SURFACE CLEANLINESS MONITORING

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IMPROVE COATING/PAINT ADHESION

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INTRODUCTION

Surface preparation is one of the most important factors in assuring proper adhesion of the rust prevention/paint adhesion chemicals to the coil and decorative/protective or laminate adhesion to prepared surface. These treatments can be defined as "coatings". Several after the fact tests are currently used in the coil coating industry to check adhesion of these "coatings" to the surface. Usually these tests are conducted on the first and last six feet of the coil. A successful test of this sample leads to the acceptance of the whole coil with the assumption that if the "processes" are kept in "control" then the rest of the coil will have good coating adhesion. Fortunately this assumption works most of the time. However there are several reasons/factors which can cause the coating adhesion on the coil to be unacceptable even though the end pieces passed the adhesion test. Knowing that the "coating" adhesion may be questionable after the "coating" is on the coil only helps prevent the shipment of bad product to a customer however it does not help in reducing the cost of scrap. The cost associated with accepting a bad coil and rejecting a good coil or producing a bad coil can be a significant set back to the profitability of a company.

This paper presents a technique known as Optically Stimulated Electron Emission (OSEE) that measures surface cleanliness, and can be a powerful tool for:

- 1. Evaluating the cleaning process or alternative cleaning processes to assure that the process is capable of producing the required level of cleanliness,
- 2. Defining the quantitative level of cleanliness required to achieve the desired adhesion strength,
- 3. Implementing on-line to assure that the required level of cleanliness is achieved on an ongoing basis thus assuring that the finished product will have the desired level of "coating" adhesion.

The basic technology and the results of some applications will be discussed along with an outline as to how this technique can be effective in cost savings and providing consistently high quality product for coil coaters.

OPTICALLY STIMULATED ELECTRON EMISSION (OSEE) THEORY

It is a well-known fact that when metals or certain other surfaces are illuminated with ultraviolet (UV) light with the proper wavelength (energy), electrons are emitted from the surface. The emitted and subsequently scattered electrons can be collected across an air gap by a biased collector and measured as a current. By maintaining the surface to collector distance relatively constant, changes in the measured photocurrent (which is in the order of 10^{-10} to 10^{-12} amps) can provide information about the surface, e.g., electronic structure, composition and chemistry. A contaminant on the surface, depending on its own emission characteristics, can either enhance or attenuate the inherent emission from a clean surface. In simple electronic terms, the clean surface is a current generator and a non-emitting contaminant acts as a resistance because the current is attenuated by interactions between the electrons and the contaminant. The thicker the contaminant, the higher the resistance and consequently the greater the decrease in measured signal.

The OSEE sensor utilizes a low-pressure mercury vapor lamp with two predominant peaks in its emission spectra, namely at 185 nanometer (nm) and 254 nm. These two wavelengths correspond to approximately 6.7 electron volts (ev) and 5.0 ev energy level. The UV light flux provides the energy needed by the material so the electrons emit (escape) from the surface. The materials/surfaces that emit, when exposed to this UV light, are called photoemitting materials and the other materials are called non-photoemitting. Both substrate and contaminate/coating can be emitting. A surface with no contaminant or coating on it usually gives a high OSEE signal. A contaminant or thin film, depending on its own emission characteristics, can either attenuate or enhance the OSEE signal from a clean substrate. Generally, contaminants/coatings attenuate the OSEE signal.

Presence of thin film, contaminant or coating has two effects. First, it reduces the UV light reaching the substrate and second it attenuates the flow of electron from the surface. Within a certain range, the signal attenuation is exponentially proportional to thickness of the thin films. The range of thickness measurement depends on the emission level of the substrate and the attenuation level of the contaminant or coating. OSEE is extremely sensitive to very low level of changes in surface chemistry. The technique is capable of detecting the presence of a partial monolayer of thin film contamination.¹

The sensor response to various materials generally depends on the magnitude of the photoelectron work function. Generally, materials with a work function of less than approximately seven EV (electron volts) will produce a photocurrent, i.e., photoemit. Both conducting and non-conducting materials can be photoemitting. For example, epoxy primer, carbon phenolic, glass phenolic, graphite/epoxy and fiberglass/epoxy will emit. Most plastics and ceramics are photoemitting. Some materials that have extremely low response include pure teflon, pure glass and magnesium fluoride. In general, measurements can be made on most of the materials of engineering importance, proving OSEE of great practical use.²

EVALUATING CLEANING PROCESS

An OSEE system can be used to measure surface cleanliness both off and on-line. In both cases, the OSEE sensor distance from the sample surface must be maintained consistently. For on-line measurements, the OSEE sensor can be mounted above the steel sheet at a location where the steel sheet vibration is minimum, e.g., over a tension or direction change roller. For steel sheet, the higher the OSEE reading the cleaner the surface is. Various factors of the cleaning process can be changed to evaluate their effect on surface cleanliness. Thus optimum combination of such factors as the chemical concentration, flow rate and rinse water flow and pressure rates can be determined to give the best surface cleanliness.

Off-line OSEE measurements can be used to evaluate the effectiveness of different cleaning processes. In order to eliminate cleaning processes that rely on CFC based chemicals, a tool such as an OSEE sensor is needed to quantitatively measure the surface cleanliness achieved by using CFC cleaning processes. This measurement will serve as a benchmark to evaluate the effectiveness of alternative cleaning processes.

DEFINING THE QUANTITATIVE LEVEL OF CLEANLINESS TO ACHIEVE THE DESIRED ADHESION STRENGTH

In order to quantitatively define the level of cleanliness needed to achieve the desired adhesion strength, a test would be required to correlate the adhesion strength to the level of surface cleanliness. To perform such a test, the following are needed:

- i) A target level for adhesion strength,
- ii) A method for consistent measurement of adhesion strength i.e. peel test or other tests,
- iii) Samples with varying degrees of cleanliness/contamination.

These samples can be prepared by either altering some factor/s of the cleaning process, thus creating samples with various degrees of cleanliness or they can be prepared by taking clean samples and applying known amounts of the most commonly encountered contaminants. The contaminants can be applied by mixing known weight of contaminants with volatile chemicals and air brushing the mixture over the clean samples. The samples can be weighed when clean, and after the contaminants have been applied. The difference in sample weight divided by the sample surface area gives a measure of contamination in mg per square foot. Thus, by varying the weight of contaminants added to a given amount of volatile chemical, samples with various and known levels of contaminants can be prepared.

After the samples have been prepared, several OSEE measurements per sample should be taken and recorded. The next step is to apply the "coating" of interest to these samples. Several adhesion measurement tests should be conducted on each sample and the adhesion strength recorded.

The mean OSEE readings for all samples should be correlated with the mean adhesion strength for those samples. The relationship between OSEE readings and adhesion strength is expected to be a natural log - linear relationship. The cleanliness level that correlates with the target level of adhesion strength then becomes the required level of surface cleanliness.

ON-LINE SURFACE CLEANLINESS MONITORING

Once the required cleanliness level has been established, the OSEE sensors can be mounted on-line to assure that the cleanliness level is being achieved, which in turn will help assure the adhesion strength of the "coating" to the steel sheet.

By monitoring the surface cleanliness to a required level of cleanliness, the number of coils produced with poor adhesion due to surface contamination will be eliminated. In addition, the result of adhesion tests done on the end of the coil will be more representative of the whole coil due to consistency in surface cleanliness.

Another advantage of on-line monitoring of surface cleanliness is that replenishment of chemicals or cleaning agents is only done when needed and not to a pre-determined, somewhat arbitrary schedule. The required level of concentration of chemicals or cleaning agents in the bath can also be objectively determined.

TYPICAL APPLICATION

It has been applied to detect contamination and/or thin film coatings in the Aerospace industry, computer hard disk manufacturing and metal finishing industry. The technique has also been applied to improve weldability of parts by assuring proper cleanliness levels, and monitoring oxide presence and growth on bare copper on printed circuit boards.

The OSEE technique has already proven itself to be a very useful and reliable method for assuring product quality through proper quantitative monitoring of surface cleanliness levels.

In the Aerospace industry, the performance of many critical components for the Space Shuttle and other flight hardware depends on the quality of bonding achieved during manufacturing. An example of a major shuttle element, where quality bonding is crucial to performance reliability and safety, is the Solid Rocket Motor (SRM). Inadequate bonding of the rubber installation to the case could result in exposure of the D6AC steel case to the hot gases from the burning propellant and result in a burn-thru which could be disastrous.

Studies were performed to correlate the relationship between the level of contamination vs. the peel strength of the bond. The relationship between the level of contamination vs. OSEE signal was also established.

Prior to the use of OSEE for this application, there was no method to detect contamination levels less than 100mg/square foot, which resulted in average bond strength of 59 pounds per linear inch (PLI). The use of OSEE has given NASA the capability of monitoring surface contamination down to 0.25 mg/square foot. Better cleaning methods were developed by using OSEE to optimize the cleaning process. As a result, an average bond strength of 196 PLI has been achieved, a three fold increase in bond strength.

CONCLUSION

OSEE is a very powerful tool to establish quantitative surface cleanliness levels, and help answer a lot of questions regarding cleaning processes, and assure consistent coating adhesion while realizing savings through optimum use of cleaning agents and less coils with poor adhesion.