Two-Dimensional Contact Angle and Surface Tension Mapping
(As Presented at Pittcon® 96)

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Objective

The objective is to use robotics and video analysis to map contact angles and surface energy over specimen surfaces and form 3-D or topographic plots of the data. The scientist or engineer can then visualize how the surface varies spatially.

This system is useful in two different situations: where spatial sampling is necessary to establish a representative mean for a specimen, and where spatial sampling is necessary to map a deterministic variation. In the first case, the contact angle may be known to vary in a random fashion and many points must be measured to obtain an accurate mean (e.g., many metallic surfaces). In the latter case, the surface may vary deterministically because of process variations (e.g., plasma treated polymer surfaces).

The instrument may also be used to study precision fluid-dispensing problems, such as occur in the development of medical diagnostic kits that employ antibody-antigen reactions.

Equipment

The FTÅ200 Robotic Mapping Analyzer is an extension of the FTÅ200 instrument described in a similar paper at the 1995 PittCon show ("A New Dynamic Contact Angle System," Roger Woodward, PittCon '95 Poster Session). A 2-D grid of points is defined on the specimen surface where measurements are to be made. The grid spacing is user-defined, but 2 to 5mm is the typical range. Figure 1 defines the various robotic axes:

X_1, Y_1: Specimen Axes. The specimen can be moved in the horizontal X_1, Y_1 plane. Each axis has a travel of 150mm. The purpose of this movement is to bring the analysis spot into the "target" position on which the camera is focused.

X_2: Needle Axis. The two needles can be moved on this axis. This allows either needle to be positioned over the target, the wash station, or the waste station. Note the dispensing needles can only be moved on the X_2 axis or the Z axis, they have no Y motion.

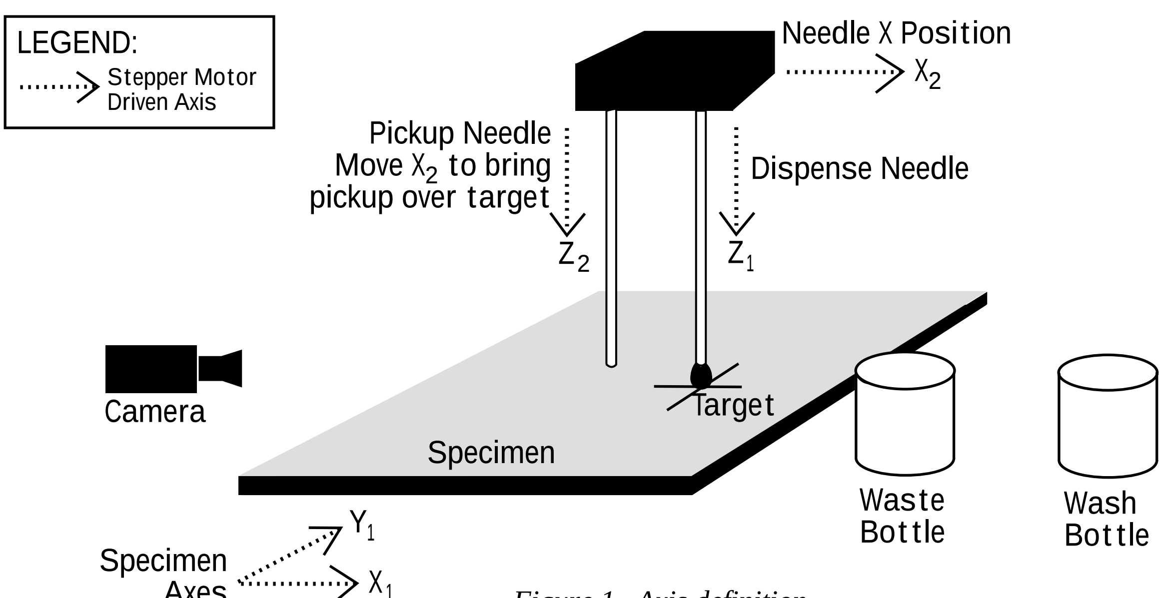

Figure 1. Axis definition.

Z_1, Z_2: Dispense Axes. Two independent liquid handling systems, each with a precision syringe pump, move vertically. Nominally, one is for dispense and the other for pickup, but under some circumstances both can be used for dispense (particularly for two-fluid contact angle studies). Each pump is equipped with a two-way valve so it may withdraw fluid from a larger reservoir and, after throwing the valve, dispense it. The travel on each Z axis is 25mm.

Additional pumps and dispensing axes can be added; so one could have two dispense needles and one pick-up needle, for example.

The robotics place a drop of test fluid on each grid point and the video system then captures an image of the scene and automatically performs the contact angle analysis. Surface energy can be extracted from contact angle by any of four analytic models.

After the point measurement is made, a separate tip is brought into place over the drop and the drop is picked up. Of course, if the drop wets the surface well, not all fluid will be removed, but most will be removed so the remaining fluid, if any, will not interfere with subsequent tests at nearby locations. Most particularly, there will not be a drop remaining to visually obscure subsequent images.

Measurement Protocol

The photos is Figures 2–6 show the process of placing a drop on the surface, capturing data, and then removing the drop.

Figure 2 shows a small pendant drop on the dispense needle. The system starts with the syringe pump primed, but no drop hanging. Then a precise amount is slowly dispensed to form the pendant drop shown in the figure.

The needle is lowered until the pendant drop touches the sample. Depending on the surface energy of the sample, the drop will either detach or remain, temporarily, attached to both the needle and the surface, as in Figure 3. This method does not affect the resulting contact angle as long as the needle is not lowered further. In particular, notice the contact angle in Figure 3 is higher than the final contact angle in Figure 4. In Figure 3 the drop has not spread completely because it is still receiving support from the needle above. After the needle is withdrawn, it will relax to its final (true) contact angle.

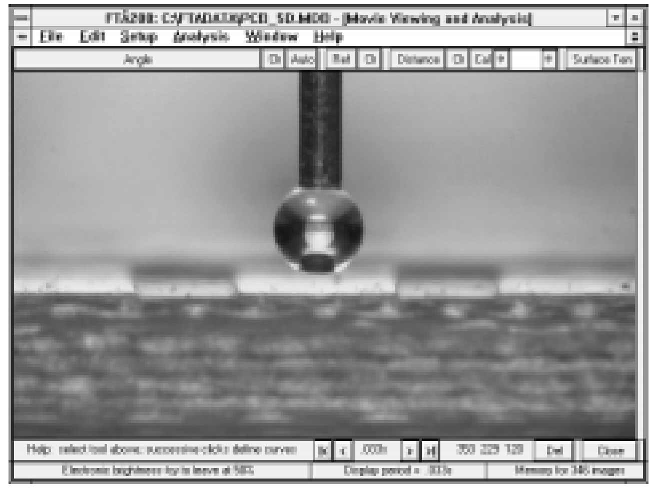

Figure 2. Pendant drop.

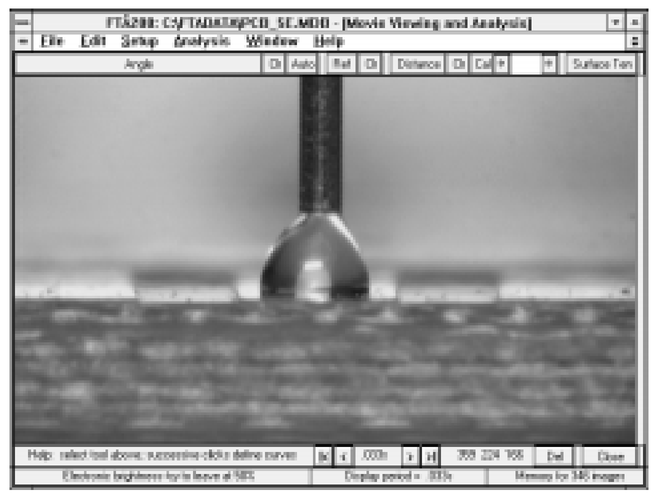

Figure 3. Touching off.

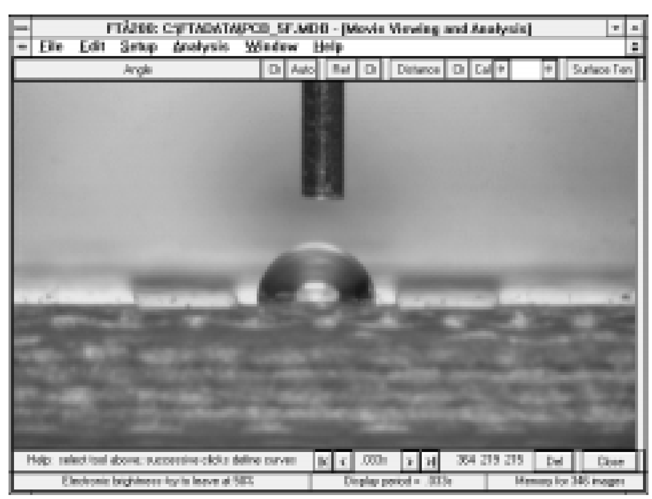

Figure 4. Data acquisition.

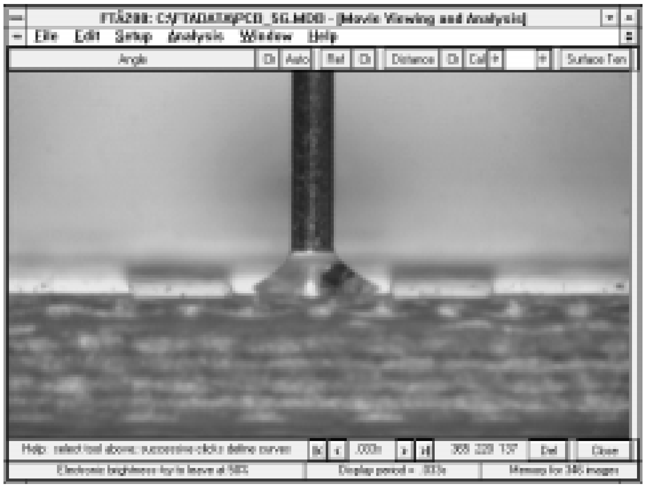

Figure 5. Pickup drop.

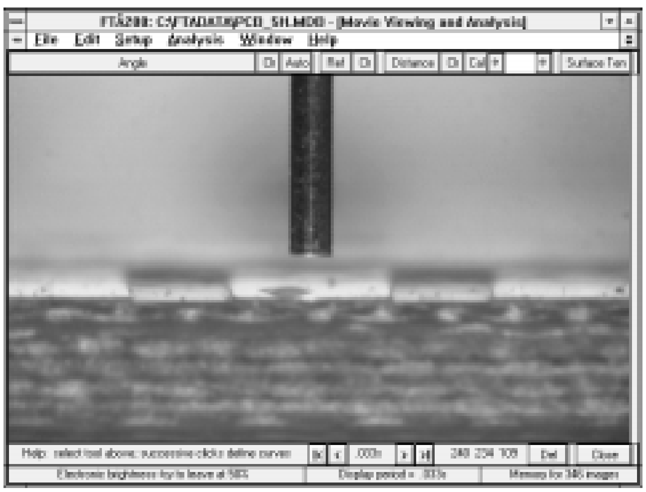

Figure 6. Residual after pickup.

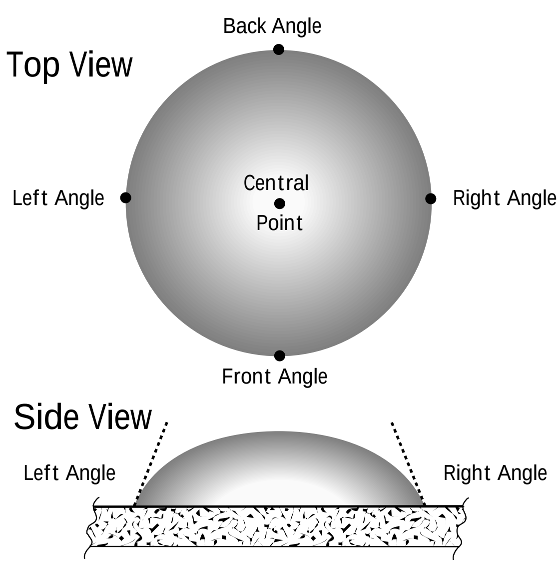

Figure 7. Measurement locations for sessile drop.

The needle is raised in Figure 4 so that it detaches from the sessile drop. The drop spreads as necessary to assume its final contact angle. This image is captured for subsequent analysis.

The pickup needle is brought into place and inserted further than the dispense needle. This is shown in Figure 5. The syringe pump runs in “reverse” to pickup the drop. The receding contact angle is visible in the photo.

At some point the sessile drop will detach from the pickup needle. The closer the needle is to the sample surface, the more of the original drop will be picked up. But eventually the “seal” will be broken and some residual will be left on the sample. This is shown in Figure 6. During this process some air will be drawn into the pickup needle. This is the primary reason to have a second needle for pickup. We could not go on to the next measurement with air in the system if we were using the same needle for dispense and pickup. If we wish to carry out a two-fluid contact angle protocol, then each needle must pick up its own drop. This requires that the needles move to the waste station and dispense enough fluid to ensure no air remains in their system; this takes additional time.

This five-step cycle takes about ten seconds when the second needle is used to pick up the drop. A grid of 100 points (10 × 10) can be tested in about 15 minutes running time. The grid format is quite flexible, so the 100 points, for example, need not be uniformly distributed over the specimen if there is an advantage to non-uniform spacing.

Data Processing

Each captured image is analyzed for contact angle. This is done automatically using prior data as a template for each new drop. The template is practical because, since the drop size is fixed, the images are similar, even though the contact angle will vary. The primary difficulty comes in establishing the baseline with a non-flat specimen. The system treats all images from the grid as one “movie,” so most images can be analyzed without operator intervention, even if a few require operator assistance.

The FTÅ200 computes a separate left-side and right-side contact angle when it is in its non-spherical model mode (best fit of polynomial to the drop’s periphery). It also measures the base width, or the distance between the left and right contact angle locations.

Figure 7 illustrates this with a side-view and a top-view of a sessile drop. The left-side and right-side measurement points are shown, as are three other possible derived measurement points. If we presume the drop is circular in plan (top view), then by linear interpolation we can estimate the front and rear values. This gives us four measurement points, of which two are clearly independent, for each drop when we use the non-spherical model.

Alternatively, we could use a spherical model (best fit of circle to curve) and assign the same value to the four (left, right, front, back) points. We could also assign the spherical value to the center of the drop.

Figure 8 illustrates the overall data flow. At this point in the description we have captured a BMP image, performed the contact angle analysis, and obtained an X-Y-Z data map. The important aspect of this map is that the X,Y locations are random (wherever the drop edges fell).

The topographic plot we desire requires data points on a regular grid. We obtain these by interpolation on the X-Y-Z map. Notice we can have a much higher resolution in the topographic grid than implied by the number of drops or measurement points.

In the example on the next page, there were 12 drops forming 24 linearly independent points and 48 measurement points in total. The topographic grid on the specimen was 23 × 23 for 529 points. Of course, this does not mean there are more "features" than could be detected by 12 drops. However, the presentation and visualization quality is greatly improved by having an excess of grid points. We have now constructed a "Z" surface on an X-Y grid.

The Z surface can be manipulated in two useful ways to further improve visualization. The first technique is to pseudo-color the surface according to the value of the Z-coordinate; e.g., darker at lower values and brighter at higher levels. The second option is to set

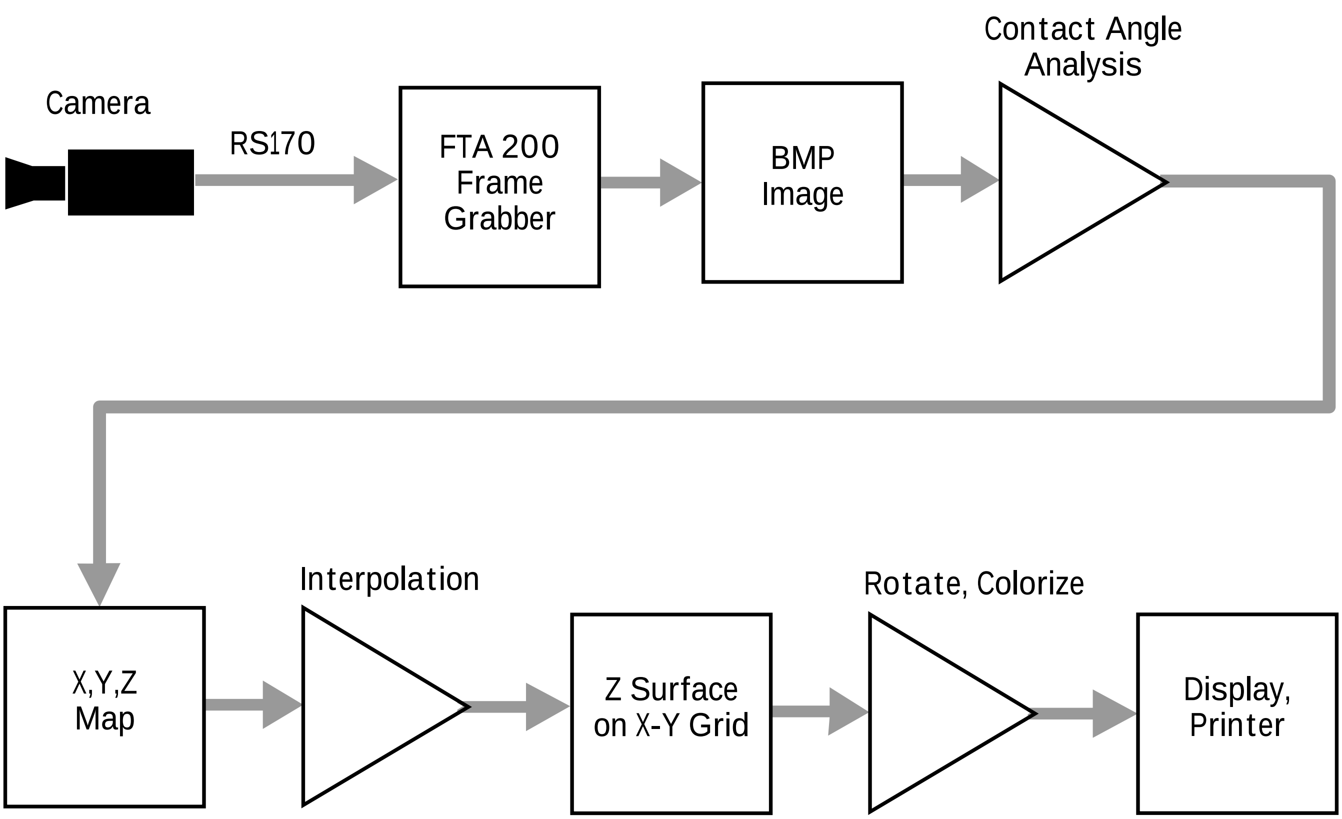

Figure 8. Data flow.

the viewing perspective of the surface. This involves rotation and elevation. The example below shows some of these operations.

Example

The sample was a printed circuit board (PCB). A photo of the part of the board that was analyzed is shown in Figure 9. The region had two gold-plated fingers, bare fiberglass board next to the fingers, and solder mask appears to the right behind the fingers in the photo. A portion of the board is cut away for the mating socket. The analysis occupied a 10 × 10 mm region in the center of this image. The white horizontal line indicates a distance of 10mm on the surface of the specimen.

Twelve drops were placed in the region; four in the solder mask and two rows of four drops each crossing from the bare board cutaway into the second gold finger. Additional data points were defined at the cutaway edge to clarify it in the topographic plots.

Figure 10 is a 2-D plot of the measurement locations. Each point in the X-Y-Z map described above shows as one "X" in this plot. Note the plot suppresses the actual Z data value. If you look carefully you can see characteristic diamond patterns of the left, right, front, and back measurement locations for each drop. You can see the measurement locations are reasonably spread over the surface, but certainly are not on any regular grid.

Figure 11 shows the topographic, or 3-D, plot from the standard perspective. With respect to Figure 9, the standard perspective is from the northwest, Figure 12 is from the west, and Figure 13 is from the northeast. The two gold fingers have higher contact angles (typically 105˚) than the area around them. The cutaway section is closest to the viewer, low and center. The solder mask has the lowest contact angle (typically 80˚). It is to the left and extends to the back corner. The region immediately before the first gold finger is bare board. It has a typical contact angle of 90˚, so is higher than the solder mask.

Figure 12 on the next page shows the 3-D plot rotated to the left so we can view the fingers from the edge more, almost as if we were the mating socket. Figure 13 on the next page is a substantial rotation to the right, so we are viewing from the interior of the board. We are looking out over the fingers toward the socket.

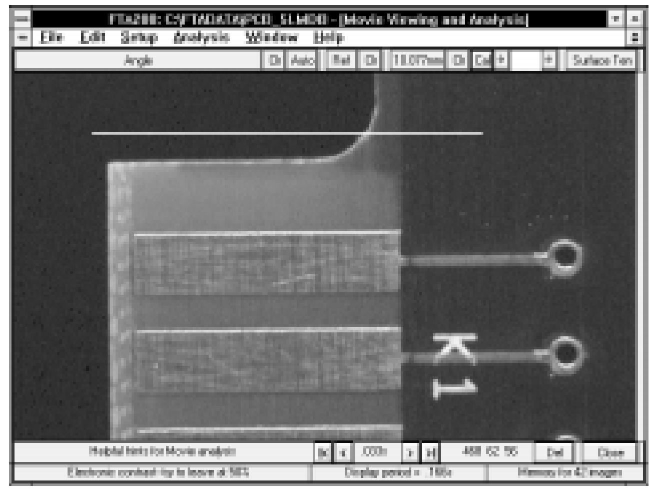

Figure 9. PCB specimen.

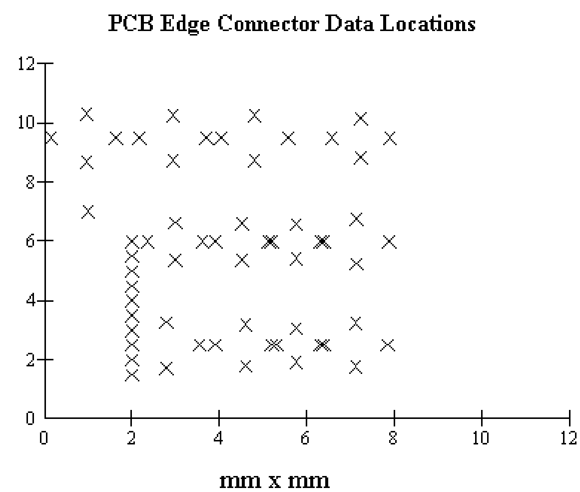

Figure 10. X-Y location map.

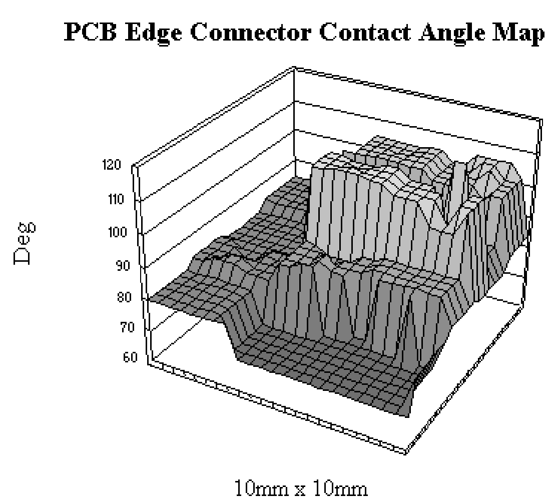

Figure 11. Original perspective.

Other Applications

Often one does not wish to visualize a surface so much as have good statistics on it. The robotics enable a large number of measurements to be taken with little operator effort. The system can be used on an absorbent material if its surface does not distort significantly from the absorption. Even if there is distortion and the drop's baseline cannot be determined automatically, the instrument can still profitably be used to gather a large number of images for subsequent analysis with operator assistance.

The initial design of the instrument was for an X-Y specimen stage, but this can easily be modified to "cylindrical" coordinates. In this case we move a web with one axis and move across the web, perpendicular to the web's motion, with the second axis.

In medical product applications, dispensing precise amounts of liquid is required. The instrument can be used in pilot labs to develop production processes because of the control available for the syringe pumps. The video camera can verify the dispensed amount and can also measure the contact angle of the dispensed fluid. Protocols are built in for washing the dispense tips in such applications.

Summary

This new system can take contact angle data over a surface with sufficient spatial resolution to form meaningful 3-D plots. Three-dimensional data presentation techniques then permit far better visualization of surface treatment problems than is possible with simple-point measurements.

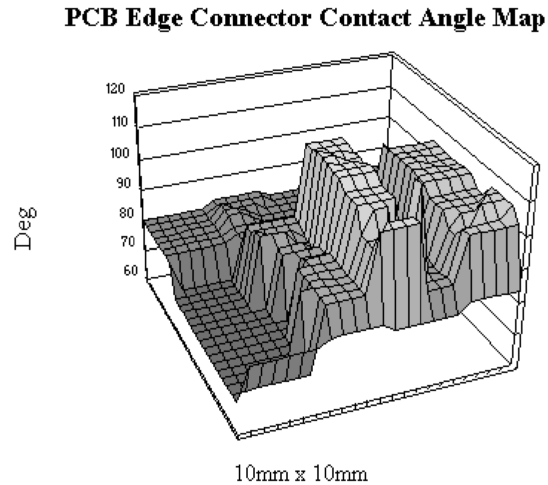

Figure 12. Rotated right slightly.

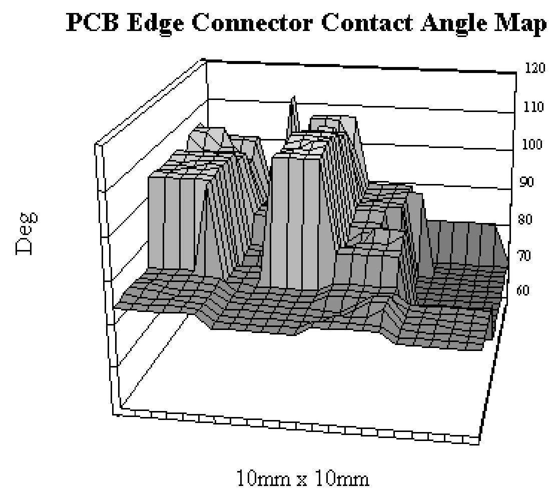

Figure 13. Rotated left substantially.