(E_k)_{max} = h\,v - \varphi$$

Here $(E_k)_{max}$ is defined as the maximum kinetic energy of a photoemitted electron; v is the wavelength of light; φ is the work function of the surface under investigation; and, h is a constant.

The photoelectric effect is the basis for the concept employed in the application of Optically Stimulated Electron Emission (OSEE): For an observed intensity of electrons discharged from a cleaned surface under a defined set of environmental conditions. any change in the surface electron emission characteristics can be attributed to changes in the surface condition specifically the presence of a contaminant.

There are two basic components that make up the OSEE equipment - a sensor and an electronics package. The functional diagram, Figure 1, illustrates the fundamentals of the sensor. An ultraviolet light source (approximately 240 nm), located within the sensor, irradiates the surface of interest. Electrons with sufficient energy to escape the surface are attracted to a collector (having a +45 volt bias), also located in the sensor. The resulting current is measured, amplified and displayed by an electronics package.

OSEE vs UV “BLACK LIGHT” INSPECTION

At present, ultraviolet (W) light, sometimes known as "black light", is often used in the manufacturing environment as a non-invasive surface inspection technique for molecular contamination. An investigation was conducted to determine the relative merits of this method as compared to the OSEE technique. The sensitivity of the "black light" method is dependent on:

(a) Molecular Composition of the Contaminant. UV "black light" inspection is based on fluorescence, which depends on the presence of carbon-carbon double and triple bonds in the contaminant. The more carbon-carbon multiple bonds present, the more the contaminant will fluoresce. Therefore, many contaminants will not be detectable, depending on their chemical molecu1ar composition.

(b) Amount of Contaminant. Our studies indicated that law levels of fluorescing contaminant are not readily detectable by the "black light" method.

(c) Operator Capabilities/Limitations. This method is subject to human operator limitations. Concerns range from whether Ambient lighting will affect the operator's ability to detect the fluorescence, to how alert he/she is when accomplishing this sometimes tedious task.

On the other hand, we have found that the OSEE technique is capable of detecting a wide variety of contaminants (e.g., oils - both hydrocarbon and silicone, fingerprints, and corrosive solutions) at very low levels. For example, D6AC steel contaminated with Tectyl 802k oil (used as a corrosion protection) was inspected using both OSEE and UV. The OSEE was able to detect as little as 1 mg/ft^2 while approximately 100 mg/ft^2 was required for even marginal fluorescence.

With regard to operator limitations, the OSEE effectively takes the operator "out of the loop". The display provides objective data, thereby avoiding the need for the operator's rather subjective observations and interpretations.

OPERATING PARAMETERS

Environmental parameters such as temperature and relative humidity were considered. In addition, inspection parameters such as scan rate, time (in relation to oxide formation), probe distance from the surface, and signal drift have also been investigated.

Temperature and humidity did not present themselves as issues in our assessment because our evaluations are performed in a laboratory where these parameters are relatively well controlled. However, drafts in the area of the OSEE sensor did present problems and a shield had to be utilized. In operation, the sensor is placed in close proximity to the surface being evaluated. In practice, a distance not greater than 0.25 inch has been found necessary to obtain a good signal.

The surface scan rate may vary based on the substrate, contaminant and gain settings of the equipment. (Higher gain settings will require longer response times.)

To overcome the problem of signal drift (e.g., due to changes in line voltage or equipment variations with time), a stable reference surface is used periodically for calibration of the equipment.

In addition, once the bare substrate and varying levels of contaminant have been characterized by OSEE, the electronics package can be set to comply with a pass/fail criteria. A typical display of varied levels of contaminant, as interpreted by an X-Y recorder, is shown in Figure 2.

OSEE APPLICATIONS

OSEE is utilized as a non-invasive technique for the detection of molecular contamination on critical surfaces. Developed by Dr. Tennyson Smith (Ref. 1) at the Rockwell International Science Center, this system was employed early in its history to determine the relative thickness of fluorocarbon 1ubricant on semiconductors and floppy discs during manufacture. Control of the film thickness is critical in achieving the desired reliability and wear life (Ref. 2).

Shuttle Motor Cases; Bonding of Insulation

After the Space Shuttle incident, NASA implemented a development program incorporating OSEE for cleanliness verification of critical bonding surfaces, in conjunction with the redesign of the Solid Rocket Motor manufactured by Thiokol Corporation (Refs. 3,4). In this application Conoco HD2 grease, a corrosion inhibitor on the steel cases during shipment and storage, had to be thoroughly removed prior to elastomeric insulation bonding to ensure a strong adhesive bond to the inner surface of the steel case. The development effort was based on correlating peel strength with the amount of contamination (mg/ft^2). The resulting graph (Figure 3) provided a means to determine the maximum concentration of contaminant allowable for the bond strength required. The OSEE was then calibrated to detect relevant contaminant levels. Having determined 25 mg/ft^2 as the maximum amount of contamination acceptable, a system for automated inspection was constructed. Robotics are used to traverse the OSEE sensor around the motor case interior, and the signal is processed by a personal computer. The result is a color map of the surface contamination present. For any area with >25 mg/ft contaminant, a re-cleaning is performed. Subsequently, Thiokol has further tightened their requirement at the edge bond areas to <15 mg/ft2 contaminant allowable.

Pre-Weld Cleaning and Inspection

Contamination on metal surfaces to be welded is another area of considerable concern. Contamination in the weld bead increases porosity of the weld, with a contaminant reduction in weld strength. As a result of this concern, OSEE has also been applied in the inspection of surfaces prior to welding (Refs. 5,6). In addition to inspection of Weldlite and other aluminum materials, various combinations of cleaning processes (solvents/detergents/methods) were evaluated by Martin Marietta Aerospace. A Taguchi experiment was utilized to establish the optimum cleaning method for contaminants common to the manufacturing process, e.g., marking pencil and oxidation. The result has been improved pre-weld surface preparation, and the ability to implement a quality control procedure prior to welding.

Beryllium Mirrors

The utility of OSEE for inspection of fingerprints on polished beryllium surfaces was evaluated for an optical mirror application. In the manufacturing environment, oils/salts associated with fingerprints generally go unnoticed until they have etched into the surface. OSEE easily detected the fingerprints on beryllium whereas visual detection was not effective.

Graphite/Epoxy, Composite Skins on Honeycomb Panels

The potential use of OSEE was briefly assessed for graphite/epoxy skins on aluminum honeycomb-core sandwich panels. The exterior cleanliness of the skins was evaluated. Both sides of an aluminum honeycomb-core sandwich were inspected, then solvent (Freon) wiped and inspected, twice. The results showed that the electron emission characteristics of the smoother surface, (i.e., the side in contact with the mold) was increased by the solvent wipe, while there was no apparent change on the rough side (in contact with the vacuum bag). Presumably some mold release had transferred to the surface and was removed by the solvent wipe. It was also noted that charge buildup occurs when using too slow a scan rate and is manifested by the display of erratic signals. This brief experiment suggests that some graphite/epoxy composite structures may be inspect able for contamination using OSEE.

Battery Vent Discs

Recently, a test program was performed to evaluate the potential of OSEE to detect thionyl chloride vapor on 316 stainless steel, assess the utility of a sodium bicarbonate cleaning procedure, and evaluate the effectiveness of a nonmetallic coating (Heresite P403) for corrosion protection. The 316 stainless steel is utilized in lithium battery vent discs with a critical thickness of 0.5 mil. The lithium batteries are filled with the thionyl chloride electrolyte solution under very dry conditions because of the hygroscopic/corrosive nature of the thionyl chloride. If the electrolyte (liquid or vapor) is deposited on the surface (e.g., during filling) and is not removed, battery surface (including the vent disc) will rapidly corrode when moved from the dry environment to ambient humidity conditions.

OSEE readily detected the presence of the electrolyte on the steel surface at levels that were not visible to the eye. In addition, the technique was extremely useful in establishing the suitability of the proposed surface cleaning process. On the other hand (not surprisingly), the OSEE was not effective in inspection of the Heresite P403 corrosion-protection coating. This is likely due to the nonmetallic nature of the Heresite coating. Unlike metals, electrons in non-metals are tightly held, thereby reducing the concentration of electrons available.

Other Applications

While the OSEE technique is expected to be 1ess effective for inspecting non-conductive, nonmetallic surface, there has been interest in evaluating its utility for certain nonmetallic materials used in space systems.

An elastomeric material was inspected using OSEE to determine its use for verification of a plasma-etch treatment prior to bonding. A comparison of OSEE readings for pre- and post-etch materials was performed. The OSEE readings for the post-etch sample were notably higher than those of the pre-etch material. This indicates that some elastomers (likely dependent on fillers) are OSEE-inspect able. However, it should be noted that charge buildup occurred when inspecting at too slow a scan rate, resulting in erratic readings.

Zinc Orthotitanate (ZOT) is a silicate thermal-control coating, often applied to vapor deposited aluminum on graphite/epoxy structures. ZOT is also utilized as a thermal-control coating on some space system radiator panels to provide low solar absorptance-to-emittance ratio (α_S/ε). Contaminants will cause the solar absorptivity, as, to increase when exposed to space radiation, thereby degrading thermal control performance. Common contaminants not visually detectable, such as pink pearl erasure and fingerprints, were detected by OSEE, albeit marginally.

In addition, non-conductors such as second surface mirrors and fused silica (with a dielectric coating) were evaluated. Contaminants for this assessment were those common to personnel such as fingerprints and spittle; however, no significant response to OSEE inspection was detected.

OTHER CONSIDERATIONS

Parameters such as scan rate, probe distance from the surface, and signal drift are variables that must be controlled to minimize the possibility of obtaining erroneous data. Humidity is likely an environmental parameter that needs to be controlled in as much as the electrons available in moisture could confuse the OSEE readings and will, over time, tend to alter the surface chemistry of substrates susceptible to oxide formation.

OSEE readings of the substrates described have generally been attenuated by contaminants present on the surface. However, same contaminating substances could conceivably enhance photoelectron emission, resulting in a higher OSEE signal than found with a bare substrate. While moderately conductive surfaces have demonstrated OSEE-inspect ability, experience indicates that OSEE functions best on conductive surfaces where electrons are available and easily replenished through an electrical ground.

CONCLUSIONS

OSEE has demonstrated great utility as a non-invasive inspection technique for the detection of molecular contamination and is a considerable improvement over "black light" inspection. The equipment can be modified and expanded to incorporate automation (especially useful for large surfaces). Preliminary efforts to obtain OSEE readings are required to characterize a new substrate and/or contaminant. OSEE can provide a quantitative measure of contamination present, providing the contaminant is known. For unknown contaminants the information would be represented as a deviation from the reading obtained for a clean bare substrate. 06EE cannot identify the nature of a contaminant.

The OSEE technique is regarded as a useful tool for rapid, non-invasive inspection to determine deviations from a standard clean surface. It is especially applicable to manufacturing process controls for which surface conditions are critical. Examples include bonding, coating and welding. It may also be applicable to optical and thermal control surfaces wherein presence of contaminants would cause the optical or thermal characteristics to degrade if not detected and removed. It has also been found useful in detecting non-visible fingerprints on beryllium mirror surfaces and corrosive electrolyte on battery surface, thereby aiding in upgrading the handling and manufacturing processes.

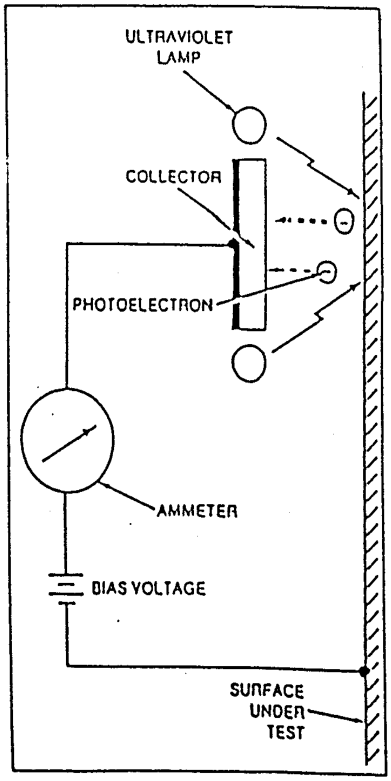

FIGURE 1

Detection of Tectyl 802A at Low Contamination Levels

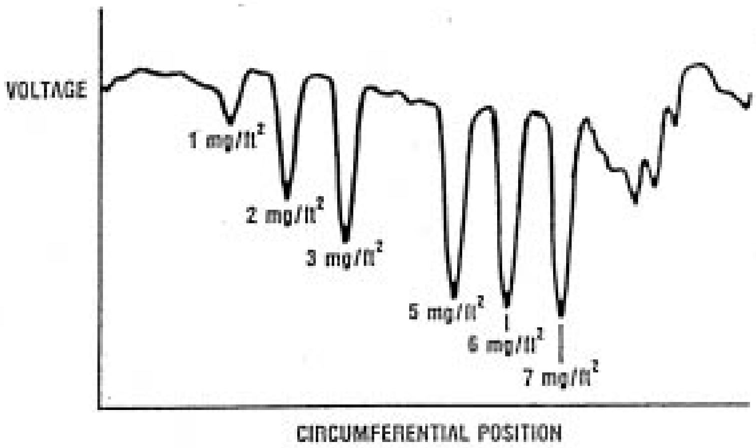

FIGURE 2

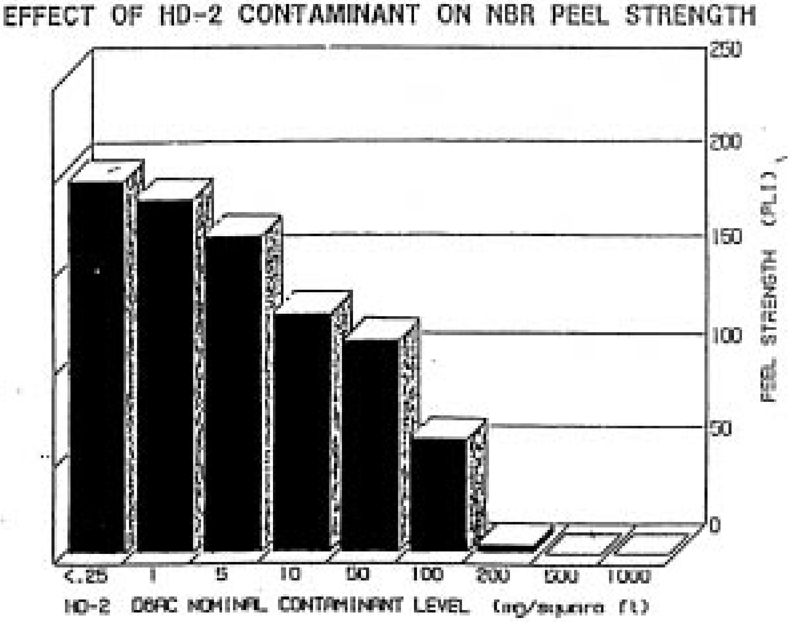

FIGURE 3

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