SURFACE QUALITY UNIT FOR INSPECTION BY NONDESTRUCTIVE TESTING (SQUINT)

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SURFACE QUALITY UNIT FOR INSPECTION BY NONDESTRUCTIVE TESTING (SQUINT) WITH PHOTOELECTRON EMISSION (PEE) IN AIR

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ABSTRACT

A tool has been developed for non-destructive inspection of the quality of surfaces that will be used for painting, adhesive bonding, soldering, coating and plating. It can also be used for quality control of semiconductor surfaces, epoxy-composite surfaces as well as monitoring defects (scratches) on metal surfaces. This tool measures current due to optically stimulated electron emission (OSEE). Of particular value is its operation in air, i.e., the tool can be used under normal factory ambient conditions. The tool works as well on epoxy or polyurethane painted surfaces as on metals and semiconductors. Since an electrical signal is produced, that is directly related to the surface quality, SQUINT can be used for feedback in the operation of automated processes.

1.0 INTRODUCTION

Many industrial processes require surfaces to be in some predetermined state. Examples are preparation of metal surfaces for adhesive bonding, painting, coating, plating, and soldering. The same is true for fiber/polymer composites and painted surfaces. There is therefore a need for quantitative quality assurance tools to assure the proper surface state has been achieved and maintained. Of particular importance is the ability of the tool to give a signal that can be used for process control and automation. The surface quality control unit for inspection by nondestructive testing (SQUINT), described in this paper, is shown to be useful for a wide variety of systems and it is expected that many uses are yet to be discovered. The principles of photoelectron emission (OSEE) in air are given in Ref. 1.

2.0 APPLICATION OF OSEE IN AIR

2.1 NDI of Epoxy Painted Surfaces

It was discovered that epoxy paints, used on the external tank (ET) of the space shuttle, are photo emitting. This discovery led to a non destructive-inspection (NDI) tool for detection of contaminated paint. This was needed because the polyurethane foam insulation was debonding from the paint due to silicone type contamination. (2)

For utilization of the OSEE inspection tool for quantitative quality assurance, adhesion studies were performed (3, 4) to identify the levels of contamination that significantly degrade the bond and therefore must be cleaned. The intensity of the OSEE inspection signal, revealed by the contamination map, can then be determined and used to discriminate between areas that should be cleaned and those that do not need to be cleaned. Once the detection technique has been established and the accept/reject signal level determined, it is only a matter of automatically scanning the tanks to produce a map of the surface that reveals those areas that must be cleaned. Remapping after cleaning will reveal if the cleaning has been adequate.

We discuss next the parameters involved in the OSEE inspection tool.

2.1.1 Photoelectron Attenuation

Attenuation of electrons follows an exponential law, (1)

$$I_P = I^o_{pm} \exp(-\acute{L}/d), \quad (1)$$

Where I_P is the OSEE current with attenuating film of thickness, d, present, I^o_P is the current at d = 0, and $\acute{L}$ is the attenuation index. To establish a quantitative measure of contamination thickness, an experiment was performed to measure the attenuation index, $\acute{L}$. An aluminum foil was bonded to a flat surface and cleaned with trichlorotrifluoroethane methylene chloride (TMC). RTV 102 contamination was put on the Al foil by placing a Kimwipe tissue (saturated with a 1% RTV 102/TMC solution) on the foil and allowing the solvent to evaporate. The deposited silicone contamination was then smeared uniformly over the surface with a clean dry tissue. To obtain different contamination thickness, the surface was wiped with a dry tissue a number of times, then with a TMC saturated tissue a number of times. A plot of OSEE vs. contamination thickness (from ellipsometry) is given in Fig. 1. The theoretical solid curve in Fig. 1 was calculated with Eq. (1), where I^o_{pm} = 9 nA

And

$\acute{L}$ = 63 Å.

2.1.2 Effect of Distance from Probe

The OSEE current should decrease as the probe is moved away from the surface. This is due to a decrease in light intensity beneath the collector and a decrease in the electric field between the surface and the collector. Figure 2 shows the OSEE decrease with distance for one of our probes.

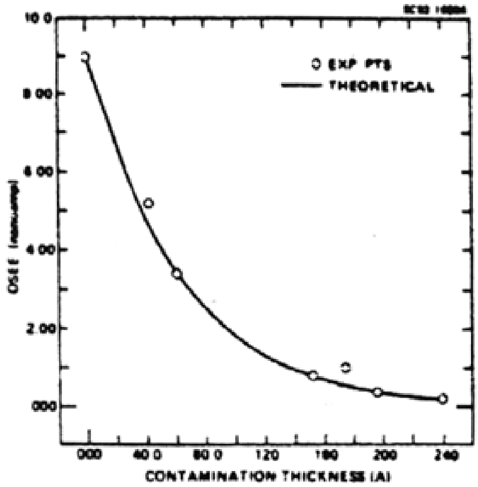

Figure 1 OSEE vs. silicon film thickness.

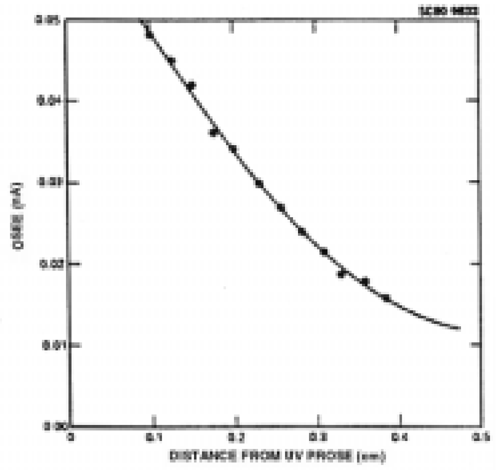

Figure 2 Effect of distance on OSEE current.

2.1.3 Correlation Between Contamination Detection and Bond Strength

The Adhesion properties of the Al 2219-T37 after surface preparation for painting and after painting for foam application have been measured as a function of contamination. The adhesion properties have been measured three different ways. First, after controlled contamination and mapping, strips of Scotch masking tape (1/2 in. wide) were pressed onto the panel such as to cross the various contamination regions. These strips were then peeled from the surface at 180° with an Instron tensile tester. Second, the paint or polyurethane foam was applied with a screen embedded as backing to give strength for a peel test. The paint or foam was peeled in 90° or 180° peel. Third, contaminated panels were bonded to uncontaminated panels with two-part polyurethane. The bonded panels were cut into lap shear test specimens, so that each specimen represented a particular contaminant and contamination level. The lap shear specimens were tested to failure and the shear strength recorded. The mode of failure was also recorded.

2.1.3.1 Scotch Tape Peel of Epoxy Painted Al 2219-T37

An epoxy painted panel was contaminated with silicon RTV 102 as follows: the silicon RTV 102 was dissolved in THF (tetrahydrofurane), then diluted to make four contamination levels. Pure THF was used for zero contamination, 1 part contaminated solution was added to 3 parts THF to get 0.25 level, 2 parts were added to THF to get 0.5 level and undiluted solution was used for level 1. A tissue paper was saturated with each of the contamination level solutions and wiped onto 4 regions of the painted panel. Figure 3a shows the OSEE as a function of the contamination level. OSEE drops dramatically, and levels off at a low value between contamination level 0.5 to 1.0. The curve in Fig. 3b shows the effect of the contamination on the peel force for stripping adhesive tape at a speed of 4 in./min at 180° peel. The peel force follows a curve similar to the OSEE curve; in fact, the peel force is almost directly proportional to the OSEE, and the adhesive strength of the paint surface can be predicted by measuring the OSEE.

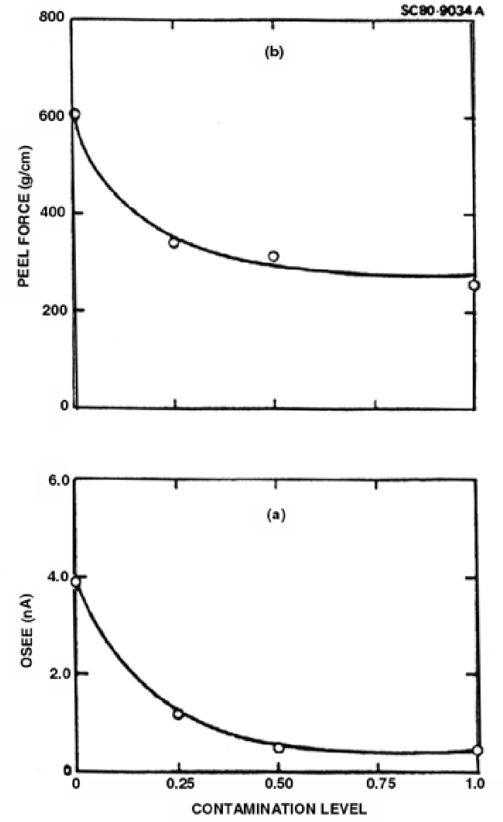

Figure 3 Effect of RTV 102 silicone contamination on (a) OSEE, and (b) peel strength.

Painted panels (1 ft x 1 ft) were divided into 12 regions, the lower regions were left uncontaminated as a control and the other regions were contaminated with fingerprints, masking tape residue, 3-in-1 oil, lubricating grease, cotton glove smudge, Kraft paper smudge, RTV 102, RTV 655 and automobile engine exhaust. The fingerprint area was contaminated by rubbing the fingers over the forehead and then on the panel; masking tape was stuck to the panel and then removed; RTV 655 was a mix of part A and B dissolved in TMC to make a 1% solution, as for the other contaminants. The region identified as car exhaust, was held for 30 s, 1 ft from the exhaust pipe.

A reduced-thickness (d/Ĺ) map is given in Fig. 4. The maximum reduced thickness (i.e., d/Ĺ) is 1.66, so that the

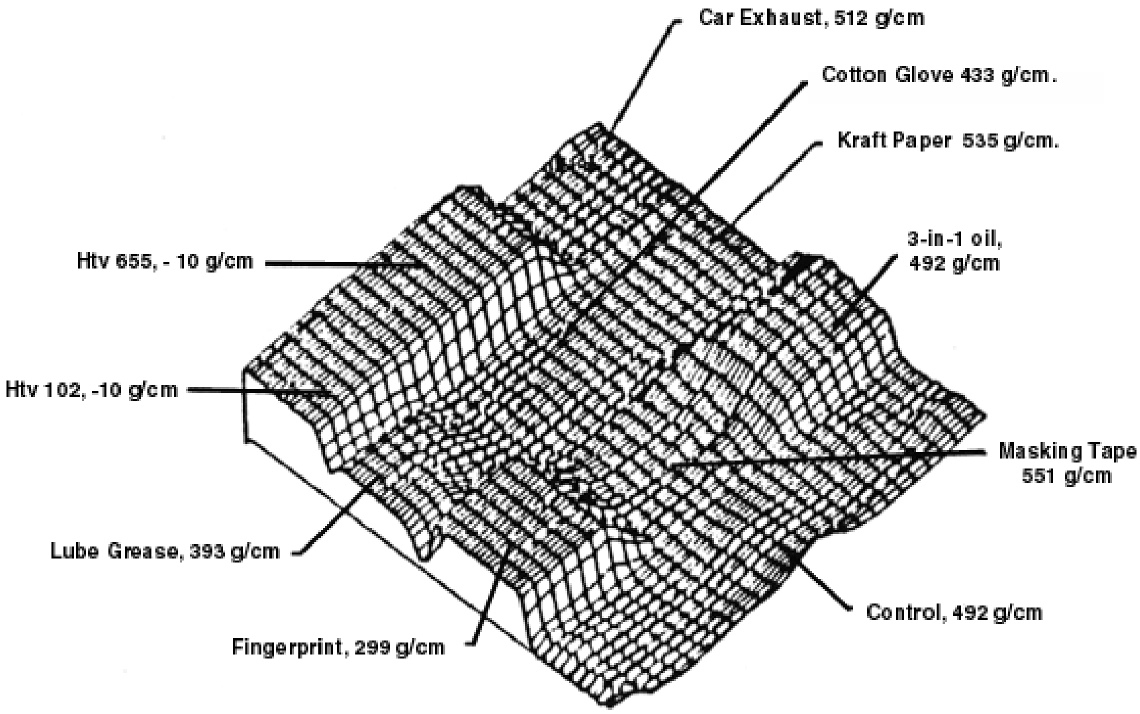

Figure 4 Map of reduced contamination thickness. The peel forces are indicated for each area.

maximum contamination thickness is 1.66 x 63 = 105Å . Figure 4 reveals very little contamination in the control, masking tape residue, cotton glove, Kraft paper smudge, and car exhaust areas. The fingerprint, 3-in-1 oil, lube grease, and silicone regions are strongly revealed.

After mapping the panel, half of each area (0.5) in. wide was bonded with 3M masking tape and the other half (0.5 in. wide) was bonded with PR-365 polyurethane one part adhesive. A fiberglass cloth scrim was embedded in the PR-365 for backing strength. The PR-365 was approximately 1/16 in. thick. The tape and PR-365 strips were cut with an Exacto knife and pulled in 180° peel at 4 in./min. The peel forces for the Scotch masking tape are indicated in Fig. 4. The control area, the masking tape residue, 3-in-1 oil, Kraft paper smudge and car exhaust areas failed between 490 and 551 g/cm. The cotton glove smudge area failed at 433 g/cm, the lube grease area at 393 g/cm, the fingerprint area 299 g/cm and the silicone area < 10 g/cm. The PR 365 formed strong bonds (>4.3 to 5.1 kg/cm) with all except the silicone areas, where the peel strength was about 0.3 kg/cm. The interfacial bond strength in areas other than silicone contamination is actually greater than 4.3 to 5.1 kg/cm because failure was at the glass scrim rather than the paint interface. The silicone contaminated regions failed at the paint-adhesive interface.

2.1.3.2 Tape Peel and Lap Shear Tests

Panels of epoxy painted Al 2219-T37, were divided into 1 in. strips and various contaminants were smeared on the different areas after wrapping Kimwipes around an aluminum block (1 in. wide) and soaking in the contaminant. For example, one area was smeared with CPR 483 foam component B, one strip was wiped once with a clean kimwipe soaked with a clean TMC, the next strip was wiped twice and so on, each time with clean TMC soaked Kimwipe, etc. In each case the contamination drastically degraded the adhesion and more than two wipes with TMC soaked Kimwipe restored the adhesion to better than the as-received condition. There is a fair correlation between the Scotch Tape peel test and the lap shear tests for the polyurethane foam joints.

2.1.4 Calibration of SQUINT

To illustrate the calibration of SQUINT, figure 5a shows plots of OSEE and peel force vs. position for an epoxy painted aluminum panel. Position 1 was smeared with a solvent solution containing RTV 655B silicone contamination; position 2 was wiped with TMC soaked Kimwipe; position 3 was wiped twice, etc. Figure 5b shows a plot of the resultant peel force vs. OSEE. The lower acceptance window value, below which the peel force dropped below 800 g/cm, is 10 nA. Similar results were obtained from a bare aluminum panel that had been prepared for painting. The OSEE window is different for the unpainted panel because of greater emission yield.

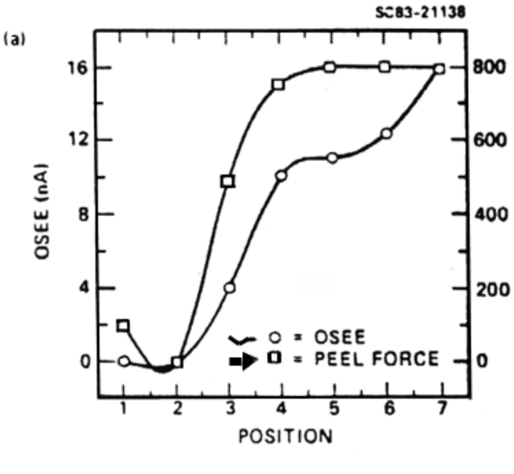

Figure 5a OSEE and peel force vs epoxy painted Al sample position, silicon contamination at left, decreasing to the right.

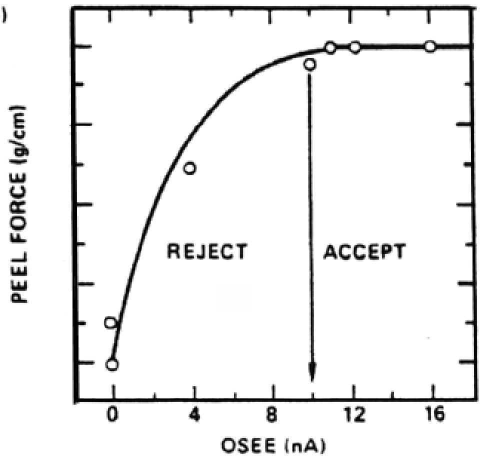

Figure 5b Peel force vs. OSEE indicating the acceptance window lower value (i.e., 10 nA).

2.2 NDI of Ball Bearings

Great care is taken in the inspection of the ball bearings after use in the liquid oxygen pump of the main engine of the space shuttle. These bearings depend on the molybdenum disulfide and Teflon for lubrication in the presence of liquid oxygen. The balls form a wear ring around the ball, i.e., an equatorial zone of heavy wear. Balls have been mapped with respect to OSEE to see if the wear ring can be distinguished from the low wear poles. Figure 6a (insert) shows the ball-OSEE configuration for mapping. The map shows OSEE vs. distance around the ball 2.5 times. A second map, reproduces the first map very well. The poles (non wear area) have low OSEE, whereas the wear ring is highly emitting. This is due to the presence of MoS which is about a factor of ten more emitting than the steel.

By rotating the ball at 90° from the initial role direction, through an azimuth of a few degrees each time, a complete map of the ball can be made, as in Fig. 6b. Each trace along the x-axis is an OSEE trace around the ball at different azimuths.

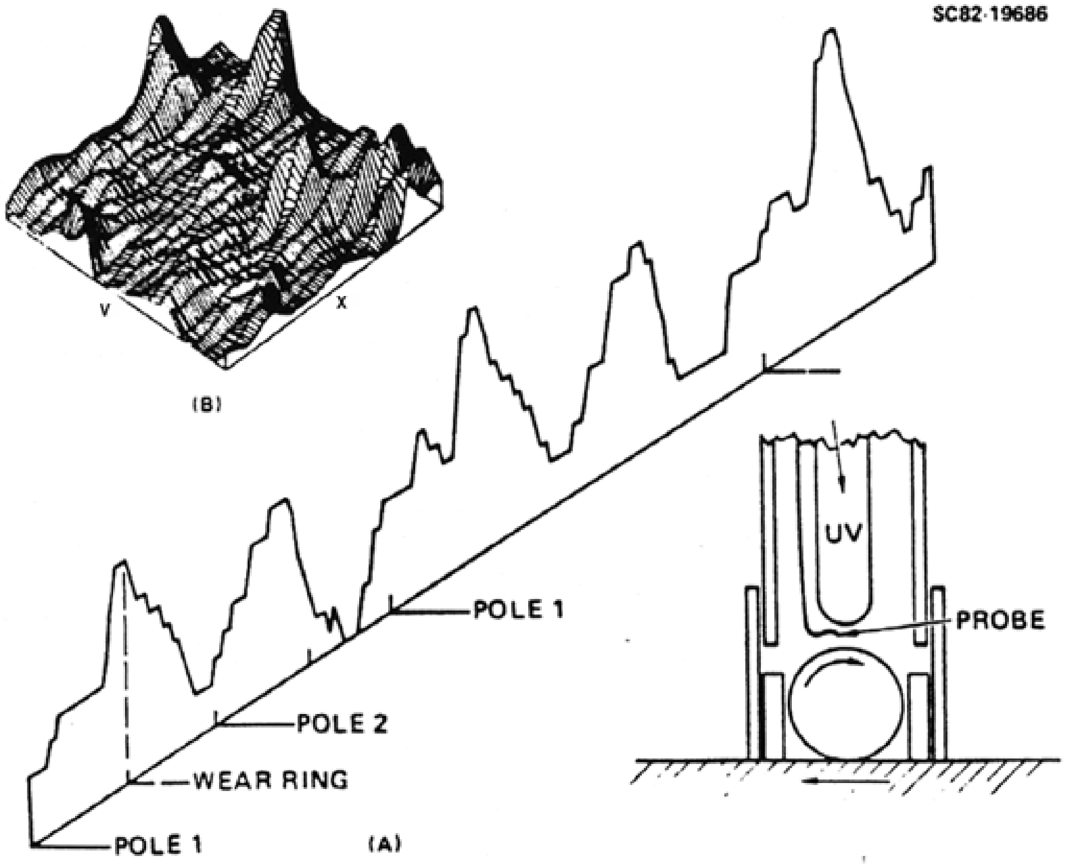

Figure 6 (a) OSEE trace 2.5 times around a steel ball bearing. Insert shows ball-OSEE system configuration. (b) OSEE map of a steel ball bearing.

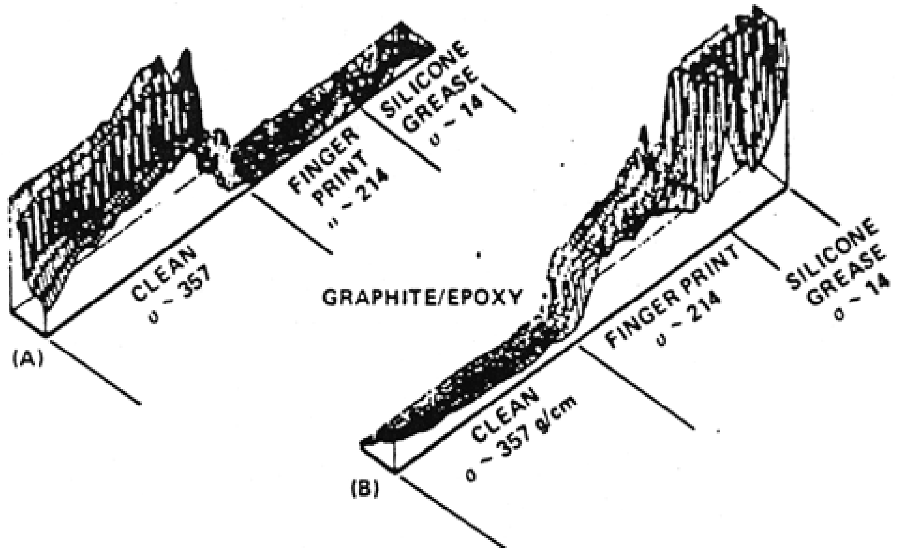

Figure 7 (a) OSEE map and corresponding peel strength for graphite/epoxy composite. (b) Reduced thickness map.

2.3 NDI for Contamination on Composite (Graphite/Epoxy)

A panel of graphite/epoxy was cleaned with acetone then deliberately contaminated with fingerprints and silicon grease. Figure 7a shows an OSEE map and Fig. 7b is the reduced thickness map. There is a direct correlation between the NDI map and the Scotch Tape peel test, the clean areas gave 357 g/cm, the fingerprint 214 g/cm and the grease area 14 g/cm. SQUINT will be excellent for inspecting epoxy composites.

2.4 NDI for Soldering

2.4.1 Contamination on Computer Chip Frames

Attaching computer integrated circuit chips to lead frames requires the frames to be clean. However, various processing problems can cause contamination. It would be valuable to be able to automatically inspect frames for contamination. Three frames strips were contaminated and two were clean.

OSEE nondestructive testing of frame strips for contamination was very successful and could be easily automated.

2.4.2 Void Free Soldering Power Transistors to Heat Sinks

We have had difficulty in soldering power transistors to copper heat sinks. It was found that the voids formed in the solder joint were caused by non-wetting of oxides by the solder. In a study to discover the effect of surface cleaning on the solderability of copper, the oxidation process was followed with OSEE, the clean copper follows a logarithmic time law, as reported in the literature.(5) Since it is the presence of the oxide that prevents wetting of the copper by the solder, it follows that the time between surface preparation and solder melting may be crucial. It was discovered that joints that proved to be void free had higher OSEE values (thinner oxide) than those that had voids.

It would appear that OSEE can be used for NDI of surfaces prior to soldering.

2.5 NDI of Semiconductor Surfaces

During the processing of semiconductors for integrated circuits, photodetectors etc., it is possible to inadvertently contaminate or make some other process error. Inspection of the surface at various stages for quality assurance may be of paramount importance.

Figure 8a is a map of four polished silicon wafers with varying oxidation time (30, 60, 120, and 300 min, left to right), and a fingerprint on No. 3. OSEE is very sensitive to oxide thickness and reveals the fingerprint contamination very well.

A silicon wafer was coated with photoresist, then UV exposed 440 s in one area (proper exposure), 220 s in the middle area and given no exposure in the remaining area.

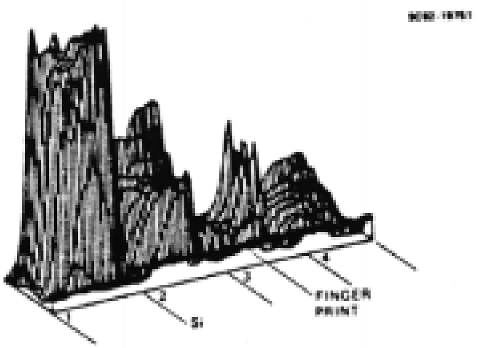

Figure 8 (a) OSEE map of four polished silicon wafers oxidized for 30, 60, 120 and 300 min (left to right).

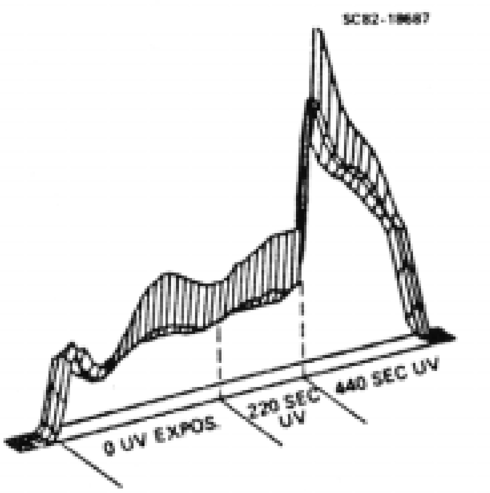

Figure 8 (b) OSEE map of silicon wafer with photoresist.

After developing, the sample was mapped with OSEE. Fig 8b indicates that although the photoresist is photoemitting, a dramatic increase in OSEE is obtained over the clean silicon.

OSEE maps of HgCd, HgCdTe crystals and ZnS/sapphire revealed surface heterogeneity, contamination and defects on these semiconductor materials.

SQUINT can therefore inspect for contamination as well as for improper processing (e.g., oxide thickness, defects and remaining photoresist) of silicon and other semiconductor materials.

2.6 Scratches on Metal Sheet

2.6.1 Aluminum

An aluminum panel was scratched in three places. The first scratch was approximately 0.05 mm in width, the second about 0.2 mm and the third about 1 mm. Figure 9a shows an OSEE map of the three scratches; the width of the map (X) is 1 cm, the length (Y) is 9 cm. Each scan on the Y-axis is 1mm, each scan on the X-axis is 2mm. Figure 9b is a map 24 h after making the scratches. The OSEE for the maximum value decreased from 0.803 nA to 0.0358 nA indicating a large attenuation due to oxidation. A map after 48 h indicated that oxidation is essentially complete after the first 24 h. However, the sensitivity is so high it is expected the scratches will be detectable for months.

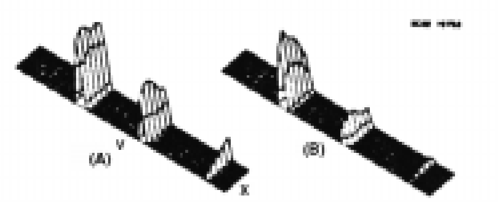

Figure 9 (a) OSEE map of three scratches on aluminum. (b) OSEE map after 24 h.

2.6.2 Speed of Response

An experiment was performed to determine the speed of response of the OSEE sensor. A plate of aluminum (21 cm diameter) was placed on a flat rotating wheel and the probe placed about 2mm above the plate. It was observed that the scratches could be easily detected at 33 ft/min.

SQUINT should be an excellent automated tool for detecting defects (e.g., scratches) on metal sheet as well as detecting contamination.

3.0 SUMMARY AND CONCLUSIONS

A photoelectron-emission-in-air technique has been developed and found to be extremely useful for nondestructive inspection for contamination on a great variety of surfaces. A general list of applications and a specific list that have been demonstrated in our laboratory is given in Table 1. The utility of SQUINT is very great, most of the application thus far have been for the detection of contamination. However, any surface that has been prepared, for whatever purpose, is likely to have unique

Table 1

Useful Application for SQUINT and a List of
Surfaces Successfully Inspected in This Report

General Applications

1. Bare metal surfaces after various surface treatments for painting, adhesive bonding, soldering etc.
2. Painted surfaces where paint and substrates are either good or very slight conductors; e.g., metals, semiconductors, most polymers, but not cellulosic or very insulating materials.
3. Graphite/epoxy composites; it should work as well on any epoxy composites, e.g., with glass, ceramic or metal fibers.
4. Inspection of semiconductor surfaces for contamination or process errors.

Specific Applications

1. Metal oxide thickness monitor, Al_2O_3/Al, NiO/Ni, etc.
2. Surface contamination on Al with and without epoxy paint.
3. NDI of ball bearings.
4. NDI of metal sheets for cold weld lamination, Al, Ni, zinc, steel Cu, brass, Ti, Be.
5. NDI epoxy composites.
6. NDI for soldering chips to lead frames and power transistors to heat sinks.
7. NDI of semiconductor surfaces, Hg-Cd, Hg-Cd-Te.
8. NDI of silicon for contamination, dust and photoresist.
9. Surface properties of ZnS/sapphire.
10. NDI of scratch defects on Al sheet.
11. Properties of lithographic plates.

photoemission properties. Whenever the surface deviates from these properties, the surface is suspect as to its intended use.

To use SQUINT as a quantitative quality assurance instrument, a surface of interest is properly prepared and SQUINT is calibrated with that surface. An acceptance window is determined by relating the signal from SQUINT to the effect of deliberate surface degradation. SQUINT is designed to give warning whenever the signal falls outside the acceptance window.

3.1 Sensitivity

SQUINT is routinely used at the Rockwell International Science Center to monitor contamination levels as low as partial monolayer absorption (<0.1 monolayers), up to thousands of monolayers.

3.2 Resolution

Our present system has a spacial resolution of about 1 mm^2.
This can be improved upon by beam focusing.

3.3 Response Speed

SQUINT has been used to monitor 0.1 mm wide scratches on an aluminum sheet, moving at 33 ft/min beneath the probe.

3.4 Stability

SQUINT is very stable, mapping samples months apart yields very reproducible maps.

3.5 Conclusion

It is concluded that SQUINT is simple to operate, gives quantitative information of a wide variety of materials and surface preparation, and gives an electrical signal that can be used for inspection automation and process control.

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