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# RESIDUAL SILICONE DETECTION

FINAL REPORT FOR THE PERIOD
November 13, 1979 through July 12, 1980

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This report describes a study of techniques for detecting surface contamination (particularly silicones) on epoxy painted and unpainted metal surfaces. Two techniques prove to be successful for the detection of silicones: photoelectron emission (PEE) and ellipsometry. The most successful technique is PEE, and this report deals primarily with PEE. Panels were deliberately contaminated to controlled levels, then mapped with PEE to reveal the areas (and level) that were contaminated. The panels were then tested with respect to adhesion (cont'd)



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## SUMMARY

This report describes a study of techniques for detecting surface contamination (particularly silicones) on epoxy painted and unpainted metal surfaces. Two techniques prove to be successful for the detection of silicones: photoelectron emission (PEE) and ellipsometry. The most successful technique is PEE, and this report deals primarily with PEE. Panels were deliberately contaminated to controlled levels, then mapped with PEE to reveal the areas (and level) that were contaminated. The panels were then tested with respect to adhesion properties by two methods. Tape was bonded over the contaminated and uncontaminated (control) regions and the peel force was measured, or the contaminated panels were bonded (with CPR 483 foam) to uncontaminated panels and made into lap shear specimens. Other panels were bonded and made into wedge specimens for hydrothermal stress endurance tests. The study was highly successful in that strong adhesion resulted if the PEE signal fell within an acceptance window, but was poor outside the acceptance window. A prototype instrument is being prepared, which can automatically be scanned over the external liquid hydrogen tank and identify those regions that are contaminated and will cause bond degradation. The instrument can also be used as a handheld tool for small parts.

# I. INTRODUCTION

## 1. The Problem

Figures 1 and 2 show flow diagrams for preparing the external liquid hydrogen tank (ET) and liquid oxygen tank, respectively. The tanks are first surface treated according to Table 1. After surface treatment, the Al 2219-T87 skin of the ET is painted with Desota 513-707 green epoxy primer to 1 mil thick. A newer formulation that contains more corrosion inhibiting dichromates is 515-346. The solid rocket booster (SRB) is first painted with Bostik #463-6-3 green epoxy primer to 1 mil, then with Bostik #443-3-1 gloss white epoxy topcoat from 1.0 to 1.8 mils thick and sanded lightly to break the gloss. The tanks are then sprayed with polyurethane foam to thermally insulate them.

Table 1

Surface Treatment Presently Used for Preparing the SRB and ET for Painting (for ET, delete Steps 6 and 7)

| Step | Material | Conc. (oz/gal) | Time (min) | Temp (°F) |
|---|---|---|---|---|
| 1. Degrease | MEK | | | RT |
| 2. Alkaline Clean | Turco 4215 | 1.25-2.50 | 10 | 150-170 |
| 3. Rinse | Water | | | RT |
| 4. Deoxidize | Turco Smut Go-1 | 14-18 | 10-15 | RT |
| 5. Rinse | Water | | | RT |
| 6. Chromate Conversion (Mil-C-5541) | Iridite 14-1 Water | 0.75-1.25 | 0.5-4 | RT |
| 7. Rinse | Water | | | RT |

Difficulties have arisen with respect to adhesion of the foam to the paint. It is suspected that during application of the ablative tiles, some of

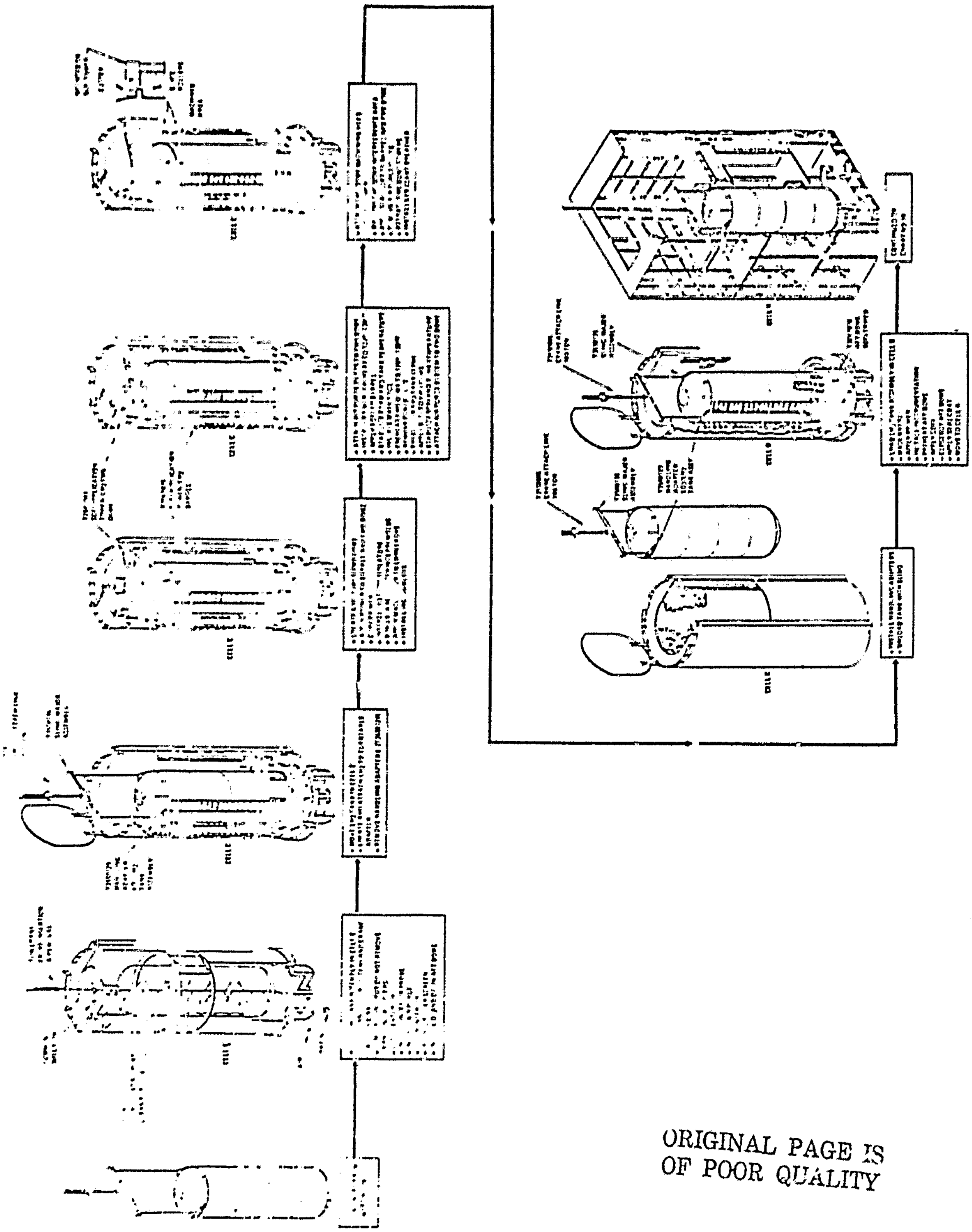

Fig. 1 Typical flow - $LH_2$ tank.

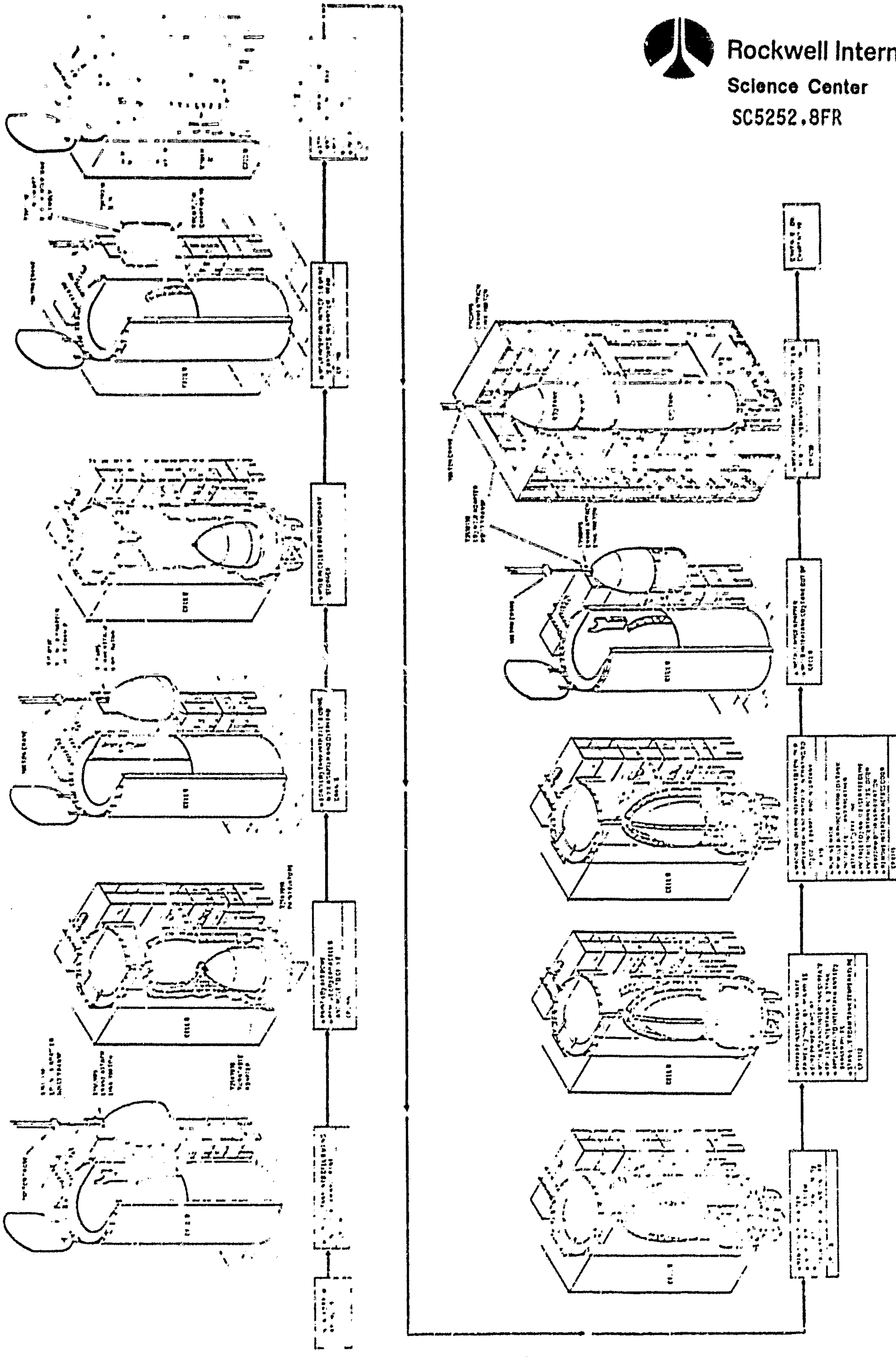

Fig. 2 Typical flow - $LO_2$ - tank.

the silicone components contaminate parts of the painted surface and thus degrade adhesion between the paint and the foam. The present remedy is to manually clean the entire tank with Scotch bright pads and TMC solvent. If the areas that are contaminated could be detected, they could be cleaned and there would be no need to clean the rest of the tank. This would result in considerable savings and might reduce the possibility of inadvertent contamination in areas that are already clean.

The problem addressed in this project is to develop nondestructive inspection techniques that will identify contaminated areas. To do this, two things are required: first, a surface technique that can detect the contamination must be found, and second, the level of contamination that significantly degrades the adhesion must be determined to establish that the instrument sensitivity is adequate.

## 2. The Approach

On a previous project[1,2] it was discovered that contamination could be detected by our different surface techniques, ellipsometry, photoelectron emission (PEE), surface potential difference (SPD) and water contact angles. These tools are described in that report and are illustrated in Fig. 3. The approach on this project was to try the first three of these techniques for the unpainted and painted surfaces of Al 2219-T37.

Painted aluminum received from NASA was cut into 1' × 1' panels. These panels were divided into a grid by pencil, then each grid area was contaminated by one of the contaminants listed in Table 2. The contamination was then removed to varying degrees by wiping with dry Kimwipe tissue or with tissue saturated with TMC.

After controlled contamination, the panels were mapped with the surface techniques to identify the contamination position and level. These panels were then tested for adhesion properties in three ways. A Scotch pressure-sensitive tape was bonded to the panels and the 180° peel test performed. In the case of controlled contamination of surface treated but unpainted panels, the panel was painted with Desota 513-707 epoxy primer that

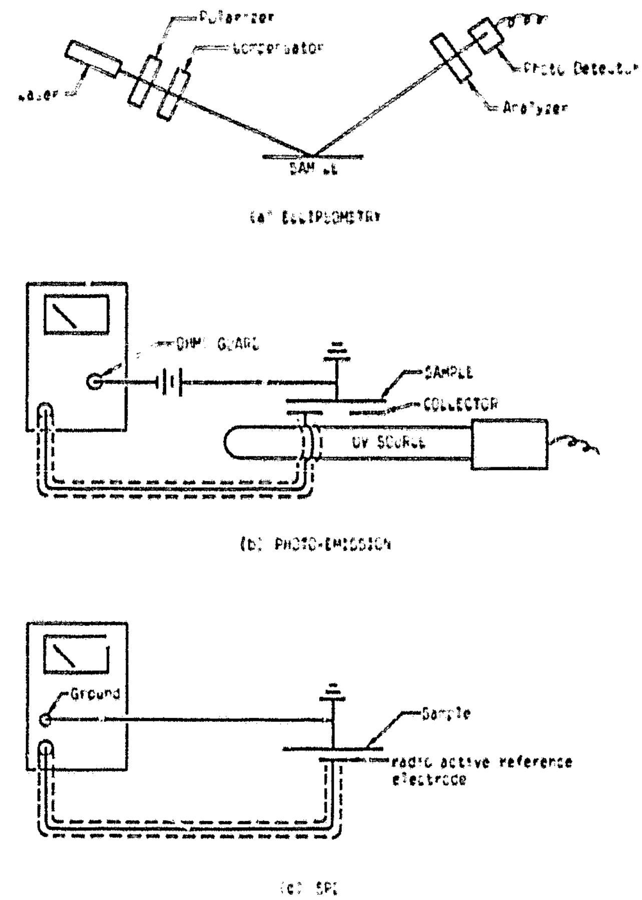


Fig. 3 (a) Schematic diagram of the ellipsometer. (b) Schematic diagram of electrical circuit for measuring photoemission. (c) Schematic diagram of electrical circuit for measuring SPD.

was embedded with a screen material to act as a backing scrim. This paint and scrim was cut in strips for peel testing the various regions. In the case of painted panels, they were bonded to clean panels with two part polyurethane foam. This polyurethane material was supplied by CPR Division of the Upjohn company (ISONATE CPR 483) and was represented as having the closest bonding properties to the CPR 488 used on the ET; however, it could be applied by simply mixing and pouring, rather than spraying.

The purpose of the adhesion studies is to identify the levels of contamination that significantly degrade the bond and therefore must be cleaned. The intensity of the 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.

Table 2. List of Contaminants

| | |
|---|---|
| 1 part RTV Silicone | RTV 102 (GE) |
| 2 Part RTV Silicone | RTV 655A and B |
| Foam application components<br>7344 Resin and 7119 Catalyst | CPR 488A and B |
| Hydrocarbon greases | 3-in-1 oil, lube grease |
| Engine Exhaust | |
| Kraft paper residues | |
| Cotton glove smear | |
| Fingerprints | |

# II. RESULTS

## 1. Sensitivity of Surface Tools

Figure 4 shows the signal level from three surface tools, ellipsometry, PEE and SPD, as a function of contamination with RTV 102 silicone. The contamination was then wiped with a tissue saturated with tetrahydrofurane (THF), twice. The ellipsometer and PEE showed a significant change in signal between the clean surface and the contaminated surface, and both returned to approximately the clean value on cleaning with THF. The SPD was insensitive to contamination on epoxy paint.

### 1.1 PEE

#### 1.1.1 Exposure to UV Light

A key discovery that contributed to the success of the project was that epoxy paints are photoelectron emitting and that emission is strongly attenuated by most contamination species. However, photoemission with 2500Å UV light is not constant with time; initial exposure produces a peak current which quickly decays under the lamp. Figure 5 shows the PEE current as a paint surface was swept past the detector. PEE increases to about 0.027 nA in 0.15 s and remains there as the surface moves about 13 cm. At 13 cm the motion was stopped; the current decayed rapidly at first, then slowed with time. A plot of the natural logarithm of the PEE current vs Ln (t + 0.23) is shown in the insert of Fig. 5. The PEE current decay of epoxy paint, on exposure to the UV light, follows the equation

$$I = 5.1\ (T + 0.235)^{-1/4} \tag{1}$$

represented by the straight line of the Fig. 5 insert. The curve at the right of Fig. 5 shows another scan over the same paint surface after turning off the UV light for 1.5 hrs. The current for the 13 cm traverse is slightly lower and drops to the decay value of the previous exposure in the position of long exposure. Scans after 6 hrs and 168 hrs were about the same, indicating no recovery of photoemission with time.

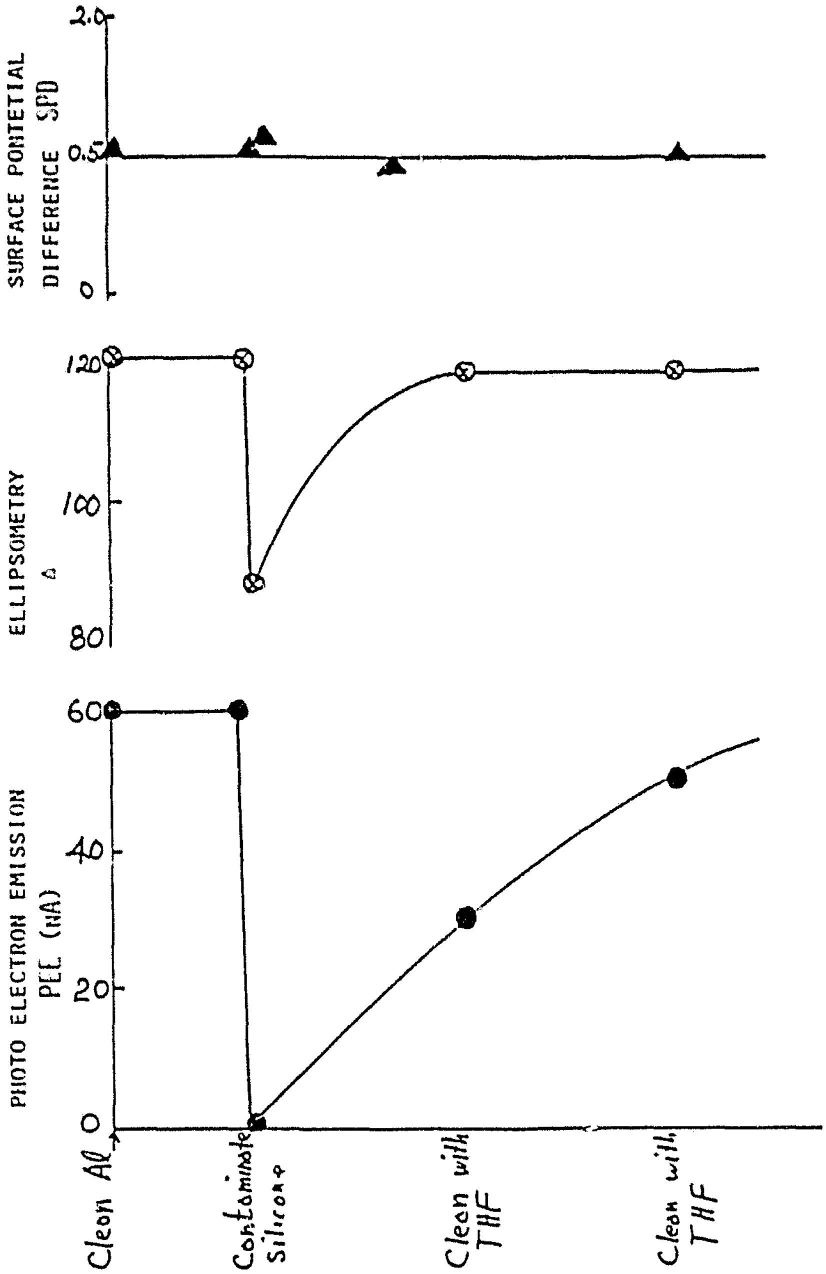


Fig. 4 Detection of silicone contamination by photoelectron emission (PEE), ellipsometry, and surface potential difference (SPD).

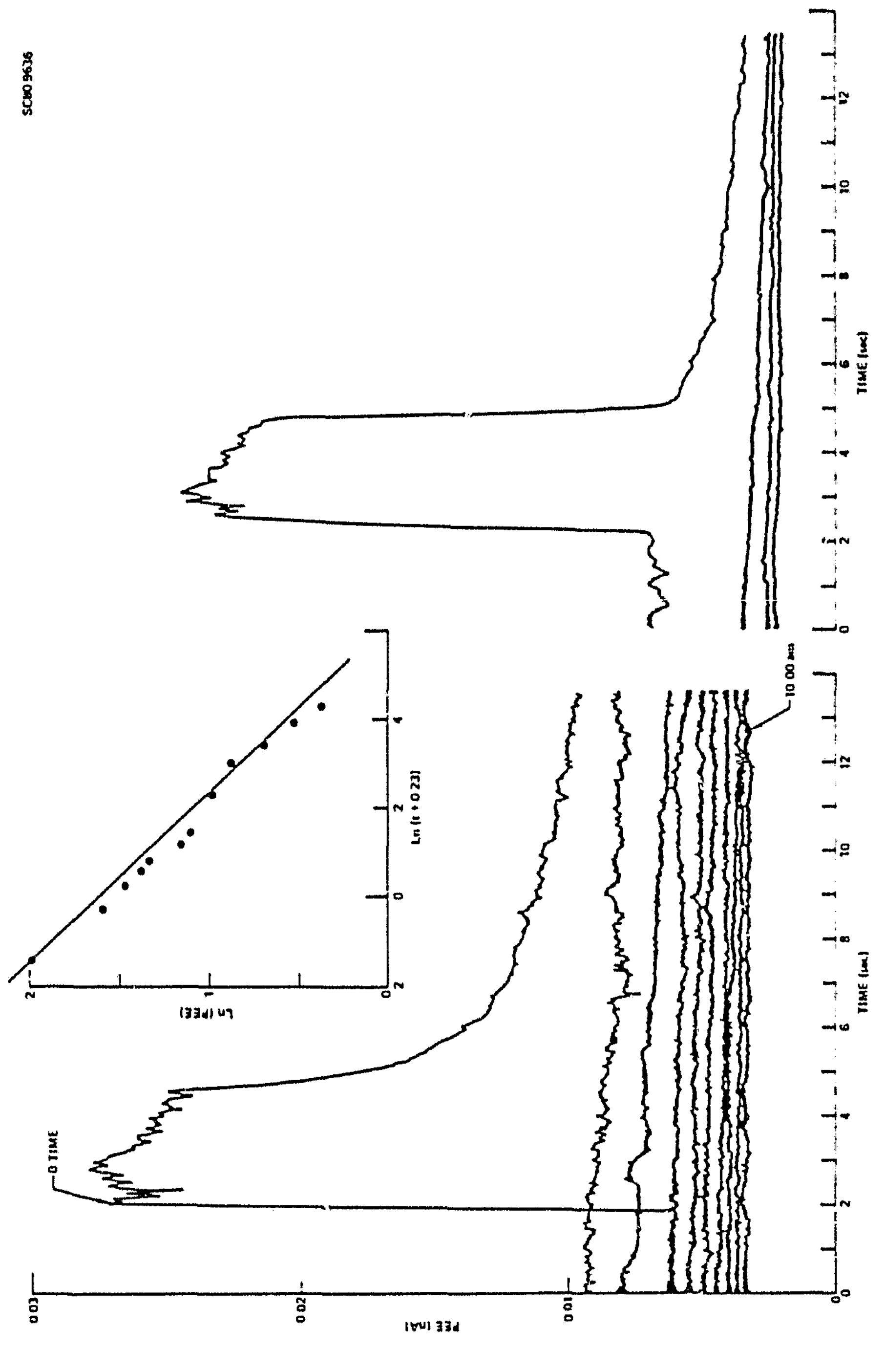


Fig. 5 PEE current during scan (2 ± 4.7 sec) and after stopping on one position. The inset is a plot of ln PEE vs ln (t + .23).

The PEE decay with UV exposure does not affect its use for rapid scanning for contamination, but must be considered if the surface is exposed. It will be shown later that exposure does not degrade the bondability, in fact exposure can be used to decontaminate the surface.

### 1.1.2 Photoelectron Attenuation

Attenuation of electrons follows an exponential law[3]

$$I = I_0 e^{-x/\lambda} \tag{2}$$

where I is the PEE current for contamination thickness x and $I_0$ is PEE at $x = o$. The attenuation index is $\lambda$ and the reduced contamination thickness is $x/\lambda$.

To establish a quantitative measure of contamination thickness, an experiment has been performed to measure $\lambda$. An aluminum foil was bonded to a flat surface and cleaned with TMC. Aluminum foil was used to measure $\lambda$ because of its high PEE current and its well known optical properties for ellipsometry. 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.

Table 3 gives the experimental data for $\Delta$ and $\psi$ (ellipsometric parameters), SPD (surface potential difference) and PEE (photoelectron emission). The last column in Table 3 gives the contamination thickness calculated from the ellipsometric data. Calculation of the attenuation index from

$$\lambda = x/\ln (I_0/I)$$

yields

Table 3. Determination of the Electron Attenuation Index

| | Ellipsometry | | | | |
|---|---|---|---|---|---|
| Al Foil Contamination | Δ (deg) | ψ (deg) | SPD (volts) | PEE (nA) | Thickness (Å) |
| Smear with 1% RTV 102 in TMC | 54.8 | 55.7 | 1.00 | 0.2 | 240 |
| 1st wipe with dry tissue | 66.4 | 47.9 | 1.17 | 0.4 | 196 |
| 2nd wipe with dry tissue | 106.6 | 43.8 | 1.00 | 0.8 | 152 |
| 3rd wipe with dry tissue | 98.8 | 42.6 | 1.10 | 11.0 | 174 |
| 4th wipe with dry tissue | 114.0 | 38.6 | 1.07 | 3.4 | 60 |
| 5th wipe with dry tissue | 116.4 | 39.2 | 1.24 | 5.2 | 42 |
| 6th wipe with dry tissue | 122.0 | 39.2 | 0.96 | 9.0 | 0 |

$\lambda = X/\ell n\ I_0/I = 63Å$

Table 4. Surface Properties of Contaminated Painted Aluminum

| | Ellipsometry | | | | | |
|---|---|---|---|---|---|---|
| Paint Contamination | Δ (deg) | ψ (deg) | SPD (volts) | PEE (nA) | $\theta_{H_2O}$ (deg) | Thickness (Å) |
| Smeared with 1% RTV 102 in | 6.4 | 24.2 | 0.55 | 0.0 | 105 | 93 |
| 1st dry tissue wipe | 8.8 | 21.3 | 0.70 | 0.0 | 105 | 135 |
| 2nd dry tissue wipe | 8.4 | 21.2 | 0.55 | 0.0 | 105 | 128 |
| 3rd TMC tissue wipe | 4.0 | 19.2 | 0.89 | 0.8 | 92 | 52 |
| 4th TMC tissue wipe | 0.8 | 18.7 | - | 1.8 | - | 0 |
| 5th TMC tissue wipe | 1.2 | 19.0 | 1.1 | 1.8 | 88 | 0 |

$$\lambda = 63\text{Å} \quad .$$

A plot of PEE vs contamination thickness is given in Fig. 6. The theoretical curve was calculated from

$$I = I_o \, e^{-x/\lambda}$$

where

$$I_o = q \text{ nA} \quad \text{and} \quad \lambda = 63\text{Å} \quad .$$

A similar experiment was performed on a painted aluminum panel from NASA. The data are given in Table 4 and plotted in Fig. 7. These results allow us to estimate the contamination thickness from the measured PEE values of reduced thickness.

### 1.1.3 Effect of Distance from Probe

The PEE 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 8 shows the PEE decrease with distance for one of our probes.

### 1.1.4 Effect of Scan Speed

The prototype PEE sensor will scan an area 9" wide. If the ET tank is 28' diameter and 154' high, and scanning is top to bottom, approximately 117 scans are needed to cover the entire tank. Most of the tests in this report were performed at 0.17 ft/s. Increasing the scan speed from 0.3 ft/s to 0.44 ft/s decreased the PEE signal from 1.6 nA to 0.9 nA. At 0.44 ft/s a scan from top to bottom takes about 6 min. At this speed it will take about 11 hrs to scan the whole tank. When the prototype sensor is complete, a check on scan speed will be made. It is anticipated that speeds of 1 ft/s will give sufficient sensitivity and will reduce the scan time to about 5 hrs. Of course this can be decreased by operating more than one detector or increasing the size of the detector.

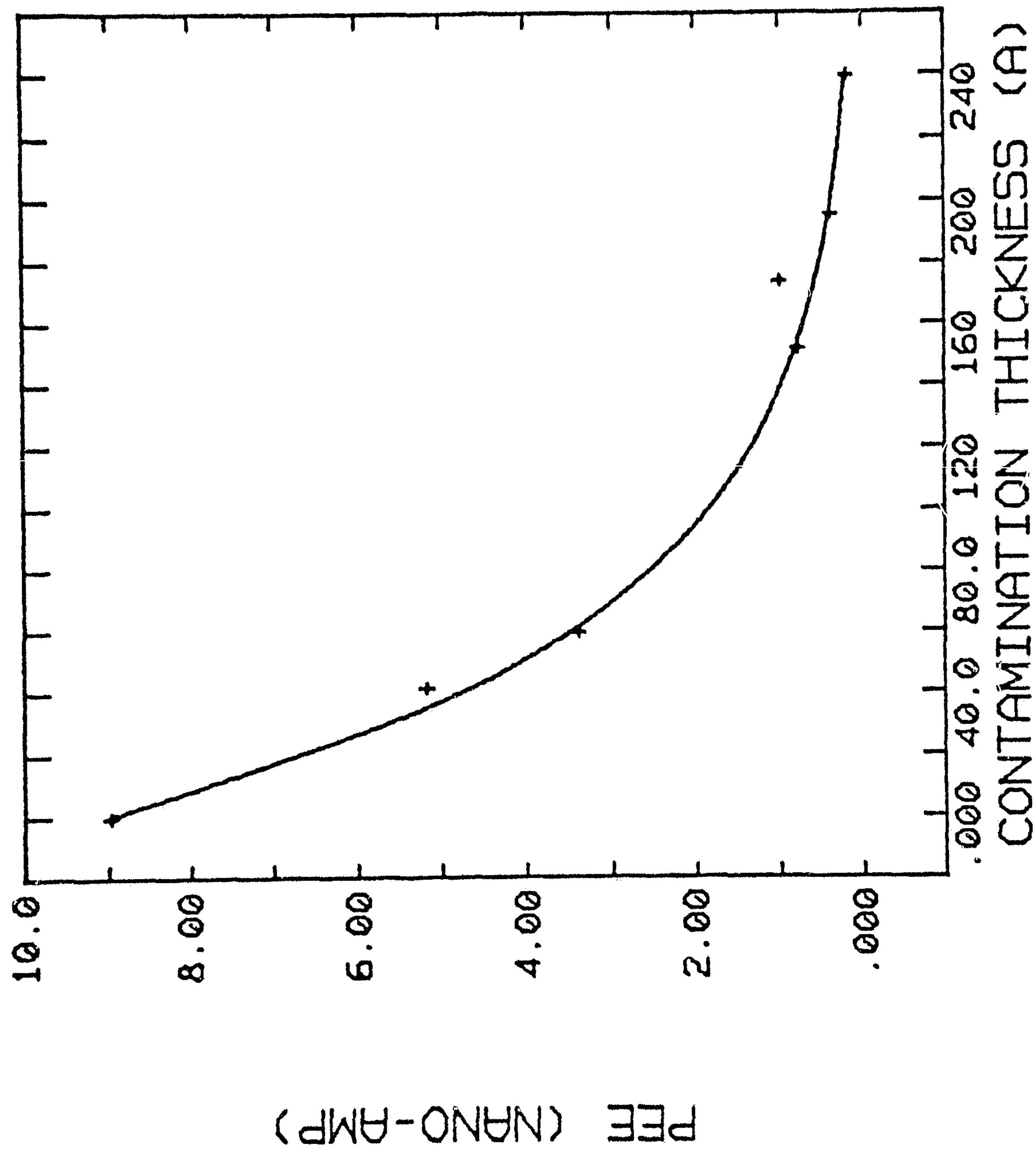


Fig. 6 PEE vs silicone film thickness.

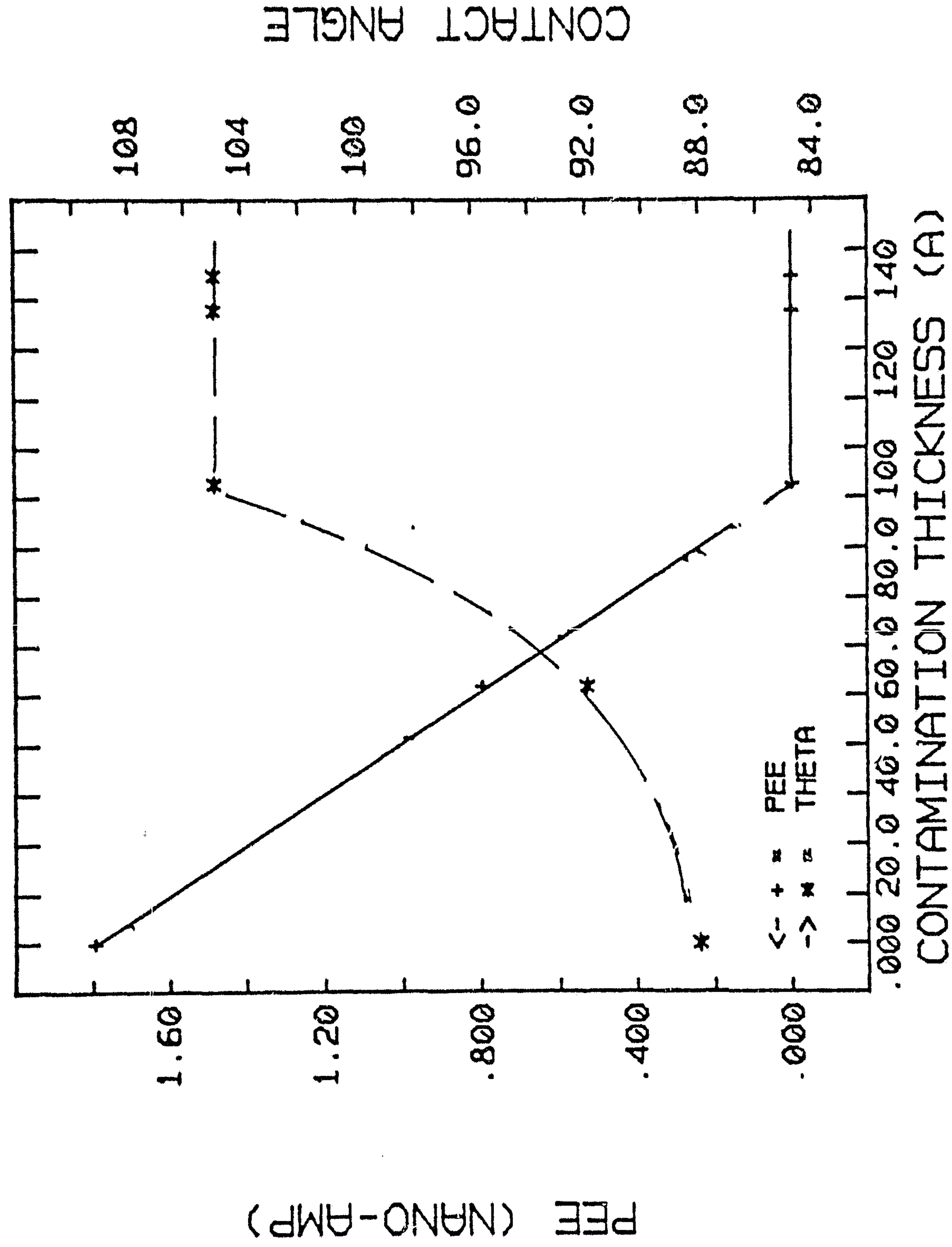


Fig. 7 PEE and theta ($H_2O$) vs silicone film thickness.

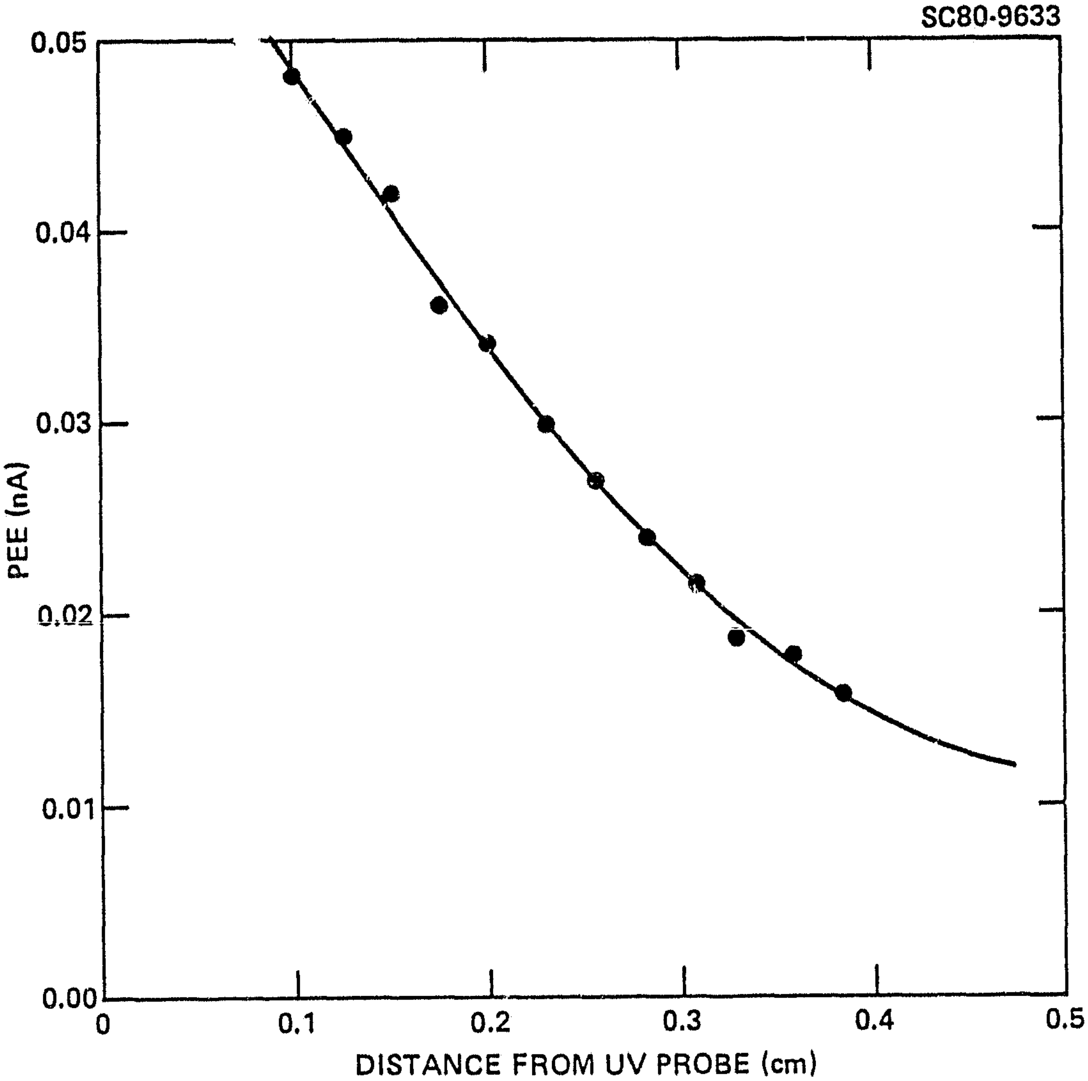


Fig. 8 Effect of distance on PEE current.

### 1.1.5 Effect of Sanding the Paint

Preparation of the ET calls for a light sand of the white epoxy top coat. Unsanded paint gave a PEE current of 0.045 nA, light sanding reduced this to 0.035 nA, heavy sanding reduced it to 0.025 nA. Cleaning the heavy sanded area with TMC increased the PEE to 0.0935 nA. Sanding should not interfere with the inspection technique if the instrument is calibrated with the painted surface in the clean sanded state. All of the tests in this report are for unsanded panels.

## 1.2 Ellipsometry

The Off NULL ellipsometric technique is described in detail in Ref. 2. The ellipsometer is nulled in the control area of the painted surface, so that a contaminated area shifts off null, increasing the light intensity to the photodetector. The thickness of the contamination in Table 3 was estimated from the $\Delta$ and $\psi$ values from standard ellipsometry. The sensitivity of ellipsometry to silicone on aluminum is high, $\Delta$ changes by 67° for a change of 240Å; this is because the index of refraction of the silicone is so different from that of the aluminum. On the other hand, the optical properties of the paint are close to those of the silicone, so that the sensitivity is greatly reduced. $\Delta$ changes by about 8° for a 135Å change in silicone thickness on paint. However, as will be seen, this sensitivity is adequate for detecting silicone contamination on epoxy paint.

## 1.3 Surface Potential Difference (SPD)

Tables 3 and 4 plus SPD maps, shown later, indicate that SPD is very insensitive to contamination on painted surfaces.

## 1.4 Water Contact Angle

Table 4 records the water contact angle on clean and contaminated epoxy painted surfaces. The clean epoxy has a contact angle of about 88°, the contaminated surfaces have a contact angle of about 105°. Figure 7 shows how the contact angle increases, then levels off with increased contamination thickness. The sensitivity is rather high but requires accurate, automated contact angle measurements to be useful for tank inspection.

## 2. 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" 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 Epoxy Painted Al 2219-T37

#### 2.1.1 Scotch Tape Peel

An epoxy painted panel from NASA was contaminated with silicon RTV 102 as follows: the silicone 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 9a shows the photoelectron emission (PEE) as a function of the contamination level. PEE drops dramatically, and levels off at a low value between contamination level 0.5 to 1.0. The curve in Fig. 9b shows the effect of the

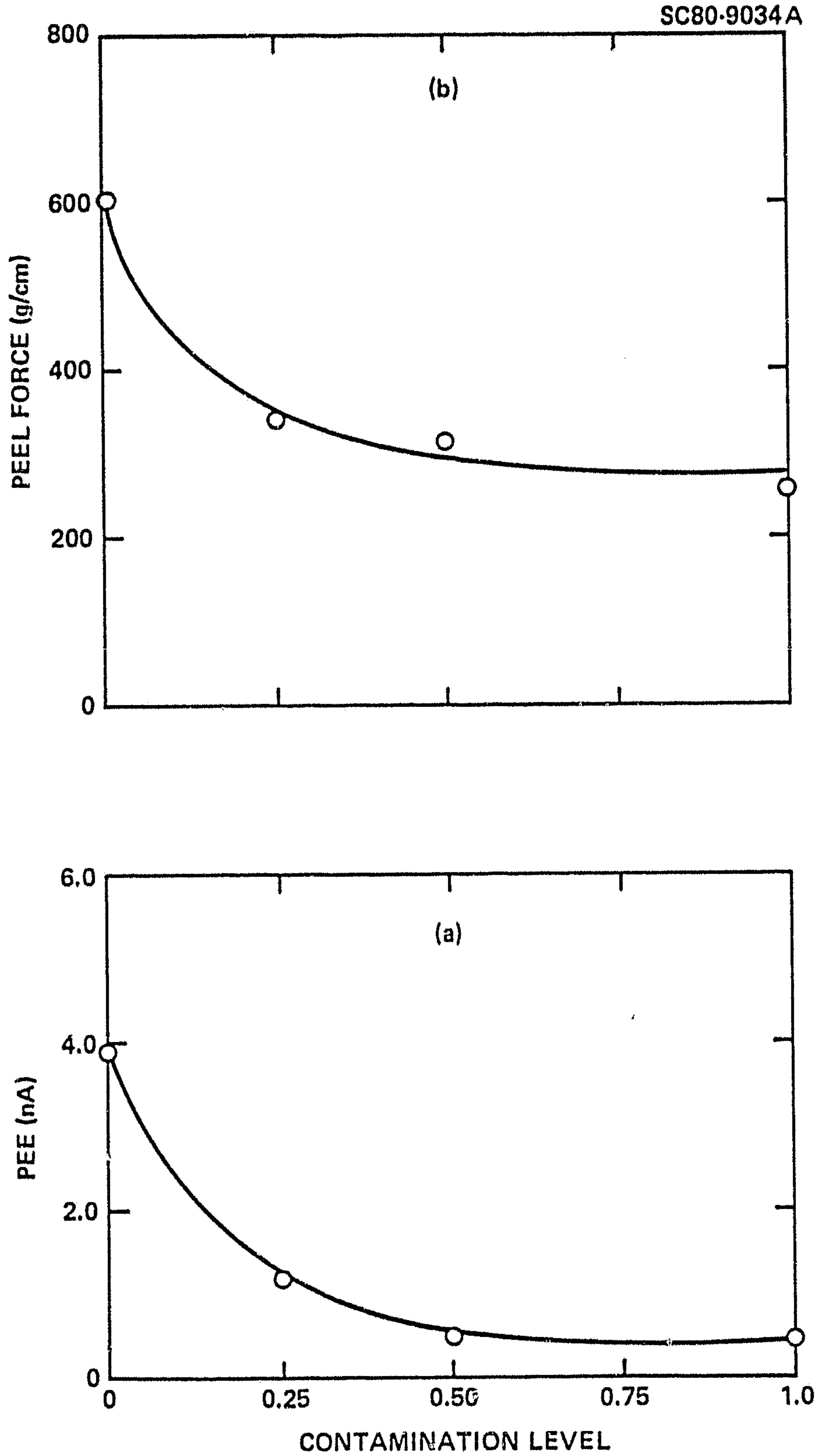


Fig. 9 Effect of RTV 102 silicone contamination on PEE and peel strength.

contamination on the peel force for stripping adhesive tape at a speed of 4"/min at 180° peel. The peel force follows a curve similar to the PEE curve, in fact Fig. 10 shows that the peel force is almost directly proportional to the PEE. That is, the adhesive strength of the paint surface can be predicted by measuring the PEE.

An epoxy (Bostik 443-3-1) painted panel (1' × 1') was contaminated with RTV 102 silicone dissolved in THF. After spraying the contaminant on, the surface was masked such that strips 1" wide could be wiped with tissue saturated with pure THF. By using the same tissue for each 1" strip, the level of contamination increased from one side of the panel to the other. A 1" strip at one edge was not contaminated to provide a control. Figure 11 is the PEE map of the contaminated panel. The high PEE currents at the far end of the panel corresponds to the clean control strip. The PEE current drops to 0.0036 nA in the high contamination region.

Scotch Tape was bonded along the contamination strips at the positions indicated by the arrows in Fig. 12 (panel is turned around with respect to Fig. 11). The peel forces to remove the tape in 180° peel are also indicated in Fig. 12. The peel force of clean paint is about the same as recorded in Fig. 1 (i.e., 650 g/cm) and decreases to 4 g/cm for the heavily contaminated region. The sharp ridges in the low contamination region of Fig. 12 (front of panel) are not caused by contamination, but are due to scratches used to separate the contamination strips.

Rather than use Scotch Tape, Polyurethane PR 365 was spread over a contaminated panel, and a layer of fiberglas cloth was embedded to give a strong backing for the peel test. The PR 365 was cured overnight at 80°C, producing a tough rubbery adhesive. Table 5 shows that the polyurethane adheres very strongly to the paint if it is clean. Either the urethane-glass scrim tape breaks or failure occurs cohesively in the polyurethane. At contamination levels of 0.01 and 0.05 for RTV 655 silicone, adhesion is strong (9 kg/cm) with cohesive failure. At contamination levels >0.1, failure becomes adhesive at the contaminated paint surface and the peel strength drops to 1-2 kg/cm.

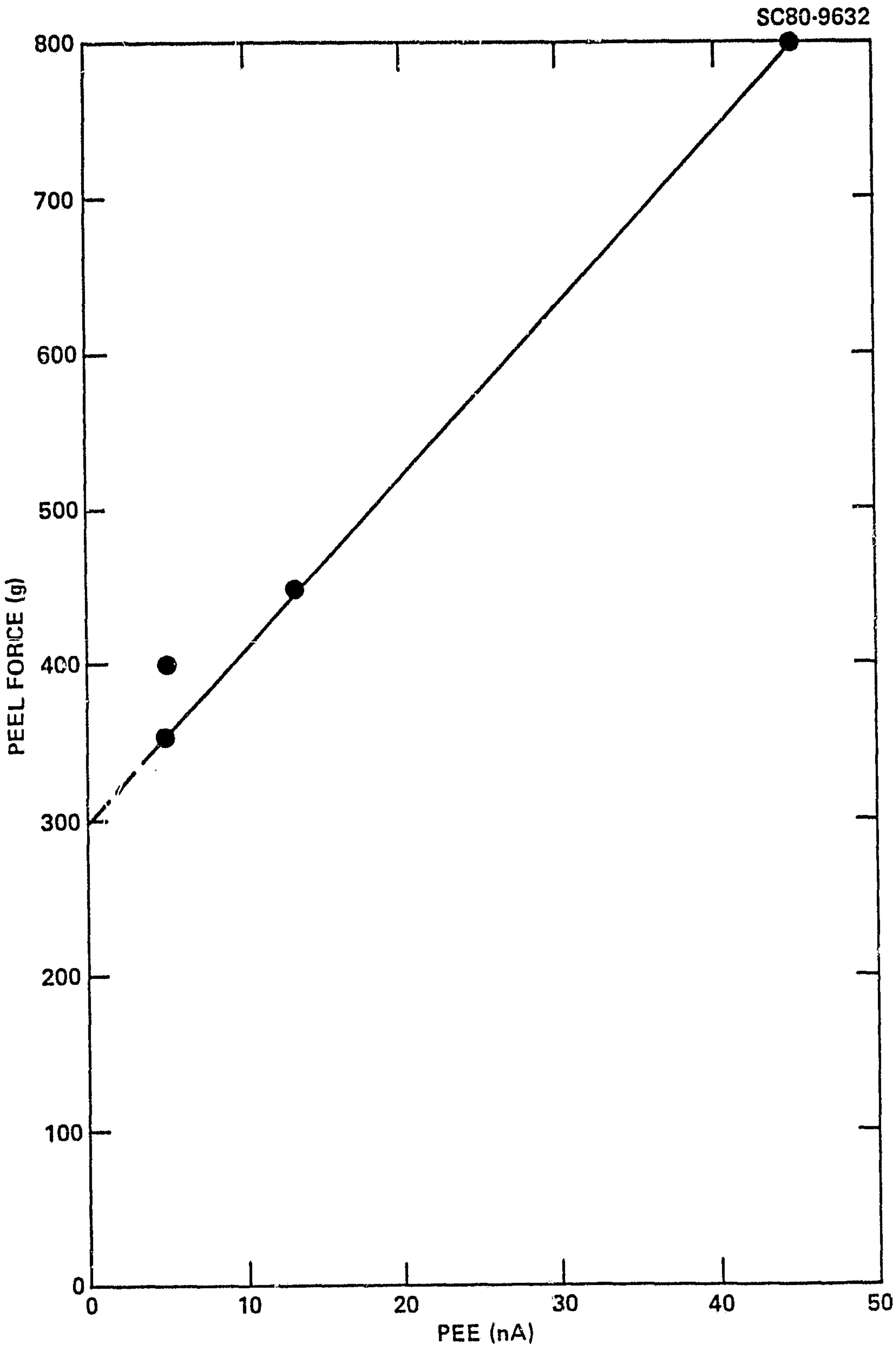


Fig. 10 Relation between peel strength and PEE.

PEE 8 APR 80

RTV-102 CONTAMINATION ON BOSTIK 443-3-1 WHITE EPOXY TOP GLOSS COAT

MIN = .3600E-02 NANO AMP
MAX = .5240E-01 NANO AMP
AVERAGE= .9344E-02 NANO AMP
STD DEVIATION = .8159E-02 NANO AMP

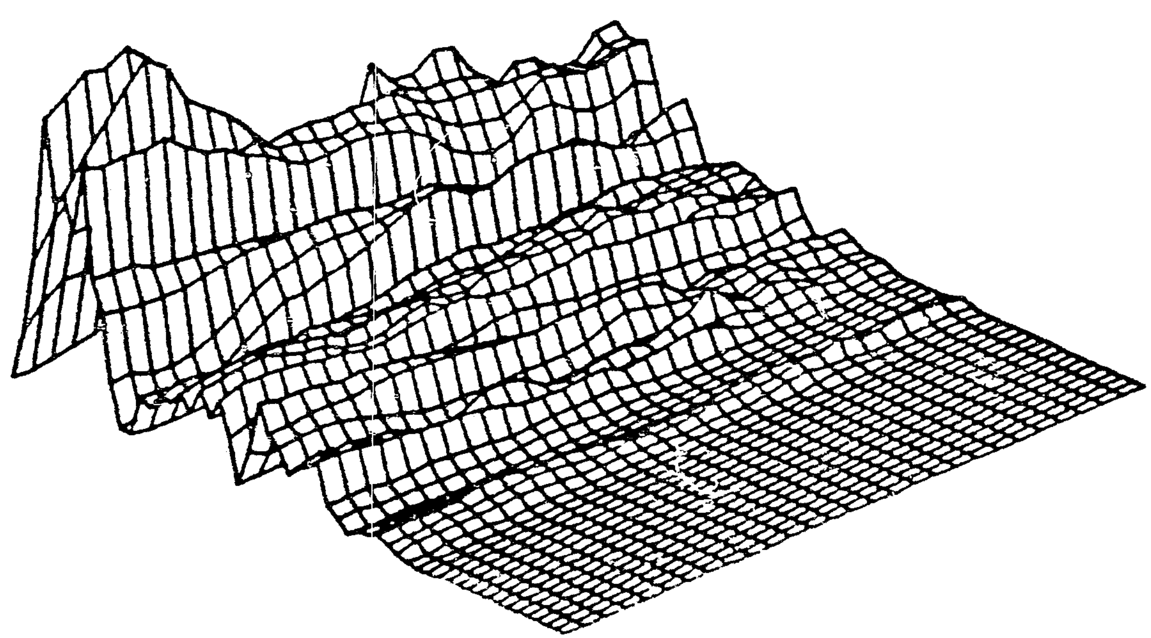



Fig. 11 PEE map.

PEE 8 APR 80

MIN =.0000E 00 REDUCED THICKNESS
MAX =.2155E 04 REDUCED THICKNESS
AVERAGE =.1629E 04 REDUCED THICKNESS
STD DEVIATION =.5661E 03 REDUCED THICKNESS

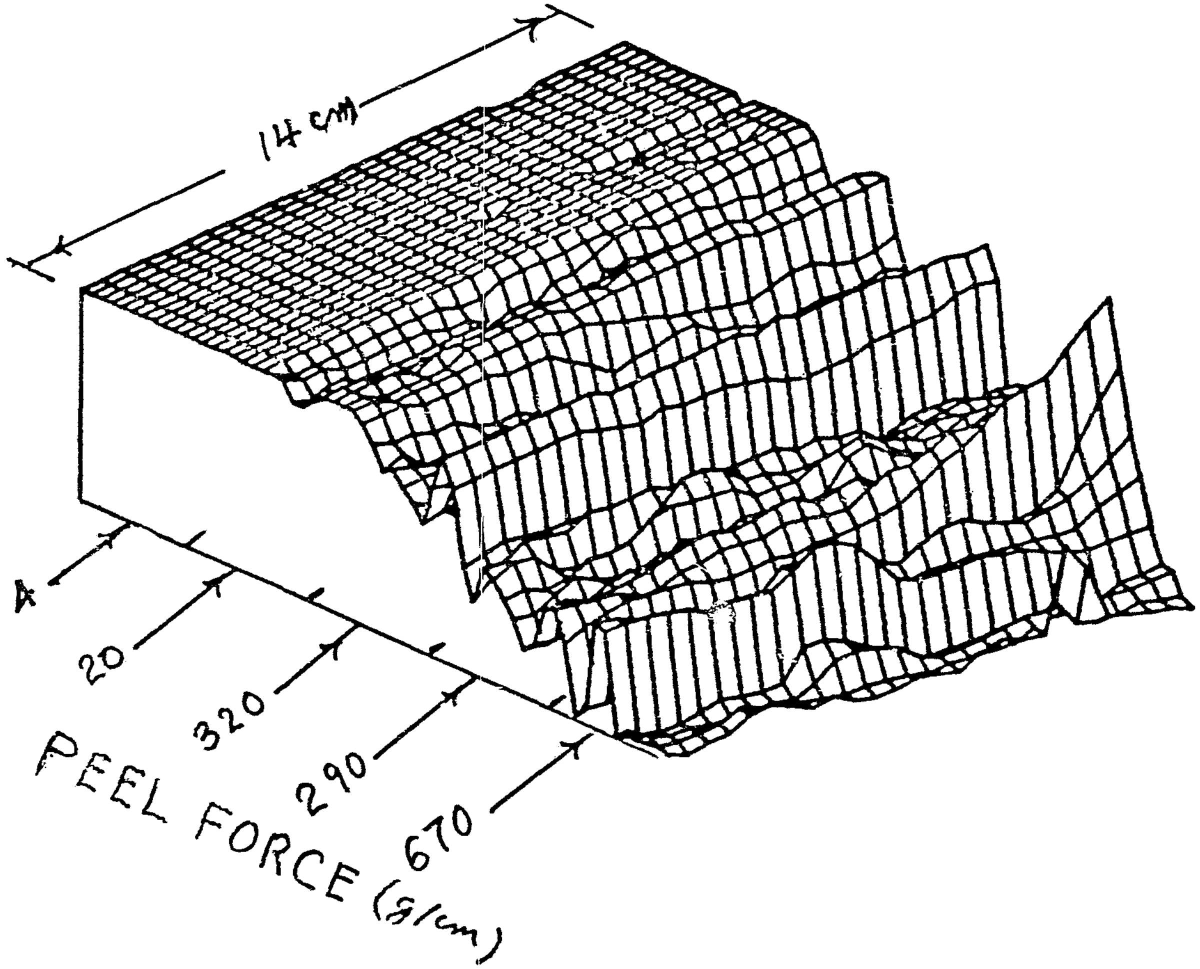


Fig. 12 Reduced contamination thickness map for RTV 102 silicone on

Table 5. Effect of RTV 655 Silicone Contamination on the Peel Strength of Polyurethane Bonded to Epoxy Paint on Aluminum from NASA

| Sample | | Contamination Level | Peel Strength (kg/cm) | Failure* Mode |
|---|---|---|---|---|
| 4-3-1 | as received | 0 | Tape Breaks | C |
| 4-3-12 | cleaned with THF | 0 | " | C |
| 4-3-3 | " | ? | 6 | C |
| 4-3-10 | | 0.01 | 8 | C/A |
| 4-3-8 | | 0.05 | 8 | C |
| 4-3-6 | | 0.1 | 1.2 | A |
| 4-3-4 | | 0.5 | - | A |
| 4-3-2 | | 1.0 | 2 | C/A |

* Cohesive Failure - C
Adhesive Failure - A

Painted panels (1' × 1') from NASA were divided into 12 regions, as in Fig. 13. The lower regions were left uncontaminated as a control. 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.

Figure 14 shows a PEE map of the panel represented in Fig. 13. A reduced thickness map is given in Fig. 15. The maximum reduced thickness (i.e., $x/\lambda$) is 1.66, so that the maximum contamination thickness is $1.66 \times 63 = 105$Å. Figure 15 reveals very little contamination in the control area, the masking tape residue area, the cotton glove and Kraft paper smudge areas and the car exhaust area. The fingerprint, 3-in-1 oil, lube grease, and silicone regions are strongly revealed.

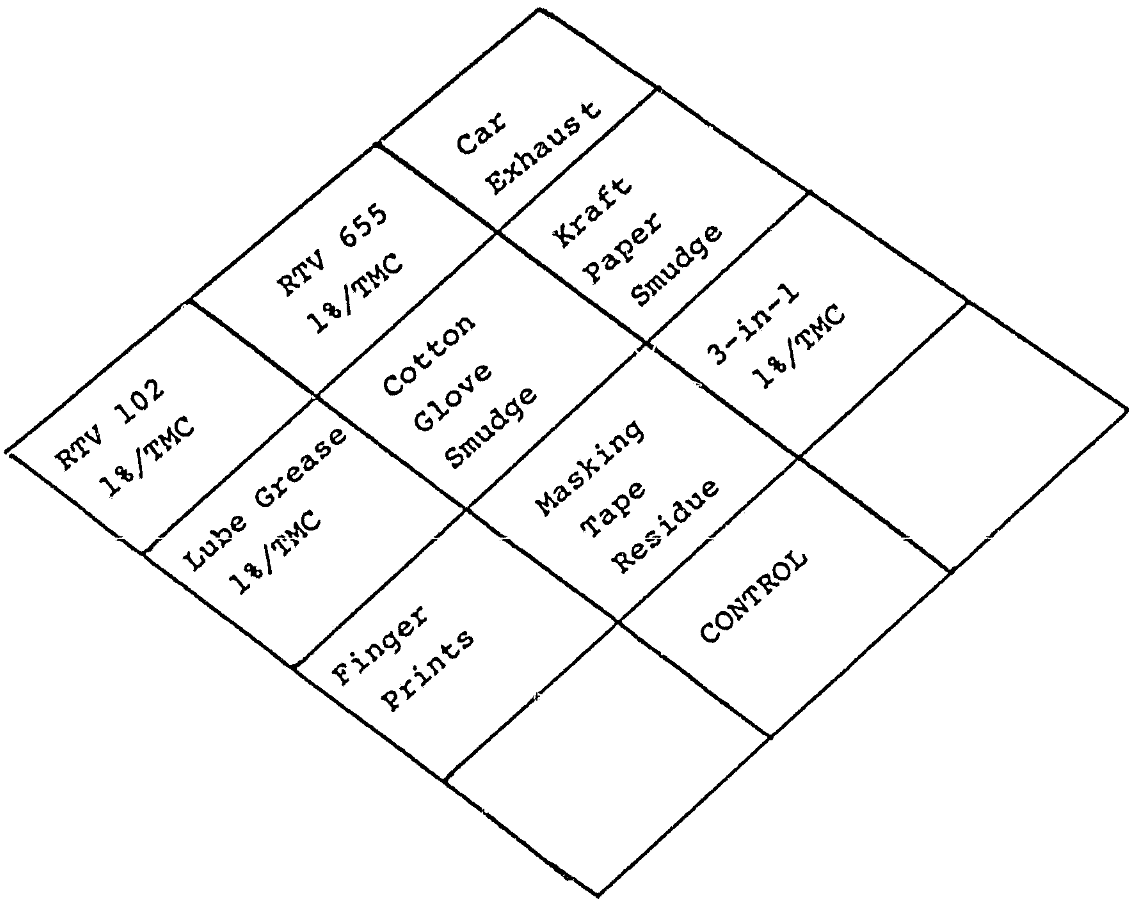


Fig. 13 Contamination pattern for 1' × 1' painted panel.

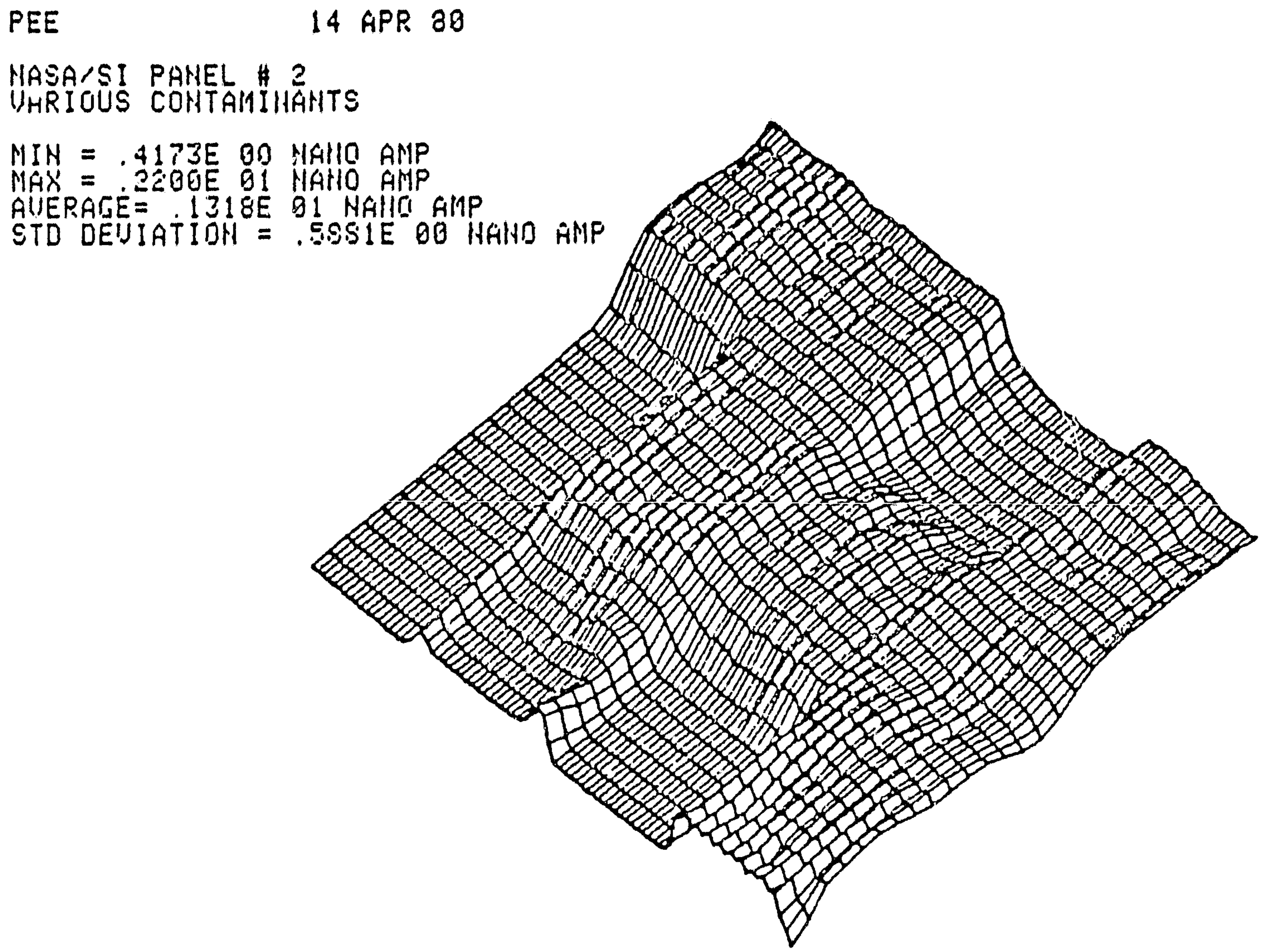


Fig. 14 PEE map of contaminated panel.

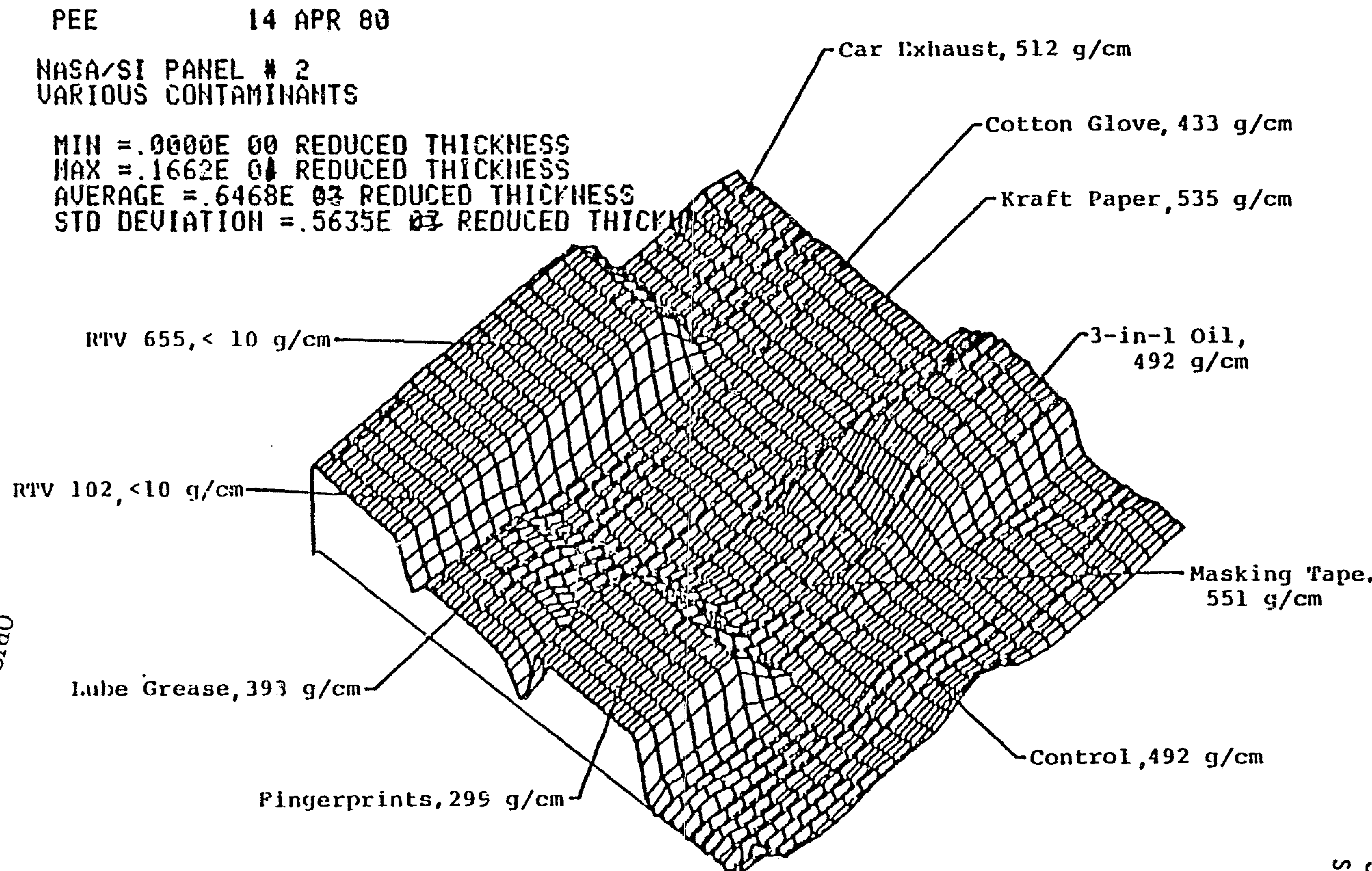


Fig. 15 Map of reduced contamination thickness. The peel forces are indicated for each area.

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After mapping the panel, half of each area (0.5" wide) was bonded with 3M masking tape and the other half (0.5" wide) was bonded with PR-365 polyurethane one part adhesive. A fiberglas cloth scrim was embedded in the PR-365 for backing strength. The PR-365 was approximately 1/16" thick. The tape and PR-365 strips were cut with an Exacto knife and pulled in 180° peel at 4"/min. The peel forces for the Scotch masking tape are indicated in Fig. 15. The control area, the masking tape residue, 3-in-1 oil, Kraft paper 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 at 299 g/cm and the silicone areas <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 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.

Although PEE is the most simple and efficient means for contamination detection, Fig. 16 shows that ellipsometry can detect silicones on paint. Ellipsometry is not very sensitive to the other types of contamination because the optical properties of the contamination and the paint are too close. Figure 17 shows that surface potential difference (SPD) measurements are very insensitive to all types of contamination on paint.

### 2.1.2 Tape Peel and Lap Shear Tests

Panels of epoxy painted Al 2219-T37, from NASA, were divided into 1" strips, as shown at the left of Fig. 18. Various contaminants were smeared on the different areas after wrapping Kimwipes around an aluminum block (1" wide) and soaking in the contaminant. For example, the top left quarter of the panel in Fig. 18, was smeared with CPR 483 foam component B. The strip marked 1 TMC wipe, was wiped once with a clean Kimwipe soaked with clean TMC. The strip marked 2 TMC wipe was wiped twice, each time with clean TMC soaked Kimwipe, etc.

NULL ELLIPS 15 APR 80

NASA/SI PANEL # 2
VARIOUS CONTAMINATION

MIN =-.2440E 03 UNITS
MAX = .1549E 04 UNITS
AVERAGE= .1301E 03 UNITS
STD DEVIATION = .2735E 03 UNITS

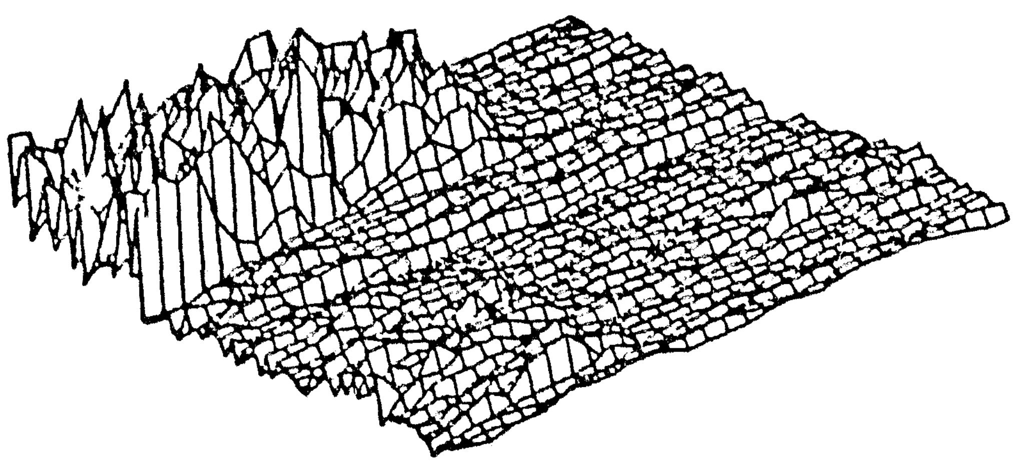


Fig. 16 An OFF NULL ellipsometric map of panel.

SPD 15 APR 80

NASA/SI PANEL # 2
VARIOUS CONTAMINANTS

MIN = .4949E 00 VOLTS
MAX = .6738E 00 VOLTS
AVERAGE= .6269E 00 VOLTS
STD DEVIATION = .2844E-01 VOLTS

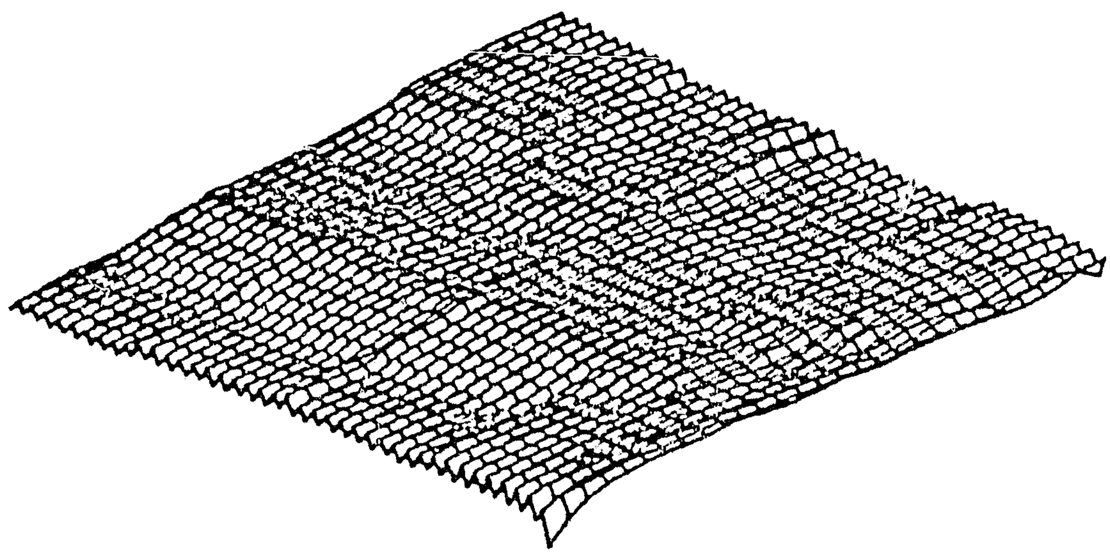


Fig. 17 An SPD map of the contaminated panel.

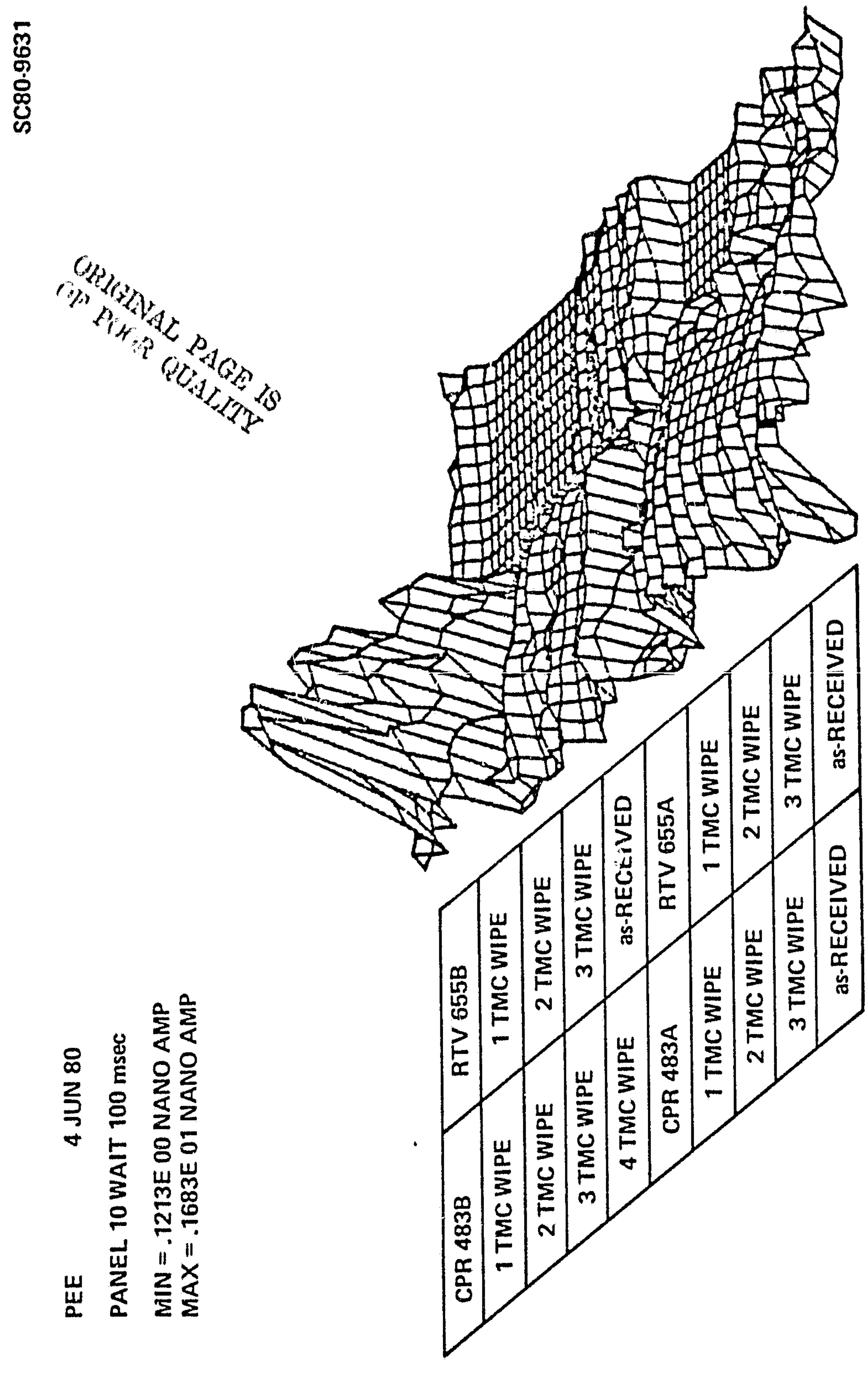



Fig. 18 PEE map.

The PEE map at the right of Fig. 18 indicates that RTV 655B attenuates the PEE electrons as does RTV 655A and CPR 483A. Wiping with TMC soaked Kimwipes, removes the contaminants and allows the PEE current to flow. The CPR 483B component is photoemitting.

The corresponding reduced thickness map is shown in Fig. 19, the low regions being clean and the high regions being contaminated. Figure 20 is a NULL Ellipsometer map. The RTV 655A and B are strongly revealed, the CPR foam components are not.

Table 6 lists the contamination levels and the corresponding Scotch Tape peel force, and the lap shear strength in columns 1, 2 and 3. In column 4 the lap shear failure modes are given. The lap shear samples were made by cutting 1" × 6" specimens from the bonded panels, then cutting each side to the bond line one half inch apart. After lap shear testing, one of the bonded ends was split by driving a chisel into the bond line. Column 5 gives the failure mode for the split joints. The average values of the PEE in each region are recorded in column 6. The code for the failure modes is given at the bottom of the tables. For example the lap shear joint for 1 TMC wipe of the CPR 483B (top left) failed adhesively at the aluminum-primer interface with some cohesive failure in the primer. The split part failed cohesively in the primer with some adhesive failure at the foam paint interface.

Figure 21 shows plots of Scotch Tape peel force (dashed lines), lap shear strength (solid lines) and the average PEE current (line-dash-line) that correspond to Figure 18, 19 and 20, and Table 6. The left hand ordinate values are for the Scotch Tape peel test in units of g/cm, and the lap shear test in units of $Kg/cm^2$. The right hand ordinates are for PEE in units of nanoamps. In each case the contamination drastically degrades the adhesion and more than two wipes with TMC soaked Kimwipe restores 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.

If the PEE acceptance window was 0.2-0.7 nanoamps, the cleaned areas would be accepted and the contaminated areas rejected in each case.

PEE 4 JUN 80
WAIT 100 ~~MICRO~~ m SEC
Panel 10

MIN =.0000E 00 REDUCED THICKNESS
MAX =.2629E 04 REDUCED THICKNESS
AVERAGE =.1761E 04 REDUCED THICKNESS
STD DEVIATION =.6369E 03 REDUCED THICKNESS

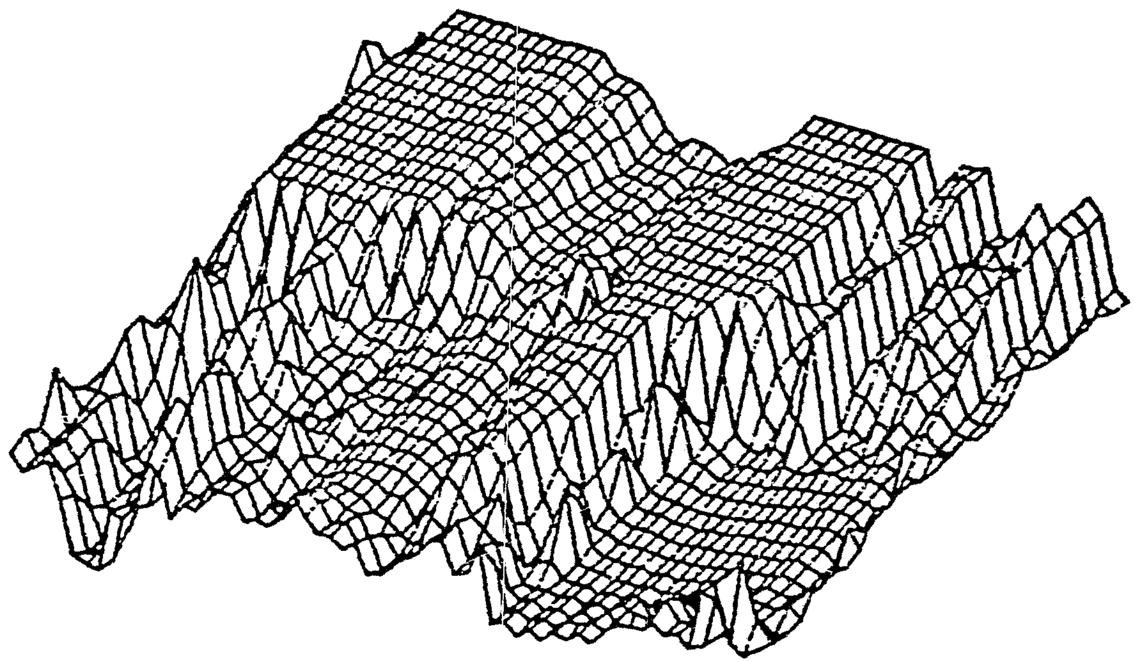


Fig. 19 Reduced thickness map.

NULL ELLIPS        4 JUN 80

Panel 10

MIN =-.1140E 03 UNITS
MAX = .1399E 04 UNITS
AVERAGE= .1878E 03 UNITS
STD DEVIATION = .2287E 03 UNITS

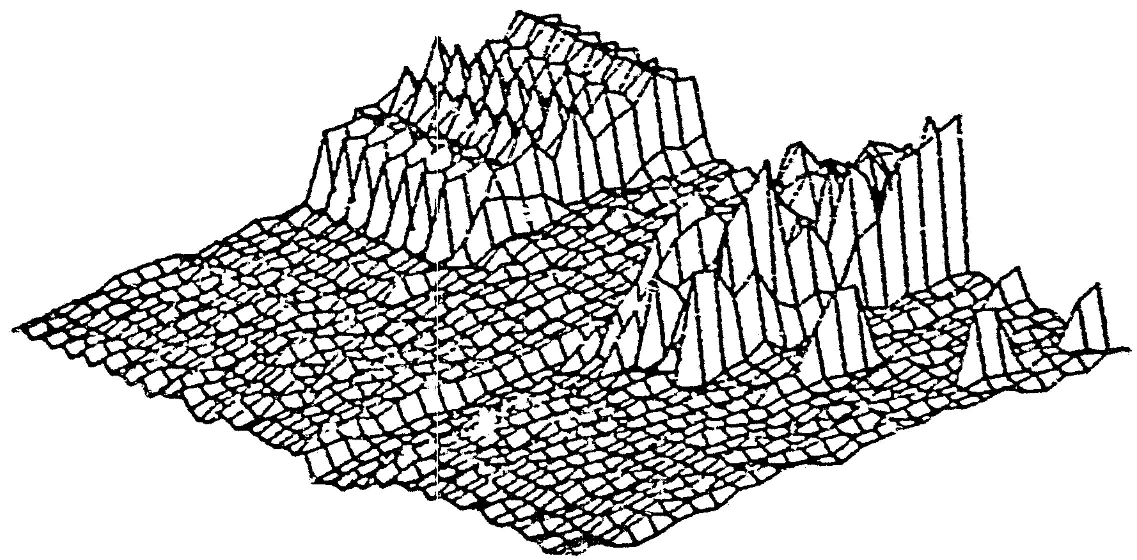


Fig. 20 Ellipsometric map.

Table 6. NASA/SI Panel #10

| | Area I.D. | Peel (Kg/cm²) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PEE Ave. (nA) | Area I.D. | Peel (g/cm) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PEE Avg. (nA) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CPR 483/B | Full Strength | <12.7 | 258 | A-F/PP | A-F/PP | 0.88 | Full Strength | <12.7 | 468 | A-F/PP | A-F/PP | 0.12 | RTV 655/B |
| CPR 483/B | 1 TMC wipe | 559 | 955 | [illegible] | C-P<br>A-F/PP | 0.36 | 1 TMC wipe | 279 | 787 | A-F/PP | A-F/PP | 0.12 | RTV 655/B |
| CPR 483/B | 2 TMC wipe | 1016 | 839 | A [illegible]<br>C [illegible] | A-F/PP | 0.41 | 2 TMC wipes | 635 | 855 | F/PP<br>C-P<br>A-Al/P | A-F/PP<br>C-P | 0.25 | RTV 655/B |
| CPR 483/B | 3 TMC wipe | 1029 | 952 | A-Al/P<br>C-F<br>A-F/PP | A-F/PP | 0.37 | 3 TMC wipes | 1130 | 1016 | A-Al/P<br>C-P | A-F/PP | 0.28 | RTV 655/B |
| CPR 483/B | | | | | | | 3 TMC wipes | 864 | 1000 | A-Al/P<br>C-P | A-Al/P<br>A-F/PP<br>C-P | 0.28 | As-Rec'd |
| CPR 483A | Full Strength | 64 | Broke while installing | | A-F/PP | 0.17 | Full Strength | <12.7 | Broke while cutting | | A-F/PP | 0.17 | RTV 655/A |
| CPR 483A | 1 TMC wipe | 876 | 694 | A-F/PP | A-F/PP | 0.48 | 1 TMC wipe | <12.7<br>762 | Broke while cutting | | A-F/PP | 0.14 | RTV 655/A |
| CPR 483A | 2 TMC wipe | 1048 | 968 | C-P<br>A-F/PP | C-P<br>A-Al/P | 0.70 | 2 TMC wipe | 1016 | 968 | A-F/PP<br>A-Al/P<br>C-P | A-F/PP | 0.21 | RTV 655/A |
| CPR 483A | 3 TMC wipe | 965 | 806 | C-P<br>A-Al/P | A-F/PP | 0.63 | 3 TMC wipe | 1092 | 871 | A-Al/P<br>C-P | A-F/PP | 0.27 | RTV 655/A |
| As-Rec'd | 2 | 889 | 661 | A-Al/P<br>A-F/PP<br>C-P | A-F/PP | 0.54 | 1 | 1016 | 635 | A-F/PP | A-F/PP | 0.32 | As-Rec'd |

Failure mode: A = adhesive
C = cohesive

Note: Peel tests - 100 mm/min
Lab shear - 0.5 mm/min

Material: F - foam
PP = paint - white gloss
P = primer - green epoxy
Al - aluminum

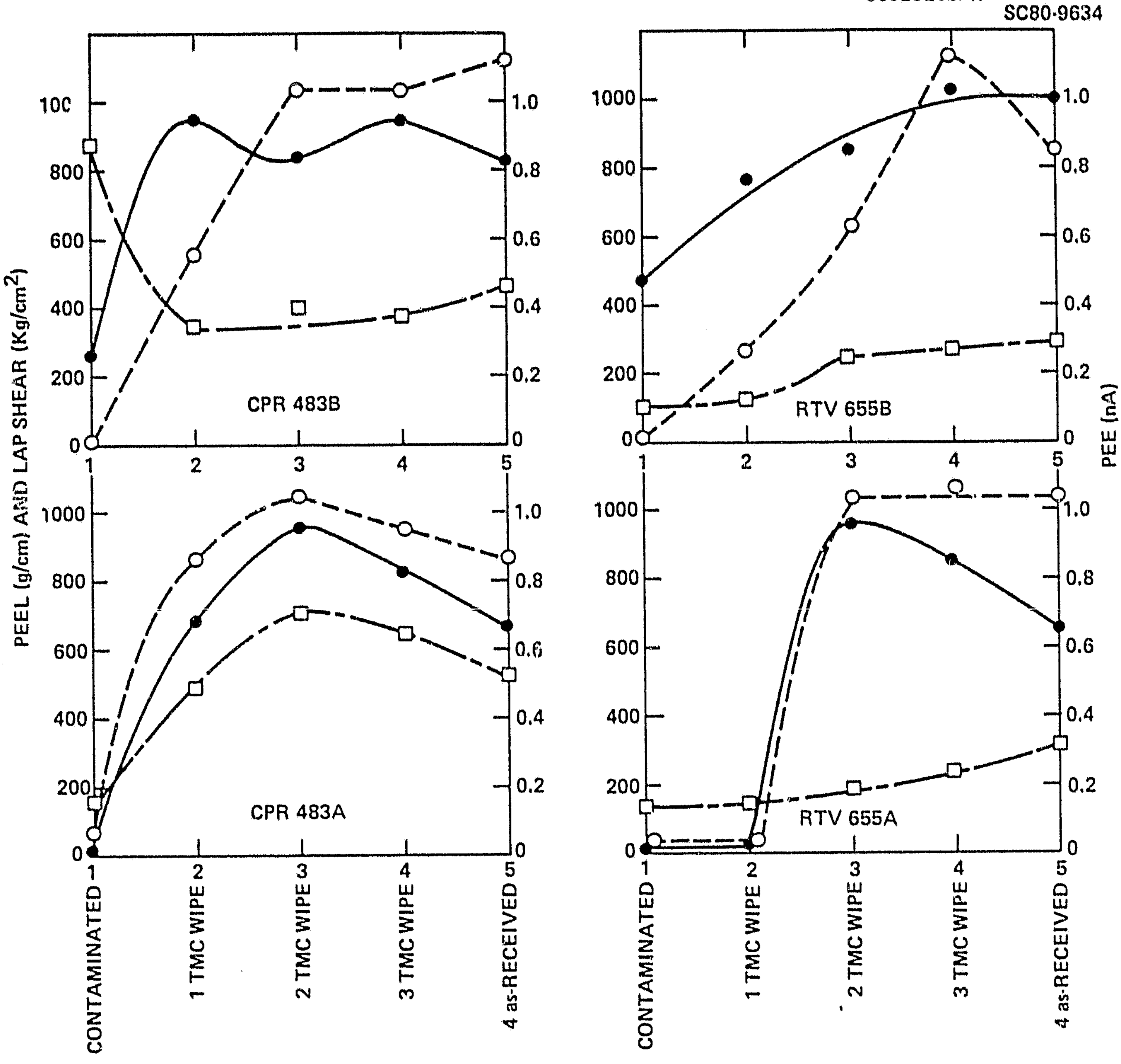



Fig. 21 Plots of peel force (dashed line) and lap shear strength (solid line) on left ordinate, and PEE values (line-dash-line) on right ordinates vs contamination and cleaning.

Figure 22 shows a PEE map for a painted panel contaminated with 7344 resin and 7119 catalyst, RTV 102 silicone, RTV 655 silicone and light oil. As for the CPR 384 part B, the 7119 catalyst is photoemitting, the rest of the contamination is electron attenuating. Figure 23 is a reduced thickness map. The photoemitting catalyst appears as a negative thickness on this map. Figure 24 is an ellipsometric map and only reveals the RTV 655 and 7344 resin.

The results in Table 7 are plotted in Fig. 25. The as-received region (lower right) yielded strong adhesion and high (clean) PEE values. On this panel, step 2 (along each ordinate) was 1 wipe with a dry Kimwipe, except for 2' (top left plot TMC Kimwipe). In each case the contamination greatly degraded adhesion, a dry wipe was inadequate but TMC clean produced strong adhesion. There are a couple of anomolous results in Fig. 25, the oil contamination degrades the Scotch Tape peel force but not the polyurethane foam strength. The TMC-cleaned oil gave an unexpected low PEE value. A recheck of oil showed that cleaning with TMC did not increase PEE.

In each case an acceptance PEE window of 0.2-0.7 nA would accept the clean (strong adhesion) area and reject the contaminated (low adhesion) areas.

### 2.1.3 New Epoxy Paint (Desota 616-346)

To demonstrate that the new epoxy paint (Desota 515-345, more chromates) behaves the same as the older formulation with respect to PEE and contamination detection and bonding, panels with this paint were obtained from NASA.

Figure 26 shows a PEE map for a painted panel contaminated with RTV 655B, oil, RTV 655 cured and cotton glove smear. The cotton glove had been rubbed over a cured RTV 655 area. Figure 27 shows the reduced thickness map, and Fig. 28 shows a different angle of the reduced thickness map to get a side view. Figure 29 is an ellipsometric map which only reveals RTV 655 as before, particularly if cured.

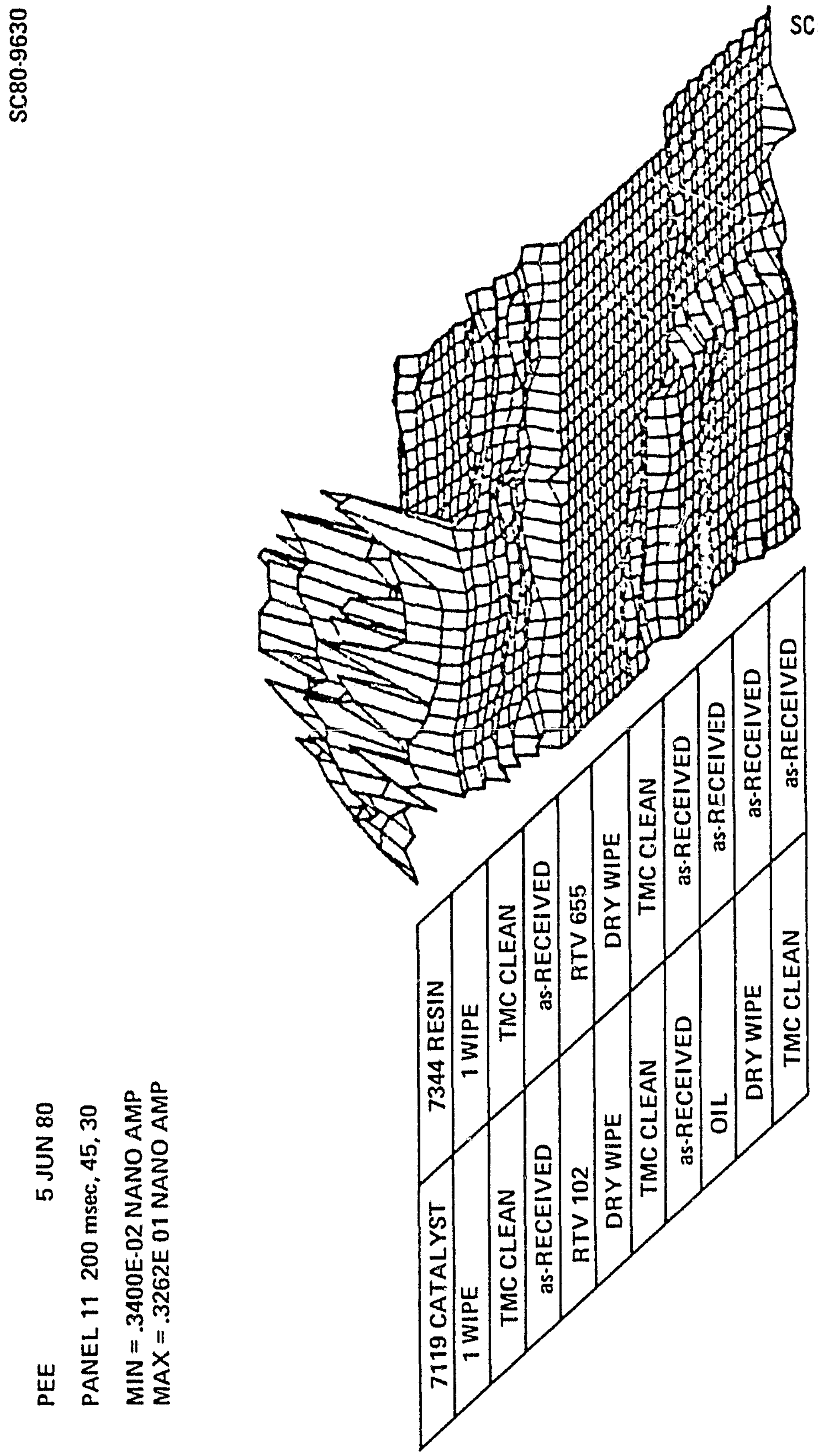



Fig. 22 PEE map.

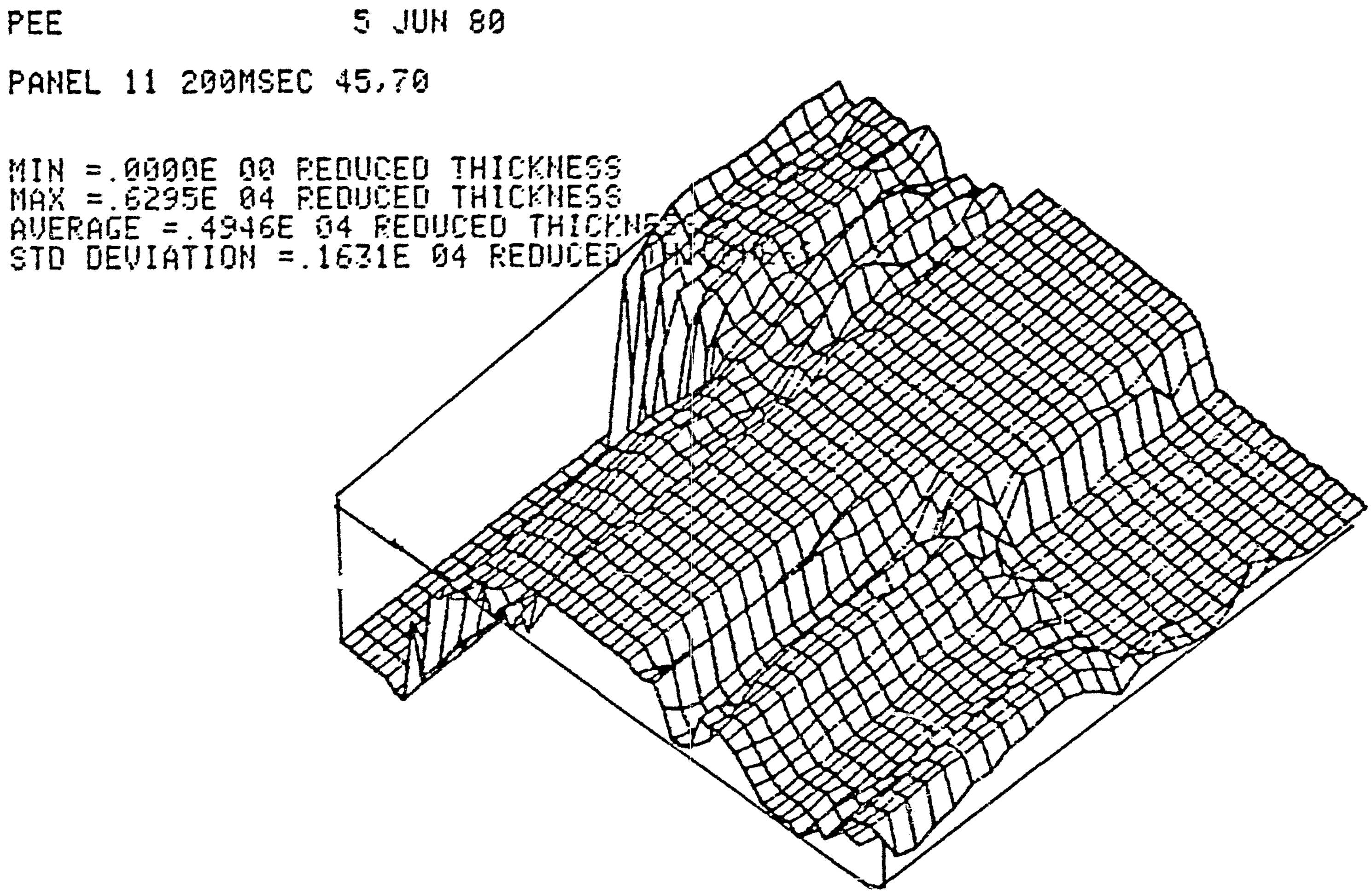



Fig. 23 Reduced thickness map.

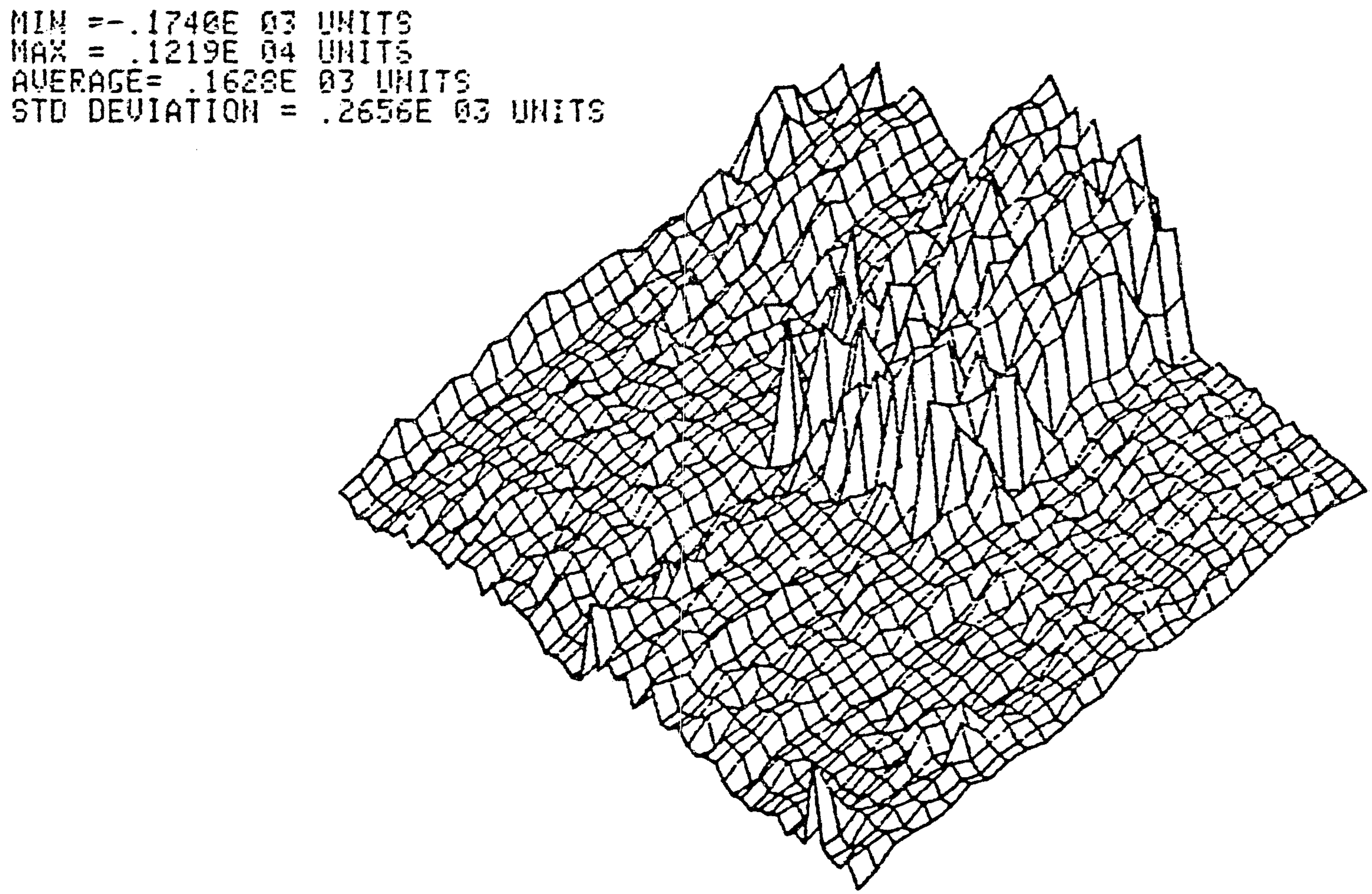



Fig. 24 Ellipsometric map.

Table 7. NASA/SI Panel #11

| | Area I.D. | Peel (Kg/cm²) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PEE Ave. (nA) | Area I.D. | Peel (g/cm) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PFF Avg. (nA) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 7119 Catalyst | Full Strength | <887 | 64 | A-F/PP | A-F/PP | 1.50 | Full Strength | <13 | 113 | C-F<br>A-F/PP1 | C-F<br>A-F/PP | 0.43 | 7344 Resin |
| 7119 Catalyst | 1 TMC wipe | 1016 | 548 | A-F/PP | C-P<br>A-F/PP | 1.66 | 1 dry wipe | <13 | 871 | C-P<br>C-F<br>A-Al/P | C-F | 0.28 | 7344 Resin |
| 7119 Catalyst | Try to "clean" TMC #1 | 940 | 822 | C-P<br>A-Al/PP | C-F<br>A-F/PP | 0.62 | Wipe with TMC | 1016 | 984 | C-P<br>C-F<br>A-Al/P | C-P | 0.34 | 7344 Resin |
| As-Rec'd #1 | 1 | 1016 | 887 | C-P | A-F/PP<br>C-P | 0.50 | 2 | 1016 | 548 | C-P<br>A-F/FP | A-F/PP<br>C-P | 0.37 | As Rec'd #2 |
| RTV 102 | Full Strength | 13 | <320 | A-F/PP | A-F/PP | 0.11 | Full Strength | <13 | Broke while cutting | | A-F/PP | 0.11 | RTV 655 |
| RTV 102 | Dry Kimwipe wipe | 25 | 300 | A-F/PP | A-F/PP | 0.11 | Dry Kimwipe wipe | <13 | Broke while installing | | A-F/PP | 0.11 | RTV 655 |
| RTV 102 | "Clean" with TMC #2 | 554 | 822 | C-P<br>A-Al/PP<br>A-F/PP | A-F/PP | 0.25 | Clean with TMC | <13 | 926 | C-P<br>A-Al/PP | A-F/PP | 0.11 | RTV 655 |
| As-Rec'd #3 | 3 | 686 | 935 | C-P<br>A-F/PP | C-P<br>A-Al/P | 0.39 | 4 | 889 | 871 | C-P | A-F/PP | 0.12 | As Rec'd #4 |
| Oil | Full Strength | 190 | 871 | C-P<br>A-Al/P | A-F/PP | 0.15 | ? | 920 | 952 | C-P<br>A-Al/P | A-F/PP | 0.61 | Undefined probably as-rec'd |
| Oil | Dry Kimwipe | 625 | 758 | A-Al/P<br>C-P | C-P | 0.33 | ? | 952 | 1000 | C-P<br>A-Al/P | A-F/PP | 0.69 | Undefined probably as-rec'd |
| Oil | "Clean" with TMC | 889 | 952 | A-Al/P<br>C-P | C-P<br>A-F/PP | 0.17 | ? | 889 | 957 | C-P<br>A-Al/P | A-F/PP | 0.71 | Undefined probably as-rec'd |

Failure mode: A = adhesive
C = cohesive

Note: Peel tests - 100 mm/min
Lah shear - 0.5 mm/min

Material: F - foam
PP = paint
P = primer
Al - aluminum

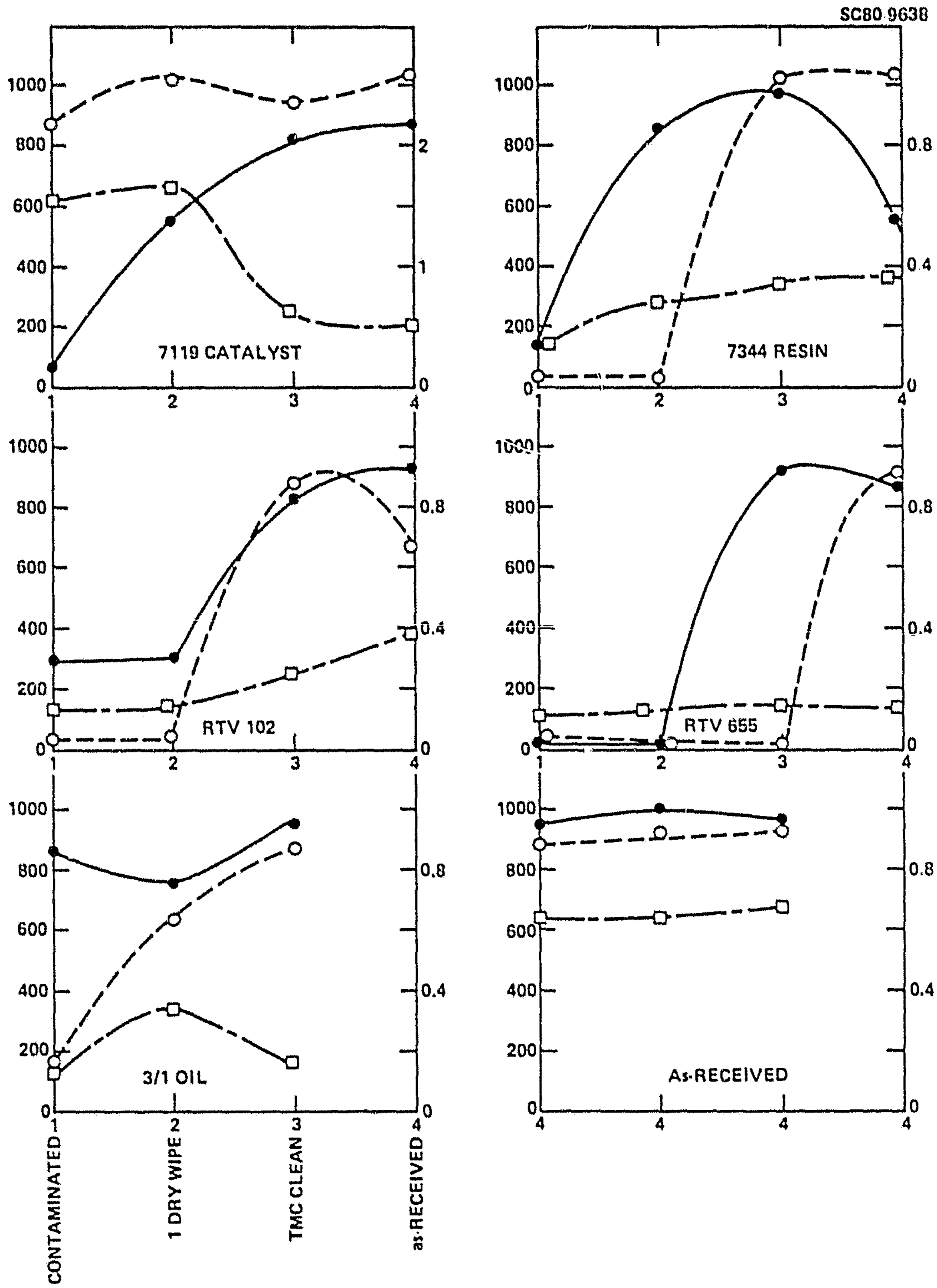



Fig. 25 Plots of peel force (dashed line) and lap shear strength (solid line) on left ordinates and PEE values (line-dash-line) on right ordinates vs contamination and cleaning.

SC80-9629

PEE 9 JUN 80

PANEL 12 - 45, 70

MIN = .1127E 00 NANO AMP
MAX = .6381E 00 NANO AMP

| | |
|---|---|
| RTV 655B | OIL |
| 1 DRY WIPE | 1 DRY WIPE |
| 1 TMC WIPE | 1 TMC WIPE |
| TMC CLEAN | TMC CLEAN |
| as-RECEIVED | as-RECEIVED |
| COTTON GLOVE FROM CURED RTV 655 | RTV 655 CURED |
| 1 TMC WIPE | 2 DRY WIPE |
| TMC CLEAN | 1 TMC WIPE |
| as-RECEIVED | TMC CLEAN |
| as-RECEIVED | as-RECEIVED |

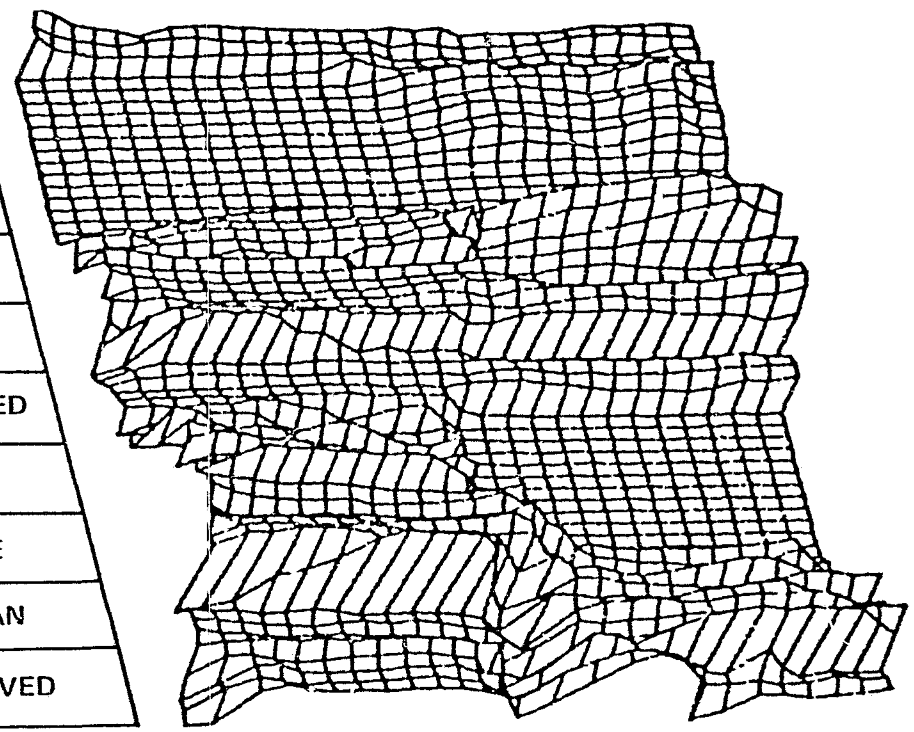


Fig. 26 PEE map.

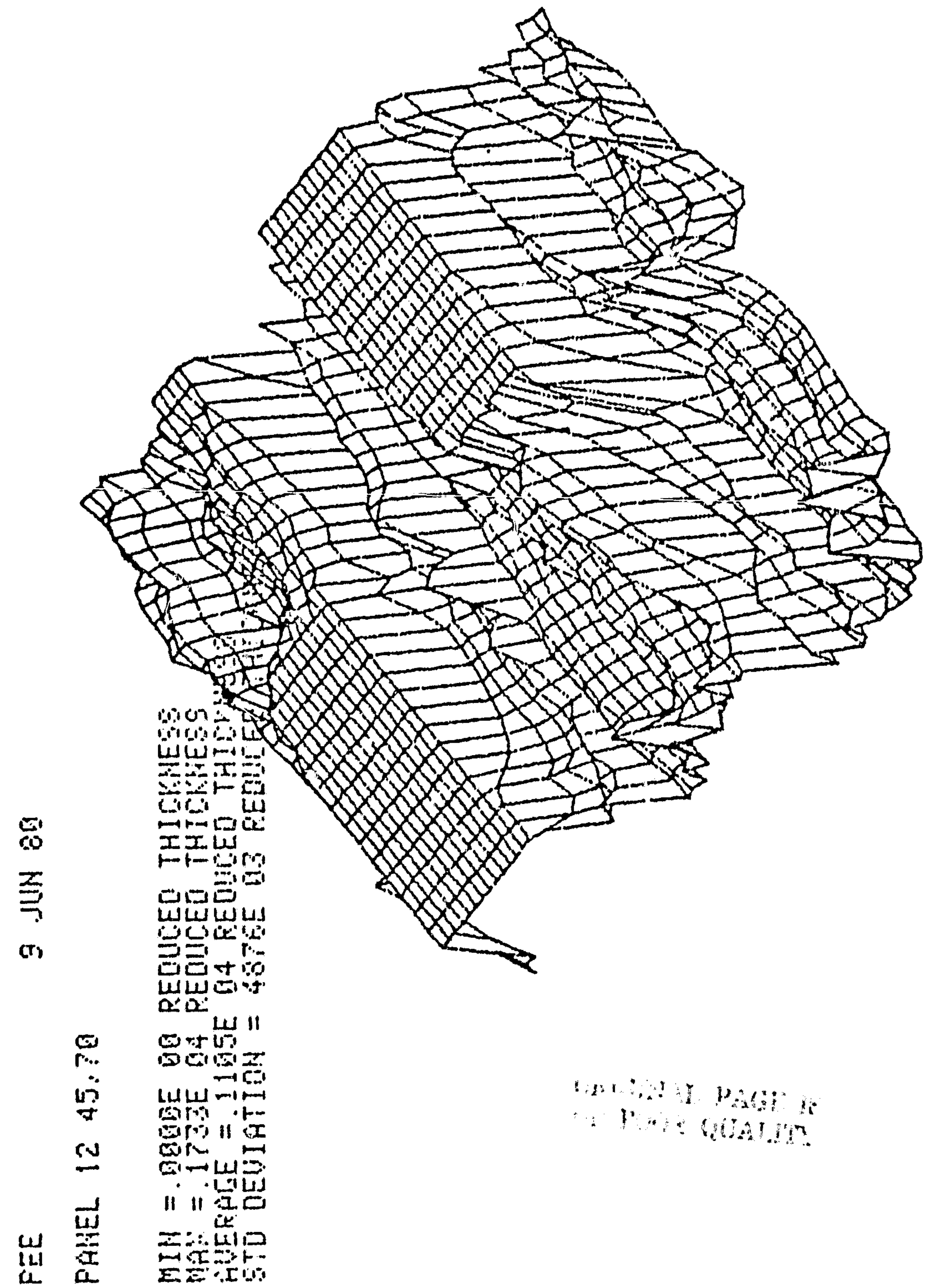



Fig. 27 Reduced thickness map.

FEE 9 JUN 80

PANEL 12 90.10

MIN = 0000E 00 REDUCED THICKNESS
MAX = .1733E 04 REDUCED THICKNESS
AVERAGE = .1105E 04 REDUCED THICKNESS
STD DEVIATION = .4876E 03 REDUCED THICKNESS

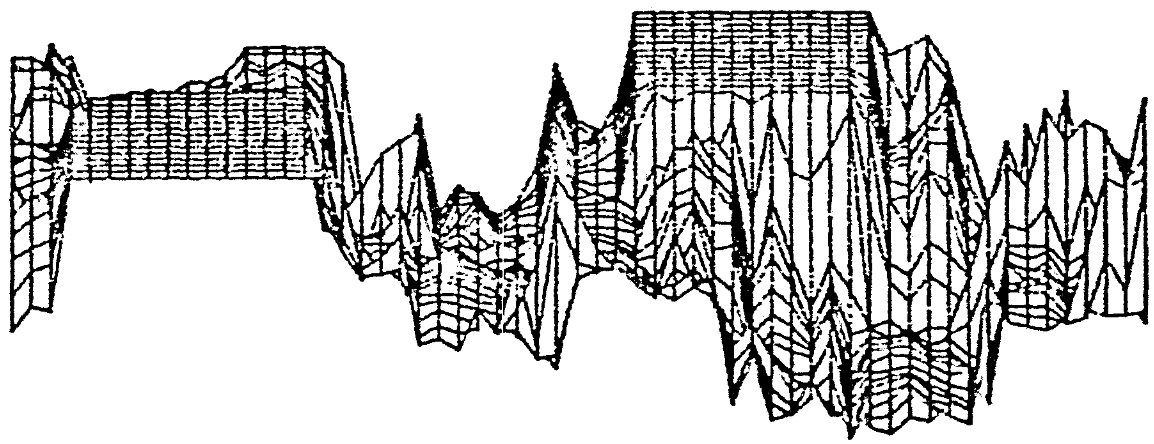


Fig. 28 Reduced thickness map (side view).

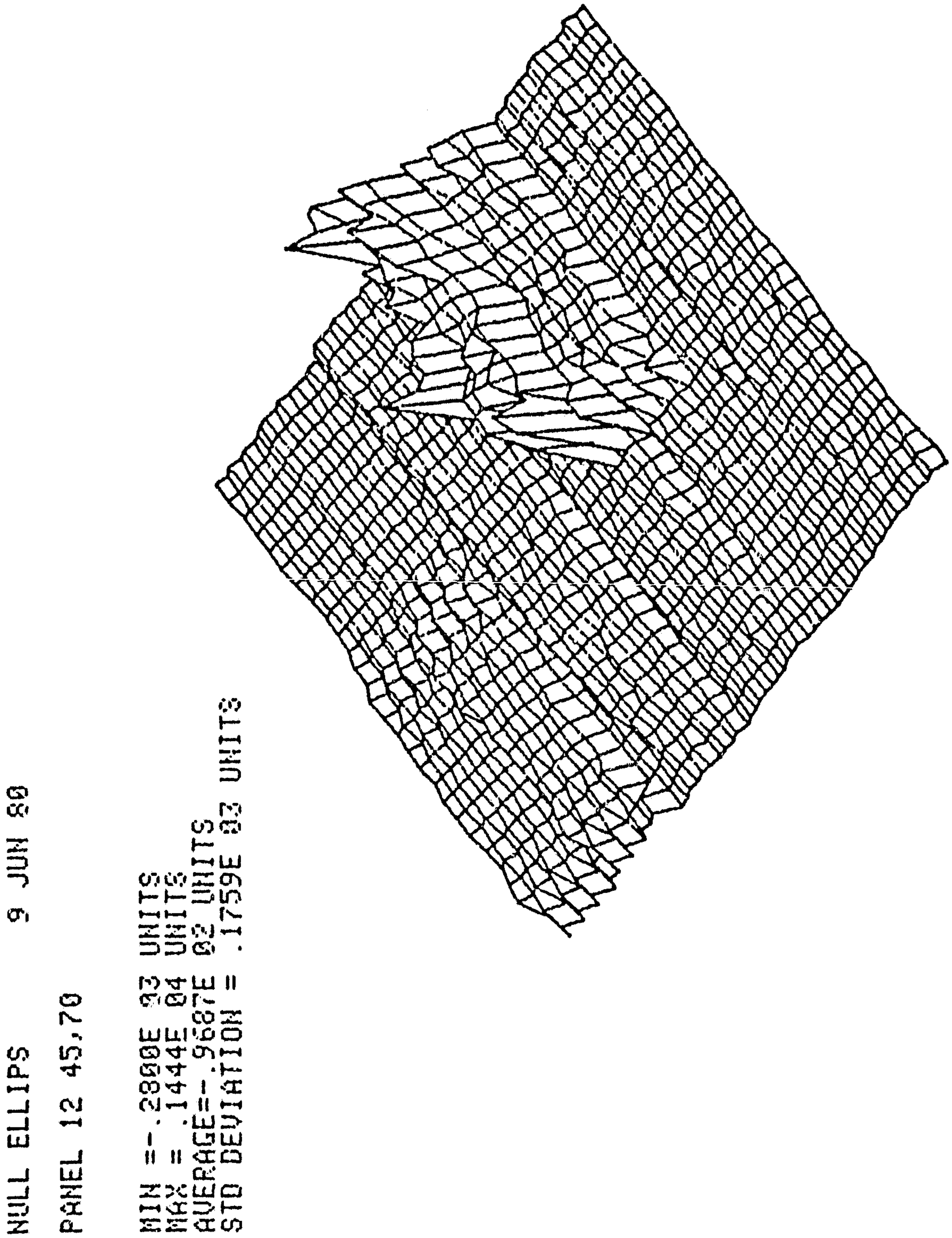



Fig. 29 Ellipsometric map.

Figure 30 shows plots from Table 8. The cotton glove does not degrade the adhesion after handling cured RT 655 although cured RTV 655 does. The oil degrades the adhesion but the surface is restored with a dry or TMC wipe. The PEE for oil contamination is ambiguous, perhaps due to non-uniform contamination. The oil is checked as a special case later.

Again, the PEE acceptance window accepts clean surfaces and rejects contaminated surfaces.

## 2.2 Unpainted Al 2219-T37

Although emphasis has been placed on the detection of contamination on painted surfaces, some work has been done to establish the detection technique for contamination on the aluminum surface after preparation for painting but prior to painting.

### 2.2.1 Tape Peel and Lap Shear Tests

Two panels (6" × 12") of Al 2219-T37 were given the surface treatment in Table 1, in preparation for painting, then contaminated in the unpainted condition. In this case 10% solutions of the contaminant in TMC were used, except for RTV 102 which was a 5% solution. Figure 31 shows a PEE map of these panels side by side. Figure 32 shows the reduced thickness map and Figure 33 gives a side view of the reduced thickness map. Figure 34 shows an ellipsometric map of the panel. The ellipsometer map shows all of the contaminants in this case, because the optical properties of the unpainted metal are sufficiently different from the contamination.

Figure 35 shows plots of adhesion strength vs contamination and cleaning for the unpainted panel from Table 9. In this case Desota epoxy paint was painted on the contaminated panel and a wire scrim was embedded for backing. A 90° peel test was performed to test paint adhesion. The paint was cured at room temperature for 48 hrs, then heated to 83°C for 10 min. The painted surface was cut in strips with an Exacto knife for peel tests. For the unpainted aluminum, the correlation between the Scotch Tape peel test and the paint peel test is excellent.

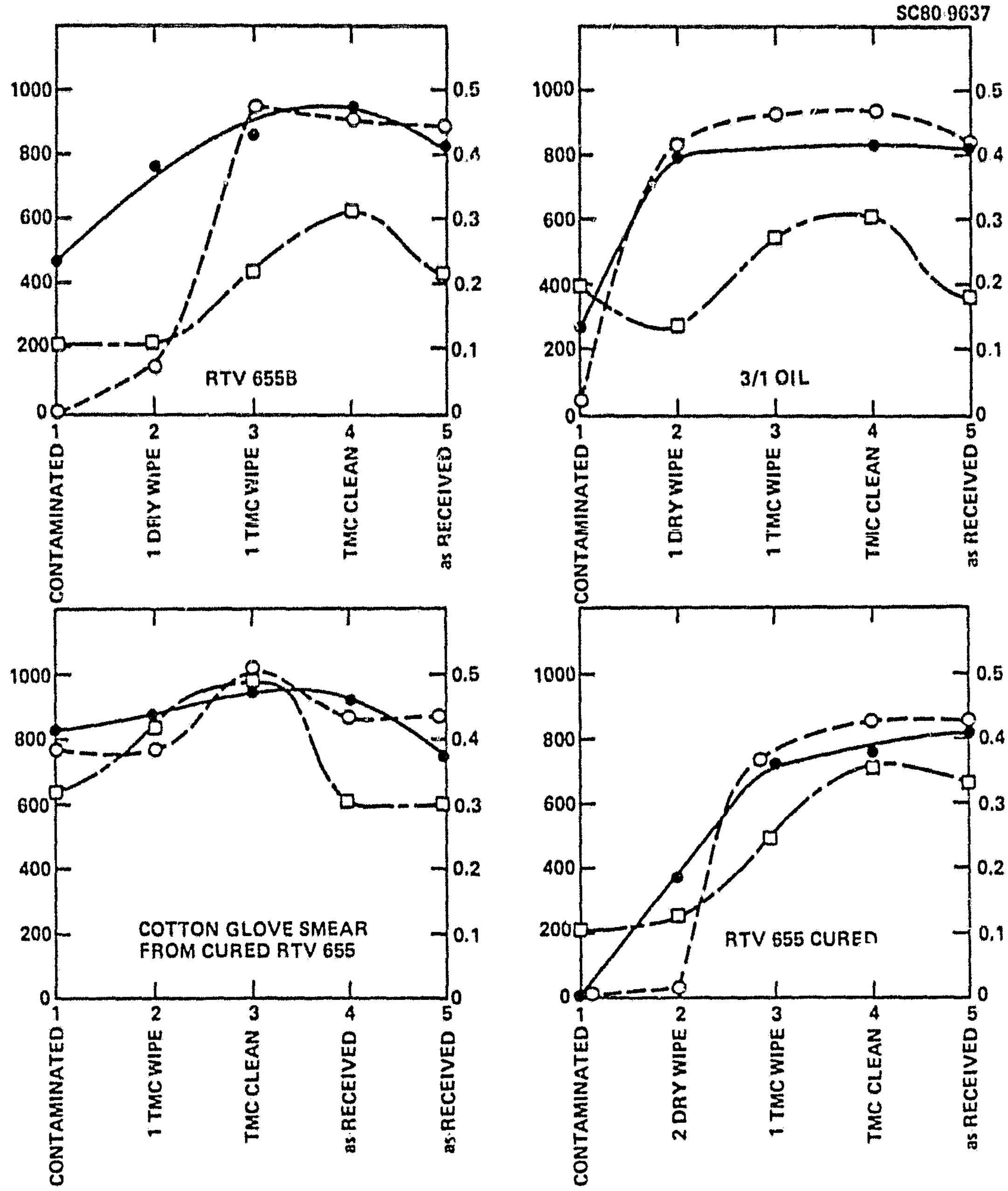



Fig. 30 Plots of peel force (dashed line) and lap shear strength (solid line) on left ordinates and PEE values (line-dash-line) on right ordinates vs contamination and cleaning.

Table 8. NASA/SI Panel #12

| | Area I.D. | Peel (Kg/cm²) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PEE Ave. (nA) | Area I.D. | Peel (g/cm) | Lap Shear (Kg/cm²) | Failure Mode Lap Shear | Failure Mode Split | PEE Avg. (nA) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| RTV 655/B | Full Strength | <25 | 483 | C-F | C-F | 0.11 | Full Strength | 51 | 252 | C-F | C-F | 0.20 | Oil |
| | 1 dry wipe | 152 | 774 | A-Al/P C-P | A-F/PP | 0.11 | Wipe dry | 825 | 800 | A-Al/P C-P | C-P | 0.14 | |
| | 1 TMC wipe | 940 | 864 | A-Al/P C-P | A-F/PP | 0.23 | 1 TMC wipe | 914 | broke installing | | C-P | 0.27 | |
| TMC | Clean TMC #1 | 902 | 958 | A-Al/P C-P | A-F/PP | 0.31 | Clean TMC #3 | 952 | 822 | C-P A-Al/P | A-F/PP | 0.31 | TMC |
| As rec'd | #1 | 889 | 806 | A-Al/P C-P | C-P A-F/PP | 0.23 | #4 | 864 | 812 | C-P A-Al/P | A-F/PP C-P | 0.18 | As rec'd |
| Cotton glove from cured RTV 655 | Full Strength | 762 | 806 | C-P A-Al/P | C-P | 0.31 | Full Strength | 19 | 0 | A-F/PP | A-F/PP | 0.11 | RTV 655 cured |
| | 1 TMC wipe | 762 | 896 | C-P | C-P A-F/PP | 0.47 | 2 dry wipe while uncured | 25 | 334 | A-F/PP | A-F/PP | 0.13 | |
| | Clean TMC #2 | 1016 | 952 | C-P | C-P | 0.50 | 1 TMC wipe | 737 | 726 | C-P A-F/PP | A-F/PP | 0.25 | |
| As rec'd | #2 | 889 | 919 | C-P | C-P | 0.28 | Clean TMC #4 | 889 | 774 | C-P | C-P | 0.36 | TMC |
| | #3 | 889 | 752 | C-P | A-F/PP C-P | 0.28 | Clean TMC #5 | 854 | 822 | C-P | A-F/PP | 0.34 | As rec'd |

Failure mode: A = adhesive
C = cohesive

Material: F - foam
PP = paint - white gloss
P = primer - green epoxy
Al = aluminum

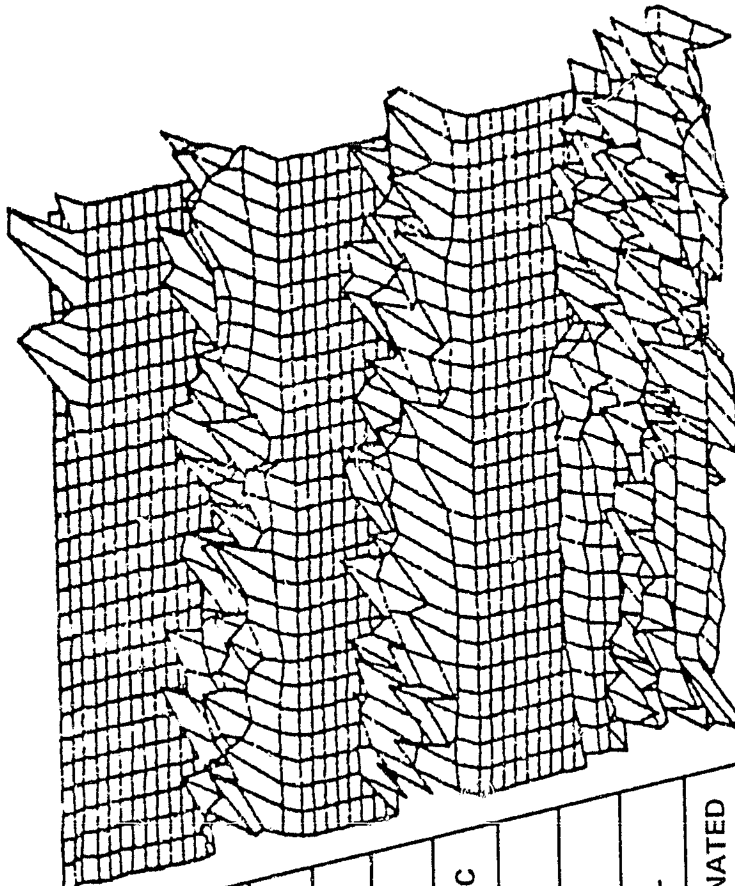
Note: Peel tests - 100 mm/min
Lab shear - 0.5 mm/min

PEE 6 JUN 80

PANEL 13 - 45, 70

MIN = .2900E-02 NANO AMP
MAX = .3269E 01 NANO AMP

SC80-9628



| | |
|---|---|
| 10% OIL/TMC | 10% RTV 655/TMC |
| 1 DRY WIPE | 1 DRY WIPE |
| TMC CLEAN | TMC CLEAN |
| FINGERPRINTS | 10% RTV 655A/TMC |
| 1 DRY WIPE | 1 DRY WIPE |
| TMC CLEAN | TMC CLEAN |
| 5% RTV 102/TMC | 10% RTV 655B/TMC |
| 1 DRY WIPE | 1 DRY WIPE |
| TMC CLEAN | TMC CLEAN |
| TMC CONTROL | TMC CONTROL |
| UNCONTAMINATED | UNCONTAMINATED |

Fig. 31 PEE map.

```
PEE                    6 JUN 80

PANEL 13 45,30

MIN =.0000E 00 REDUCED THICKNESS
MAX =.6634E 04 REDUCED THICKNESS
AVERAGE =.4676E 04 REDUCED THICKNESS
STD DEVIATION =.2366E 04 REDUCED THICKNESS

PRINT DATA ?

NEW FILE ?
```

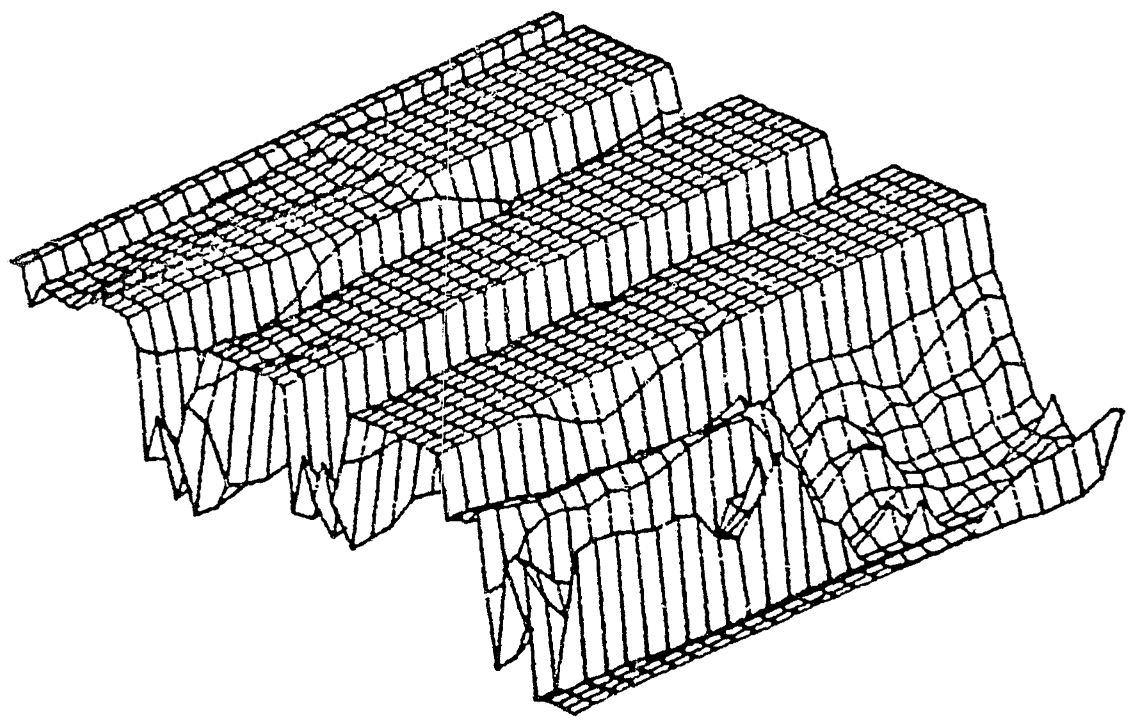


Fig. 32 Reduced thickness map.

FEE 6 JUN 80

PANEL 13 90,10

MIN =.0000E 00 REDUCED THICKNESS
MAX =.6634E 04 REDUCED THICKNESS
AVERAGE =.4676E 04 REDUCED THICKNESS
STD DEVIATION = 2366E 04 REDUCED THICKNESS

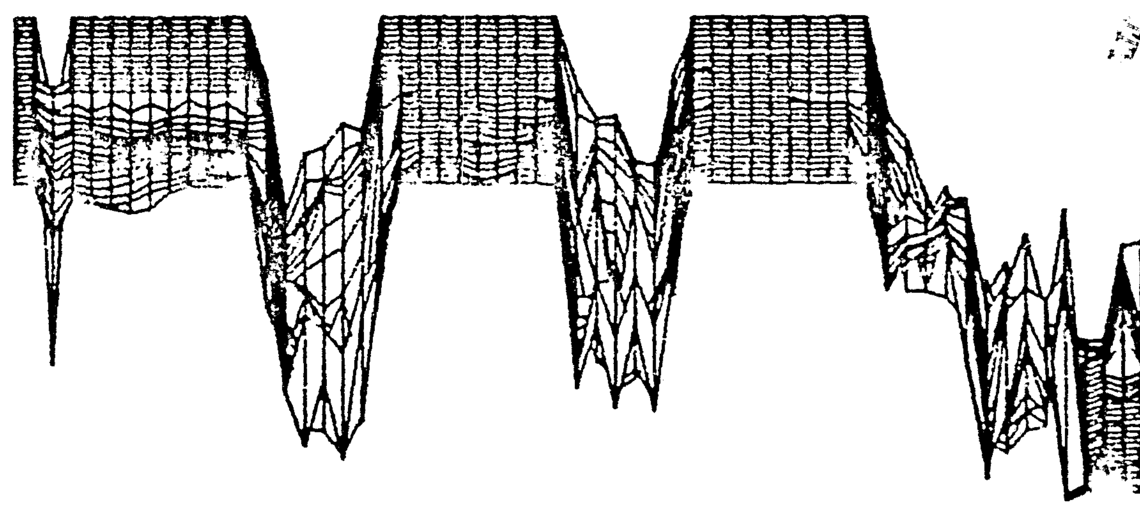


Fig. 33 Reduced thickness map (side view).

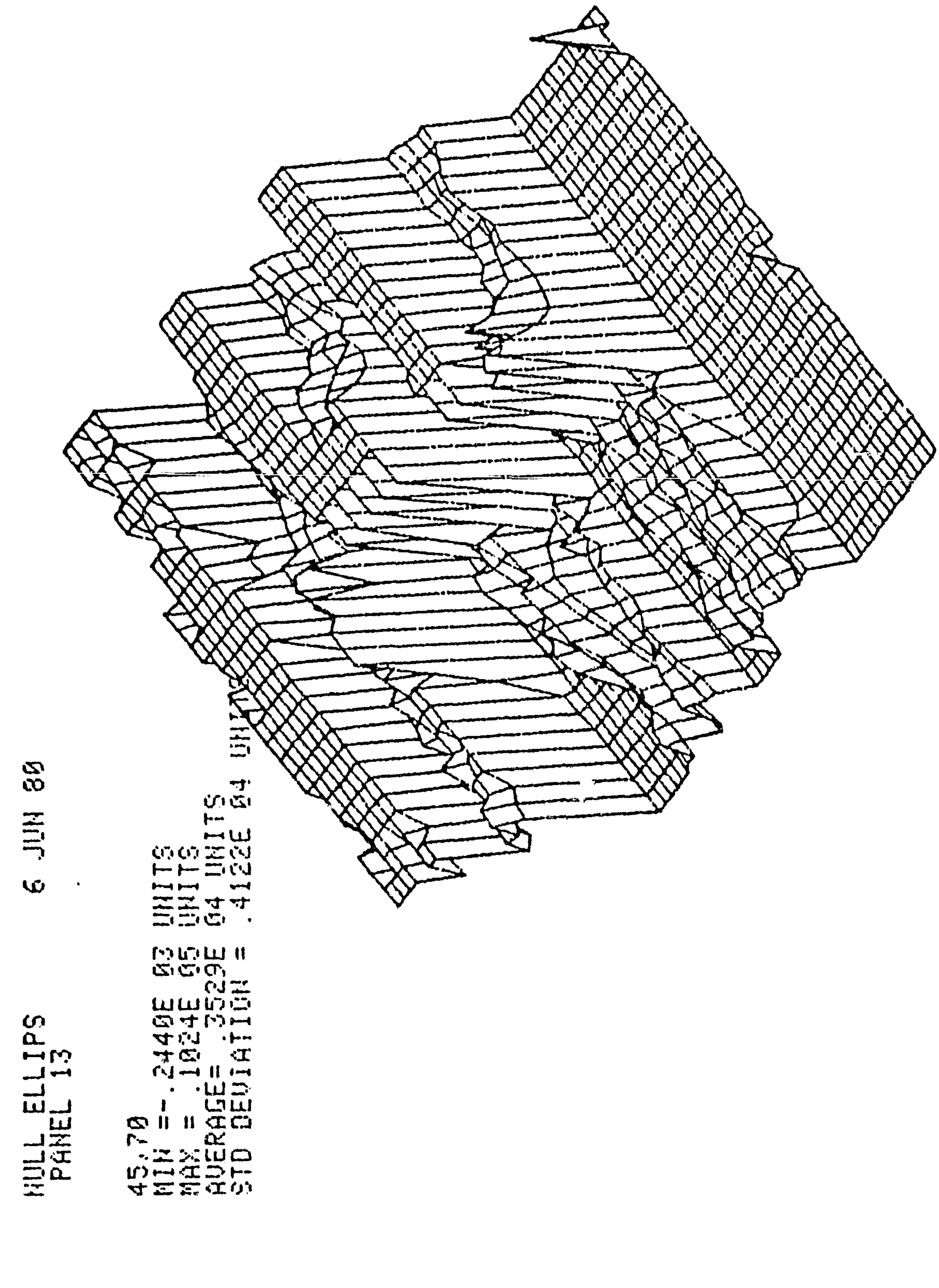



Fig. 34 Ellipsometric map.

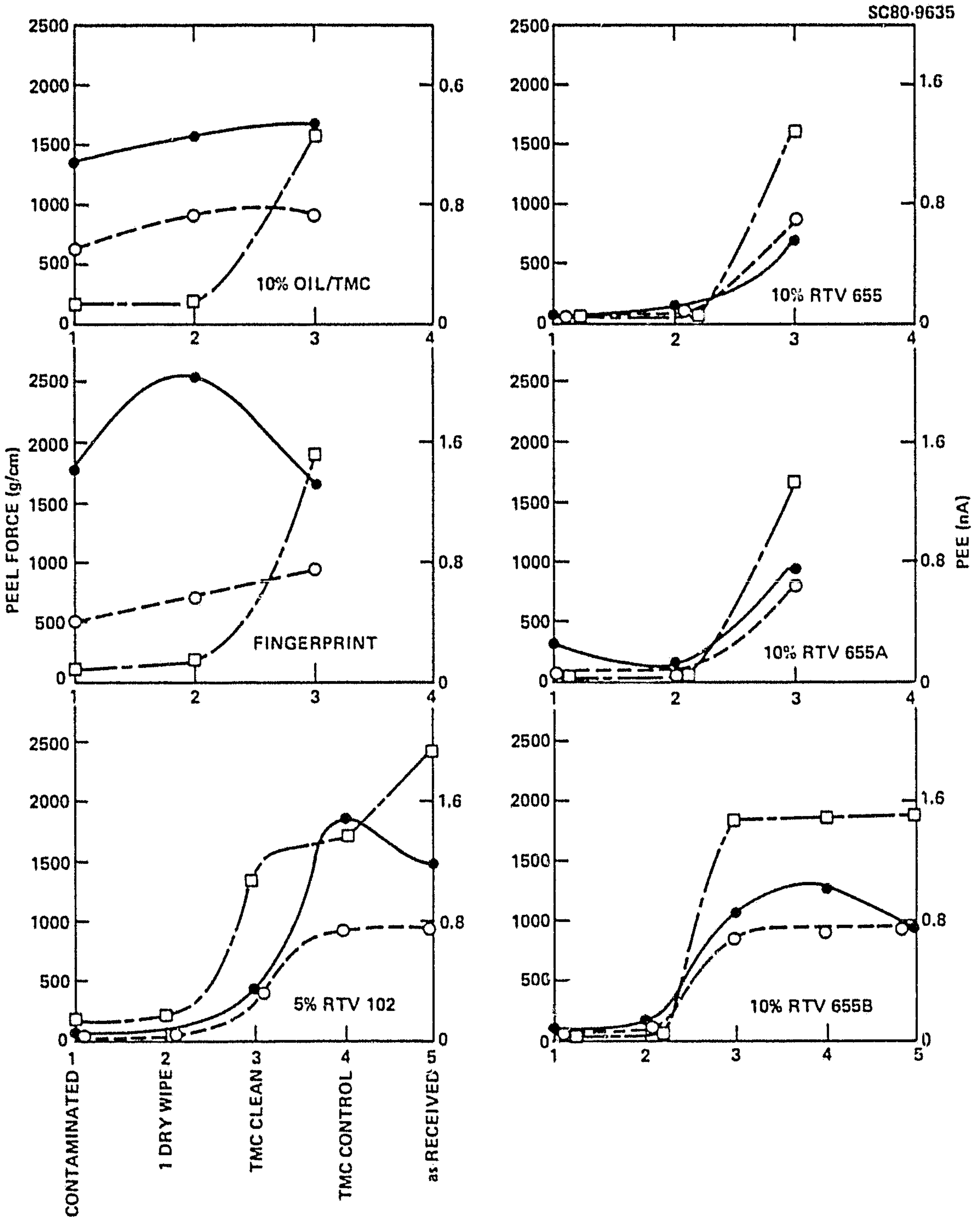



Fig. 35 Plots of peel force for Scotch Tape (dashed lines) and epoxy paint (solid lines) vs contamination and cleaning. The right hand ordinates are PEE values (line-dash-line).

Table 9. NASA/Si Panel #13 Unpainted

| | Area I.D. | Tape Peel (g/cm) | Paint Peel (g/cm) | Failure Mode | PFE Ave. (nA) |
|---|---|---|---|---|---|
| Oil 10% | Full Strength | 673 | 1362 | A-Al/PP | 0.16 |
| | 1 dry wipe | 889 | 1524 | A-Al/PP | 015 |
| | TMC clean | 889 | 1651 | A-Al/PP | 1.31 |
| Fingerprint | Full Strength | 500 | 1778 | A-Al/PP | 0.12 |
| | 1 dry wipe | 698 | 2540 | A-Al/PP | 0.12 |
| | TMC clean | 952 | 1651 | A-Al/PP | 1.54 |
| RTV 102 5% | Full Strength | 12 | 76 | A-Al/PP | 0.11 |
| | 1 dry wipe | 12 | 50 | A-Al/PP | 0.13 |
| | TMC clean | 406 | 470 | A-Al/PP | 1.10 |
| TMC Control | 1 | 990 | 1905 | A-Al/PP | 1.46 |
| As rec'd | 1 | 927 | 1486 | A-Al/PP | 1.94 |

(Left)

| Area I.D. | Tape Peel (g/cm) | Paint Peel (K/cm) | Failure Mode | PFE Avg. (nA) | |
|---|---|---|---|---|---|
| Full Strength | 12 | 12 | A-Al/PP | 0.11 | RTV 655 10% |
| 1 dry wipe | 12 | 127 | A-Al/PP | 0.11 | |
| TMC clean | 864 | 698 | A-Al/PP | 1.33 | |
| Full Strength | 12 | 342 | A-Al/PP | 0.11 | RTV 655 10% |
| 1 dry wipe | 12 | 184 | A-Al/PP | 0.11 | |
| TMC clean | 800 | 990 | A-Al/PP | 1.38 | |
| Full Strength | 12 | 95 | A-Al/PP | 0.11 | RTV 655/B 10% |
| 1 dry wipe | 12 | 158 | A-Al/PP | 0.11 | |
| TMC clean | 850 | 1080 | A-Al/PP | 1.48 | |
| 2 | 876 | 1270 | A-Al/PP | 1.48 | TMC Control |
| 2 | 889 | 889 | A-Al/PP | 1.54 | As rec'd |

(Right)

Failure mode: A = adhesive
C = cohesive

Material: PP - paint
Al - aluminum

Peel: Crosshead speed = 100 mm/min
"90° peel"

The oil had only a small effect on the peel strength, but in this case was strongly detected by PEE. Surprisingly, the oil and fingerprints gave exceptionally high peel strength as compared to uncontaminated areas. RTV 102, RTV 655A and B and A-B mixed degraded the paint adhesion dramatically. A dry wipe does not help (except for fingerprints) whereas TMC clean restores adhesion strength.

The PEE window is different for the unpainted panel because of greater emission yield. PEE values >0.4 nA reveal areas of acceptable adhesion; below this value, the surface should be cleaned.

3. Correlation Between Contamination, Detection and Humidity Endurance

To prepare wedge test samples for humidity endurance, two-part foaming urethane (CPR 483) was mixed and poured onto a NASA painted (Bostik 443-3-1) 1' × 1' panel. The mixture was spread evenly over the surface and another panel was placed on top. To prevent too much foaming, the mating panels were placed in a press and pressure was applied for 1/2 hr. After allowing the urethane to cure, the panel was cut into wedge specimens 1" × 6". One end of the specimen was split open by forcing a 1/8" wedge into the glue line. The initial crack length was recorded and the specimens were placed in a humidity chamber set for 60°C and 100% RH. The crack extension was recorded after 15 min, after 1 hr and after 16 hrs. The specimens were then split open.

Table 10 lists the type of contamination and level, increasing in the order 1, 2 and 3, in the left column. The next column lists the initial crack length for wedge insertion under dry conditions. Column 3 lists the crack extension after 15 min of humidity exposure, column 4 after 1 hr and column 5 after 16 hrs. The next three columns identify the mode of failure, cohesive (C), adhesive (A) or mixed (C/A) , during the initial crack formation (Initial), during crack extension in the humidity-chamber (RH) and during final splitting (Final).

Except for the RTV 102 set, which split completely, the control (uncontaminated) specimens averaged 2.5 ± 0.4 inches initially and opened to 3.3 ± 0.2 in. in 15 min. They only opened about 0.1 inches in 16 hrs. The

Table 10. Effect of Humidity on Contaminated Bond Joints
NASA/SI Panel #4

| Sample I.D. | Crack Length (in.) Original Crack | 15 min | 1 hr | 16 hrs | Failure Mode Initial | RH | Final |
|---|---|---|---|---|---|---|---|
| RTV 655/A | | | | | | | |
| Control | 2.1 | 3.1 | 3.2 | 3.2 | C | A | C |
| Level 1 | 2.3 | 2.7 | 2.8 | 3.2 | C | C | A |
| 2 | >5 | | | | A | - | - |
| 3 | >5 | | | | A | A | A |
| Lube Grease | | | | | | | |
| Control | 2.9 | 3.3 | 3.4 | 3.4 | C/A | A | A |
| Level 1 | 2.6 | 3.1 | 3.2 | 3.2 | C | C | C/A |
| 2 | 2.9 | 3.0 | 3.0 | 3.0 | C | C | A |
| 3 | 2.3 | 3.4 | 3.7 | 3.7 | C | C | C/A |
| 3-in-1 oil | | | | | | | |
| Control | 2.8 | 3.5 | 3.6 | 3.6 | C | A | A |
| Level 1 | 3.1 | 3.6 | 3.6 | 3.6 | C | C | C/A |
| 2 | 2.1 | 2.3 | 2.4 | 2.4 | | | |
| 3 | 2.6 | 3.6 | 3.7 | 3.7 | C | C | C/A |
| RTV-102 | | | | | | | |
| Control | >5 | | | | C/A | | |
| Level 1 | 3.2 | 2.9 | 4.0 | 4.0 | C/A | C/A | C/A |
| 2 | >5 | | | | A | | |
| 3 | >5 | | | | A | | |
| Fingerprint | | | | | | | |
| Control | 2.1 | 2.9 | 3.0 | | C/A | A | A |
| Level 1 | 2.5 | 3.5 | 3.6 | 3.6 | C | C | C |
| 2 | 2.5 | 3.2 | 3.3 | 3.3 | C | C | C |
| 3 | 1.9 | 2.9 | 3.0 | 3.0 | C | C | C |
| RTV 655 | | | | | | | |
| Control | 2.6 | 3.6 | 3.7 | | C/A | A | A |
| Level 1 | 3.4 | 3.8 | 3.9 | 3.9 | C | C | C |
| 2 | 3.7 | 4.1 | 4.2 | 4.2 | C | C | A/C |
| 3 | >5 | | | | A | | |

uncontaminated control samples failed cohesively or mixed during initial wedge insertion and adhesively at the paint-urethane interface while in the humidity chamber.

The surprising observation is that contamination shifted failure from adhesive type to cohesive type, with very little crack growth in any case. It appears that the polyurethane-epoxy paint bond is very insensitive to humid degradation. To check this further, urethane foam components were mixed and poured onto a painted panel that had been contaminated with fingerprints, lube grease, 3 in 1 oil, RTV 102, RTV 655 and RTV 655A. All of these contaminants (except fingerprints) were dissolved in TMC to make a 1% solution. PEE mapping revealed the contamination in every case. A wire screen was embedded to provide a backing for peel tests. The foam was cut into strips on the surface. With these low contamination levels none of the strips would peel, i.e., all strips failed by breaking the scrim. This panel was placed in water for three days. After the water soak, it was still not possible to separate the foam from the paint by the scrim or by scraping, chiseling, etc.

## 4. The Prototype Sensor

The prototype sensor is designed for mounting on the ET elevator for computer controlled scanning. The digital readout of the electrometer indicates whether the surface is contaminated or not, and a red light will come on if contamination is present. The prototype instrument and instructions for its use will be forwarded prior to Aug. 12.

## III. CONCLUSIONS

It is concluded that the PEE technique should be excellent for the nondestructive detection of contamination on either the unpainted or painted (epoxy) surfaces of the ET or SRB. Ellipsometry could be used for most contamination on unpainted aluminum but is restricted to silicone contamination on painted surfaces. The surface potential difference (SPD) technique is very poor for contamination detection on painted surfaces.

## ACKNOWLEDGEMENT

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