CLEANING LABORATORY EVALUATION SUMMARY

SCL #:	2000
DateRun:	01/23/2000
Experimenters:	Jason Marshall, Nicole Vayo
ClientType:	Metal Working
ProjectNumber:	Project #1
Substrates:	Alloys
PartType:	Coupon
Contaminants:	Cutting/Tapping Fluids, Lubricating/Lapping Oils, Oil
Cleaning Methods:	Mechanical Agitation
Analytical Methods:	Goniometry, OSEE
Purpose:	To identify a method for analyzing part cleanliness.

Experimental Procedure:

OPTICALLY STIMULATED ELECTRON EMISSION
Optically Stimulated Electron Emission or PEE, Photo Electron Emission is based on the principle that metals and certain surfaces emit electrons upon illumination with ultraviolet (UV) light. These electrons can be collected, measured as current, converted to a voltage and digitally displayed. A surface contaminant will either enhance or attenuate this signal, depending on it own photoemissive nature. While OSEE will not identify a contaminant, it is a good comparative tool to determine the degree of contamination. This method is best suited for thin films (oils, etc.) and not particulate matter (dust, for example).

A few samples that are known to be clean should be tested to select best gain settings on the sensor and front panel. Gain settings should be selected such that the highest possible reading is obtained from these clean samples, but should not be more than 1000. Once the best gain settings are established, these should be recorded. The attenuation of the OSEE signal is inversely proportional to the thickness of contamination or coating within a certain range of the thickness of the contamination or coating. This range vases from material to material and the type of contaminant or coating. Typically this range is approximately 500 angstroms with a maximum of 7500 angstrom range recorded on HD2 grease on D6AC steel.

OSEE is a comparator and it gives readings in OSEE units and not in thickness units, e.g. angstroms. To determine the relationship between OSEE response versus the thickness and the range of thickness within which the relationship is proportional, tests need to be conducted on samples with known but different amount of contamination or coating thickness. OSEE measurements should be taken from each sample preferably in the same area where measurements were taken with the other method. This would prevent any errors due to variations in thickness across a sample. OSEE readings should be recorded for each sample. It is recommended that at least five samples of each thickness should be measured and an average of five samples be used in establishing OSEE vs. thickness relationship.
A statistically large enough sample should be taken of both coated and uncoated parts. OSEE readings should be taken and recorded for each sample in both groups. Mean and standard deviation for both groups should be calculated to establish ~ 3 standard deviation limits for both groups.

The natural log of OSEE readings should be correlated with thickness readings obtained with the other method. Using linear regression, the best fitting straight line can be found and the slope and intercept of that line can be established. A graph should be prepared to make sure that the relationship is linear over the range of thickness measured. If relationship is not linear over the full range, then the linear regression should be done only on the linear range. The relationship derived will be of the type: $Y = C - mx$
where m is the slope, C is the intercept, Y is the thickness and x is natural log of OSEE. This relationship will also define the range of thickness that can be measured for this particular substrate and the type of film/coating.

GONIOMETRY
Like OSEE, laser or optical contact angle goniometry is the measurement of a secondary effect to extrapolate surface cleanliness. A small drop of deionized water is placed on the substrate of interest. A light is shown to reflect the droplet's interface with the surface. Usually, the higher the contact angle (that is, the height of the bubble), the greater the contamination. Conversely, water dropped on a clean surface generates a much smaller, flatter contact angle. An example of this effect is noticeable after waxing and then washing a car; the remaining wax acts as a contaminant and the residual water on the surface of the car 'bubbles up.' The technique is limited in that only the cleanliness under the tiny drop is measured so that several readings must be taken. Flat surfaces are more conducive to accuracy with this method.

Before measurement, the angle measurement card must be calibrated with the laser. The laser should hit the measurement card directly in the center. When calibrating do not turn the laser from side to side because it will affect results. Then place the sample onto the sample stand. Adjust the stand so that the laser skims the surface of the sample. This will happen when the laser creates a line across the surface of the sample. When the laser skims the sample surface a vertical line should be on the measurement card. Adjust the card so that the line starts at the center of the card and goes directly along the 0 line. If the line is bent this means that the sample is not level and it must be adjusted.

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Once calibrated, place a 2 microliter drop on the surface of the sample with the syringe. Move the sample with the control knobs so that the laser skims over the surface of the sample and through the location where the water droplet meets the sample surface. Two lines should appear on the measurement card. The angle between these two lines is the angle of contact between the water droplet and the sample.

SUBSTRATE MATERIAL: Tantulum metal sheets
CONTAMINANTS: Mobil Oil Corp, Vactra Oil Light
CONTAMINATING PROCESS USED: Received contaminated
CLEANING METHOD: Analysis only

Results:

OSEE
After determining the appropriate settings of the OSEE instrument, readings of both the clean and dirty sample provided were analyzed. Clean readings averaged 228.5 and the dirty readings dropped to just under 45. The number of standard deviations were calculated using all six values for the clean sample. One of the samples from the dirty readings was well outside the projected average reading and was not used. Table 1 lists the readings made as well as the calculated average and standard deviation. OSEE has the potential to be used as analysis tool.

Table 1. OSEE Readings

Reading	Clean	# Std Dev	Dirty	# Std Dev
1	257	1.771	49	0.402
2	240	0.715	141	>3 not used
3	227	-0.093	44	-0.076
4	223	-0.342	52	0.688
5	208	-1.274	54	0.88
6	216	-0.777	25	-1.893
Average	228.5	44.8		
STDevP	16.09	10.46		

GONIOMETRY
Contact angle readings were not easily obtained from the parts sent to the lab. The pieces had to be clipped to flat piece of metal in order to obtain measurements. Once a level surface was created, contact angles were measured for both the clean and dirty sample. The clean readings were about 20 degrees lower than the dirty readings. All of the values recorded fell within three standard deviations from the average value. Table 2 lists the numbers recorded for contact angle goniometry.

Table 2. Contact Angle Readings

Reading	Clean	# Std Dev	Dirty	# Std Dev
1	46	-1.4	69	1.2
2	46	-1.4	69	1.2
3	50	0.6	67	-0.1
4	50	0.6	66	-0.8
5	51	1.1	65	-1.5
6	50	0.6	67	-0.1
Average	49		67	
Std Dev		2		1.5

This method also could be used to determine if samples met cleanliness levels as long as certain steps are taken. The clipping of the samples to a rigid surface may limit its usefulness in production. Also, the addition of water drops may pose a problem to the end product.

Summary:

Substrates:	Alloys
Contaminants:	Cutting/Tapping Fluids, Lubricating/Lapping Oils, Oil

Company Name:	**Product Name:**	**Conc.:**	**Efficiency:**	**Effective:**	**Observations:**
Environmental Technology	RB Degreaser Cleaner		0.00	☑	

Conclusion:

Both OSEE and Contact angle goniometry were shown to be effective in determining a difference between clean and dirty samples. The next phase will be to create a correlation between known contaminant levels with analysis readings.