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Cleanliness Inspection Tool for RSRM Bond Surfaces

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ABSTRACT

Using optically stimulated electron emission (OSEE), Thiokol has monitored bond surfaces in process for contamination on the Redesigned Solid Rocket Motor (RSRM). This technique provides process control information to help assure bond surface quality and repeatability prior to bonding.

This paper will describe OSEE theory of operation and the instrumentation implemented at Thiokol Corporation since 1987. Data from process hardware will be presented.

INTRODUCTION

In 1987 NASA introduced a NDE method of bondline inspection to evaluate the consistency of substrate preparation prior to bonding. This technology is referred to as Optically Stimulated Electron Emission (OSEE), known at NASA Marshall Space Flight Center (MSFC) and Thiokol Corporation by the acronym "CONSCAN" for contamination scanning.

Tensile testing of the nozzle bondline has demonstrated sensitivity to contamination even at low levels of Conoco HD-2 grease ($< 5.0 \text{ mg/ft}^2$). Tapered double cantilever beam data demonstrates that Conoco HD-2 grease in the Dexter Hysol EA913NA bondline degrades fracture toughness to critical values under 10.0 mg/ft^2 . OSEE or Conscan has been baselined on D6AC steel for on-line contamination monitoring and has been used in RSRM manufacturing for the past 7 years.

THEORY OF OPERATION

OSEE is based on the photoelectric effect where electromagnetic radiation of the proper wavelength is impinged upon a substrate surface which subsequently yields electrons. The surface monitored by OSEE is irradiated with ultraviolet light with spectral lines at 2537 \AA and 1849 \AA . The electrons ejected from the surface are accelerated across a fixed distance or air gap by a potential on the collector around the mercury vapor lamp. The inspected surface is grounded with reference to the collector and the photoelectrons flow across the gap creating a photocurrent. The photocurrent, which is in the picoamp to nanoamp range, is detected by the sensor front end amplifier and displayed on the control unit or computer. The control unit displays the amplified signal in arbitrary units proportional to the photocurrent and are read as centivolts (CV). A contaminant on the surface will attenuate the photocurrent and produce a lower reading on the control unit.

The attenuation of the photocurrent, resulting from a contaminant on the surface is the result of the photoemitting substrate not being irradiated as strongly because of UV absorption in the contaminant covering the surface. The UV intensity is attenuated, as a function of Beer's law, as it passes through layers of contaminant. As the thickness of the layer of contamination on the a surface increases, the resulting intensity of UV on the underlying surface decreases and the photocurrent detected decreases

proportionately. Fewer photons reach the photoemitting surface and fewer electrons are emitted. The photoelectron flux passes through an accelerating field produced by the positive potential maintained on the collector ring around the UV source. See Figure 1.

The kinetic energy imparted to an electron by an incident photon is given by the Einstein photoelectric equation

$$E_e = h\nu - \Phi_0 = \frac{1}{2}m_0v_e^2$$

where $h\nu$ is the energy of the incident photon, Φ_0 is the energy required to remove an electron from the surface irradiated, terminating with zero velocity (known as the work function), m_0 is the rest mass of the electron and v_e is the velocity of the electron as it leaves the irradiated surface.

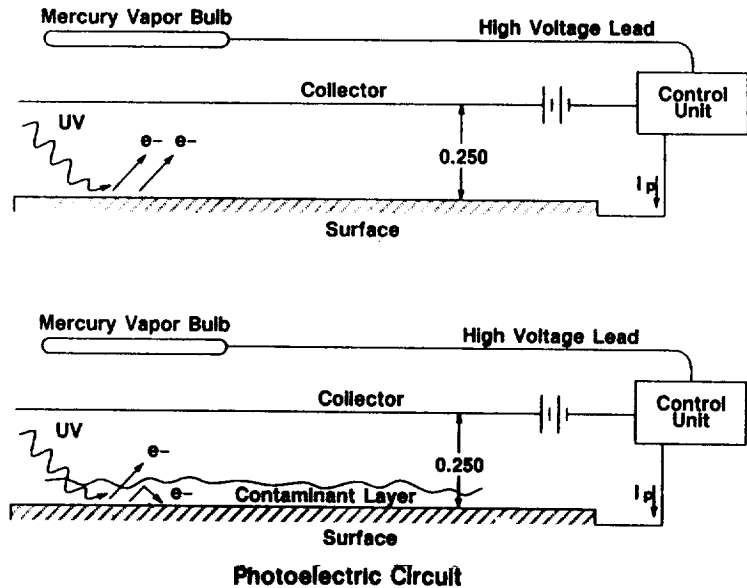


Figure 1

TECHNIQUE SENSITIVITY

There are many factors that influence OSEE measurements besides the cleanliness of the substrate being evaluated. The most significant of these is the sensor to surface distance. This causes a data variation of approximately 1% per 0.001 inch departure from a nominal standoff of 0.250 inch. This fact causes poor repeatability in data.

OSEE measurements are sensitive to not only surface contamination but oxidation/aging causes the signal to attenuate with time after grit blasting the surface. Studies of how the signal is attenuated on the same substrate with elapsed time have been conducted. See Figure 2.

OSEE response to various levels of Conoco HD-2 grease on D6AC steel was studied at NASA MSFC laboratories in 1987 under the direction of Dr. R. L. Gause¹. These correlation studies resulted in the formulation of a functional relation between Conoco HD-2 grease contamination level and OSEE level that became known as the Gause curve. This function was utilized in the implementation of OSEE in the RSRM production facilities at Thiokol to develop cleanliness criteria in case inspection.

Tests were performed at Thiokol to correlate OSEE response to varying levels of contamination on steel and aluminum bonding substrates and bond strengths to contamination levels.

OSEE is the most sensitive instrument to date for the detection of hydrocarbons and silicones on freshly grit blasted surfaces. It can evaluate large surface areas in reasonably short time periods but does not work on irregular surfaces.

OSEE is a good process control indicator when the data are compared at equivalent time-lines in the process. OSEE cannot, however, differentiate between oxides and contamination or between various benign and bond degrading contaminants.

It has been known since the implementation of OSEE that alternative, more analytic surface monitoring techniques would be required to solve in-line process questions that arise during routine OSEE inspections.

Over the past 7 years, other investigators have developed alternate surface contamination monitoring techniques and devices. Thiokol Science and Engineering department developed the diffuse reflectance infrared technology marketed through TMA Technologies as SurfMap II®. Bill Nerren of

NASA MSFC formed the Surface Contamination Analysis Team (SCAT) in 1991. One of the goals of this team is to develop a system for surface contamination analysis composed of two or more complimentary technologies to aid in on-line decision making about the acceptability of the cleanliness of bond surfaces. Thiokol is an active participant in the SCAT team at MSFC.

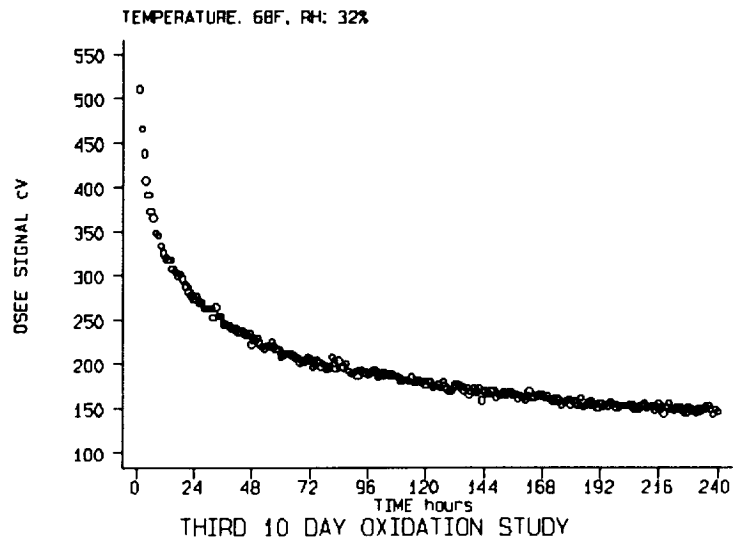


Figure 2

RSRM APPLICATION

CASE PREPERATION

OSEE was first implemented in the case preparation manufacturing area at Thiokol. The hardware is covered with a preservative grease, for shipment and storage, that must be removed before continued processing. The case cylinders are vapor degreased allowed to cool to ambient temperature and then they are spot checked with the manual OSEE probe to verify sufficient degreaser effectiveness. If a part fails to meet the criterion limit then the part is vapor degreased again before further processing can proceed².

The case components are sent to the assembly stand and assembled into casting segments. In this process grease is used on the O-rings for assembly and lubrication. The grease often gets smeared on the adjacent bonding surfaces and must be removed. The casting segment is then placed in the paint pit and the top coat, a two part epoxy paint, is applied. The ends of the segments are covered during this operation to prevent overspray from depositing on the internal bond surface. The painted segment is temperature cured in the pit.

The segment is then ready for primer application, which is the first step in the process of vulcanize bonding the NBR rubber insulation to the case wall. To assure bond surface cleanliness the case is inspected with OSEE prior to the primer application. See Figure 3.

NOZZLE BONDING

Engineering information data was collected with OSEE on metal nozzle housings to determine process criteria limits. Metal-to-adhesive bond failures in some test articles lead to extensive adhesive bond

testing. An OSEE baseline photoemission study was performed to determine what the maximum OSEE signal should be just after processing through the grit blast. These test results are still used to evaluate on-line performance. See Figure 4. The test has been performed on a daily basis recently to show OSEE values just after grit blast and 24 hours later. See Figure 5.

Three correlation studies were run to determine the functional relation between OSEE signal and contamination level. A control was run with each test and nominal levels of 5,10,20 and 30 milligrams of Conoco HD-2 grease per square foot were applied. The substrate was D6AC steel and tensile buttons were bonded using Dexter Hysol EA913NA epoxy adhesive.

The data were added to the data from the previous correlation studies and regression analysis was performed. The three new studies were also analyzed without previous data for comparison. The results were extremely similar. See Figure 6.

The regression of the whole set of data from all studies to date can be modeled with the function:

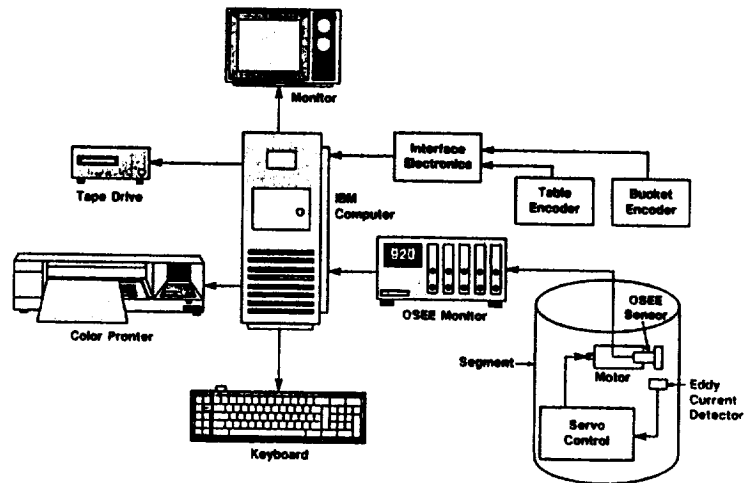
$$\text{OSEE} = -97 \ln [\text{contamination}] + 637. \quad (2)$$

The regression of the MSFC data alone resulted in the function :

$$\text{OSEE} = -103 \ln [\text{contamination}] + 640. \quad (3)$$

Ten milligrams of Conoco HD-2 grease per square ft. would be expected to read 414 centivolts.

The tensile strength was reduced 50% with the application of 5 mg/ft² of Conoco HD-2 grease. The further



AUTOMATED CASE SCAN SYSTEM
Figure 3

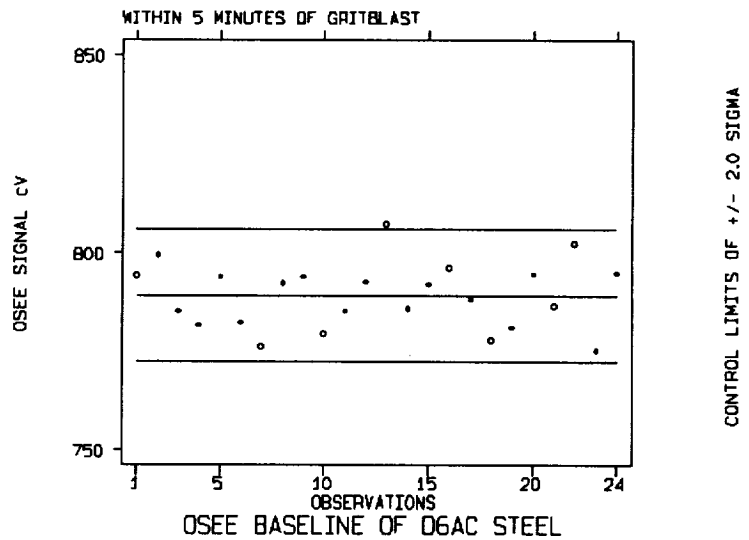


Figure 4

addition of contamination resulted in only slight further reduction of tensile strength. See Figure 7. The reason for this is not fully understood. The adhesive failure mode was greater than 50% with the application of 5 mg/ft² of Conoco HD-2 grease, approaching 100% with 20 mg/ft². See Figure 8.

This indicates that, although the tensile properties are not reduced catastrophically with the bondline contaminated, the failure mode is shifted to the metal-to-adhesive interface with low levels of Conoco HD-2 grease.

It was determined that cleanliness criteria should be implemented in the nozzle bonds. An OSEE limit equivalent to 10 mg/ft² was implemented in the process.

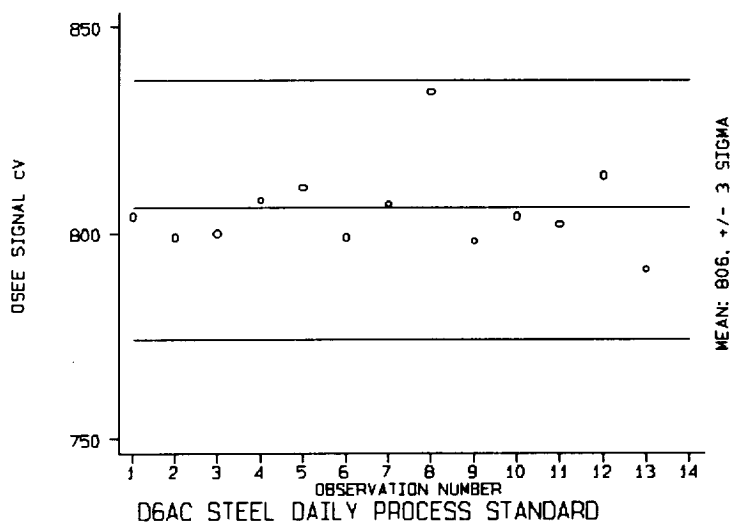


Figure 5

OTHER RSRM BOND SURFACES

OSEE has been implemented on the igniter, the weatherseal and stiffener stub and flex bearing shims.

The igniter is similar to a miniature solid rocket motor and is used to start the combustion in the full scale motor. NBR insulation is vulcanized to the internal and external surfaces of the igniter case. These surfaces are inspected for cleanliness with OSEE prior to primer application.

The weather seal is made of EPDM rubber vulcanized to cover the factory joint and seal out sea water upon retrieval operations. Each casting

segment is composed of two or, in the case of the aft segment, three cylinders. These cylinders are held together with hardened steel pins which are held in place with Inconel[®] retainer bands. The joint area is protected with masking tape during the top coat application and must be removed and the masking adhesive

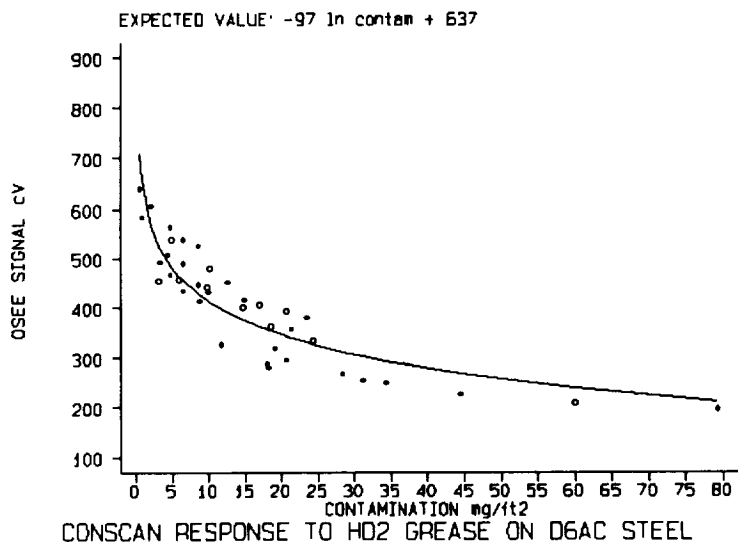


Figure 6

must be removed also. Grease, used to assemble and lubricate the O-rings is squeezed out when the segment is rotated in the horizontal rotator during insulation lay-up. The grease must also be removed before bonding of the weather seal. The surfaces are checked for cleanliness using OSEE after the cleaning operation before primer is applied.

The flex bearing allows the nozzle to gimbal and maintains the structural support to the aft dome. The bearing is composed of shims and end rings (concentric conic frustums) vulcanized together. To assure as clean a surface as possible the shims are checked with OSEE before primer application.

ON-LINE PROCESS DATA

The OSEE data are used to detect shifts in substrate preparation during processing. See Figure 9 for an example of mean OSEE values on the throat inlet ring bond surface for 16 housings processed.

CONCLUSION

OSEE has been used to monitor contamination on bonding surfaces in the manufacturing area for seven years at the Thiokol Corporation. The OSEE technique is still a developing technology. It has proven useful as a process aid in cleaning bonding surfaces to a clean and acceptable level. The OSEE inspection technique is an improvement over blacklight inspection because of its lower threshold of

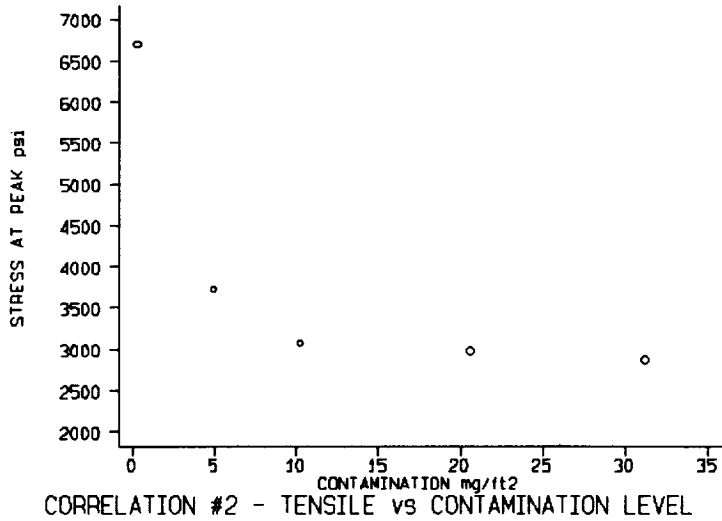


Figure 7

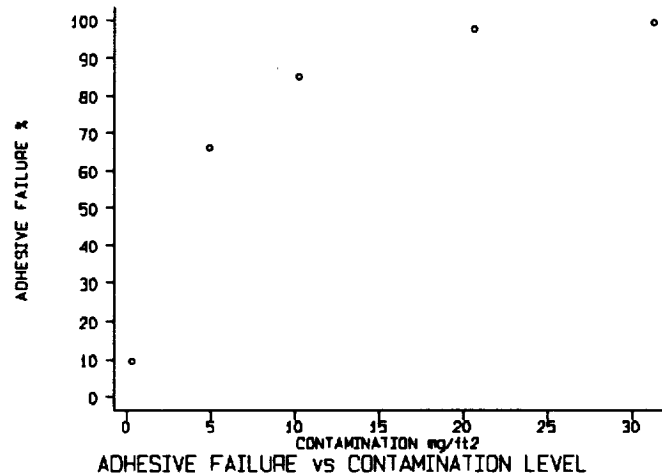


Figure 8

detection. OSEE provides an objective, documentable method of verifying bond surface cleanliness. The reliability of the OSEE equipment has continued to improve and additional improvements for industrial applications are anticipated with the third generation OSEE.

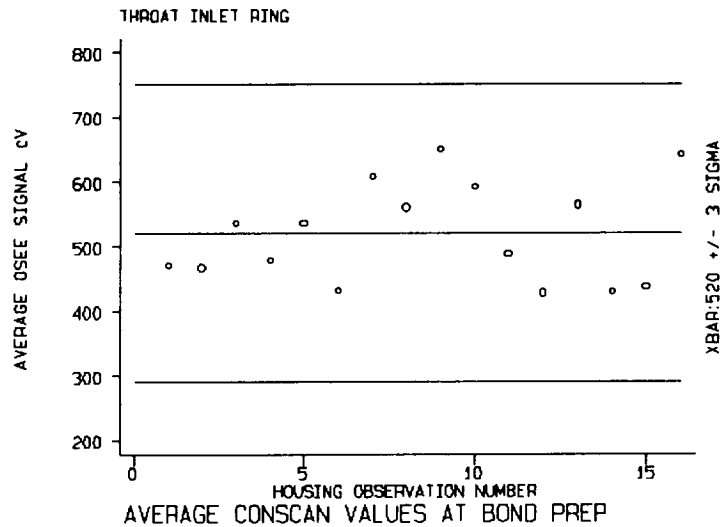


Figure 9

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- 1). Gause, R.L., "A Noncontacting Scanning Photoelectron Emission Technique for Bonding Surface Cleanliness Inspection", EH11-87-10, NASA, George C. Marshall Space Flight Center, AL 35812, December 1987.
- 2.) Mattes, R.A., "Contamination Monitoring of RSRM Bonding Surfaces using OSEE", Proceedings 35th Annual Technical Meeting, Institute of Environmental Sciences, 1989.

