Diode Fabrication Laboratory

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Abstract

This paper entails the process that Ted Gould and Jill Sprague went through to create a p-n-diode in a non-clean room environment. This experiment took place at Rose-Hulman Institute of Technology from December to February of 1999-2000. The processes involves taking a slightly n-type wafer, growing an oxide layer, etching through that in a fixed area, and doping that area p-type. This eventually created a p-type well inside the n-type wafer. Metal contacts were then placed on the surface and the wafers were connected to a curve tracer to find their fitness.

Table of Contents

List of Figures

List of Tables

Introduction

In a classroom setting, it is really easy to talk about the n and p sides of a junction, and not really worry about how they got there. In lab it is a completely different story. In lab one has to worry about how the silicon wafer is grown originally, and how to get a very small area of that wafer exposed to the chemicals that transform it into something useful. In this lab, a lone silicon wafer is taken from a state of uselessness to be transformed into a diode. Along the way it must have a small p-well developed inside of it. It must also have contacts put in it, and be tested for its fitness. This lab starts with a story about six wafers (Al, Bob, Chuck, Dork, Eloise and Fred) and their journey to becoming diodes.

Section 1: Creating the Wafers

All of the wafers started out from the same 5" silicon wafer. The smaller wafers were broken from this larger wafer by scoring using a scribe. The scribe was run across the wafer until a reasonably sized piece broke off of the wafer. Six pieces were separated and labeled. Their approximate shapes and labels are located in Fig. 1. The pieces range from 2 - 3 cm in length and 0.5 - 1 cm in width. All curved edges (show by round curves) are due to the smaller wafer being on the edge of the 5" wafer. Those occur on both Chuck and Eloise.

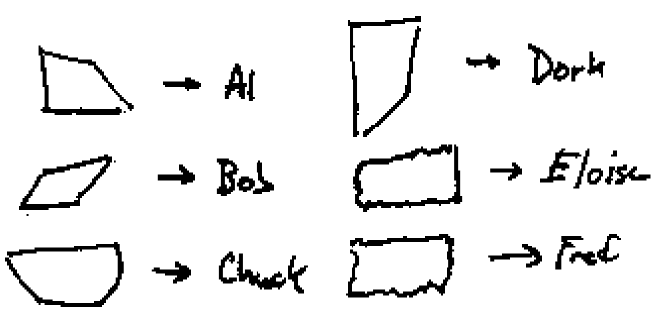

Figure 1 Sketches showing the original shapes of the diode wafers and their names.

Section 2: Growing the Oxide Layer

The next step is to apply an insulating layer to the top of the silicon wafer. This is was done by placing the wafer in an oxygen rich environment at 1000 K. This should cause the silicon atoms to combine with the water in the air, thus creating a silicon dioxide layer on top of the wafer. This reaction is described by the equation: $Si + O_2 \rightarrow SiO_2$. The wafers were put into the high temperature oven in sets of two by placing them on a quartz glass boat. The glass boat is then fed down the quartz tube into the oven. While the boat and wafers are in the oven, an oxygen supply causes an oxygen flow to move through the tube, over the wafers, and into the exhaust. To create the oxygen flow, an oxygen tank and boiling water were used. This creates an oxygen flow through the tube. When the boat was in the oven the heater on the water was kept at 28° C, to heat the water, the heater was set to 50° C. By looking at the

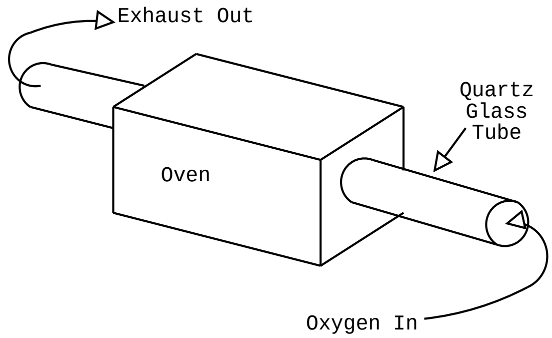

Figure 2 The high temperature oven with the oxygen flowing through the quartz tube

Table 1 Data taken from heating the wafers in the oven

Diode Set	Time in Oven	Color	Oxide Thickness
Al & Bob	20 min.	blue	1100 Å
Chuck & Dork	20 min.	black/brown	650 Å
Eloise & Fred	30 min.	blue/violet	1120 Å

color of the wafers as they were removed from the tube information on the thickness can be gained. This is not very accurate so, ellipsometer readings of the thickness of the oxide were also taken.

For two of the wafers additional data was taken. For each of the wafers a ‘map' was created across a 3.5 mm x 3.5 mm area. This map is the ellipsometer data for the thickness of the oxide in the area. The maps of the two wafers can be seen below:

Table 2 Data of the oxide thicknesses on Fred, taken with the ellipsometer

1117	1119	1123	1127	1124	1110	1116
1114	1132	1128	1123	1125	1115	1113
1115	1141	1161	1125	1111	1110	1111
1117	1122	1125	1121	1118	1110	1114
1116	1118	1122	1114	1115	1108	1112
1117	1122	1120	1117	1112	1114	1108
1118	1120	1123	1118	1113	1115	1106

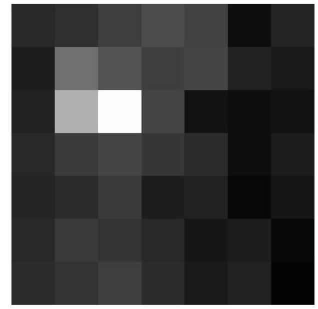

Figure 3 Graphical map of the ellipsometer readings for the oxide thickness on Fred (lighter color is thicker)

Table 3 Data of the oxide thicknesses on Chuck, taken with the ellipsometer

666	638	645	637	648	653	648
651	646	645	649	651	639	628
660	653	655	652	655	639	626
643	636	657	664	667	634	636
651	655	647	652	631	625	625
635	650	644	645	639	621	621
629	634	637	632	628	608	604

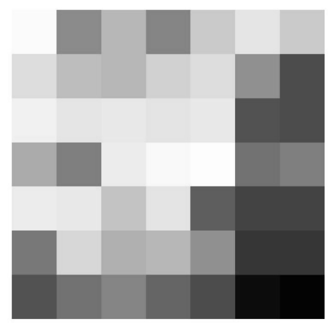

Figure 4 Graphical map of the ellipsometer readings for the oxide thickness on Chuck (lighter color is thicker)

Section 3: Applying the Photoresist

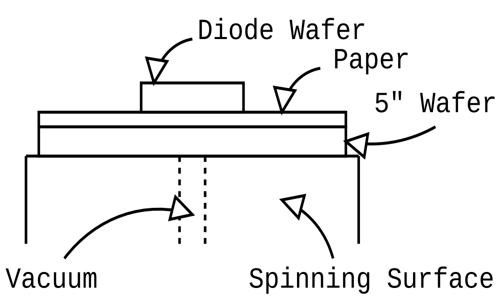

Figure 5 Spinning apardus with wafer an paper on top.

In order to designate a section of the wafer to be doped p-type, photoresist was applied to the wafers. This can later be removed in exact locations using UV light. To apply the photoresist to the it must be ‘spun' onto the wafers. This process involves placing the wafer on a spinning surface, placing a few drops of photoresist on the wafer, and the spinning the surface for a short amount of time. In all cases the wafers were spun for 25 seconds. To secure the wafers on the spinning platform a vacuum was used. This vacuum came up through the spinner, in a small hole on the surface. To balance the wafers, and for ease of clean-up, the wafers were taped to a piece of paper, which was attached to a 5" silicon wafer. The locations of the tape on the smaller wafers are noted in Fig. 6. The times and number of drops on the individual wafers are shown in the

Table 4 Data for the applying of photoresist to the wafers the first time

Wafer	Drops of Photoresist	Rotation Speed	Rotation Time
Al	2 cc	1.7 krpm	25 sec
Bob	2 cc	2 krpm	40 sec
Chuck	2 cc	1.7 krpm	25 sec
Dork	2 cc	1.7 krpm	25 sec
Eloise	2 cc	1.7 krpm	25 sec

Table 4. Unfortunately all of the wafers did not expose properly with the first spin and development cycle. Problems with that are noted in the next section. When the wafers were respun on successive applications of the photoresist the tape was placed in the same locations, and a standard set of parameters were used: 25 sec, 2070 rpms, and 2 drops. The only wafer to make it through the lab with it's initial spin (i.e. the data from Table 4) was Fred.

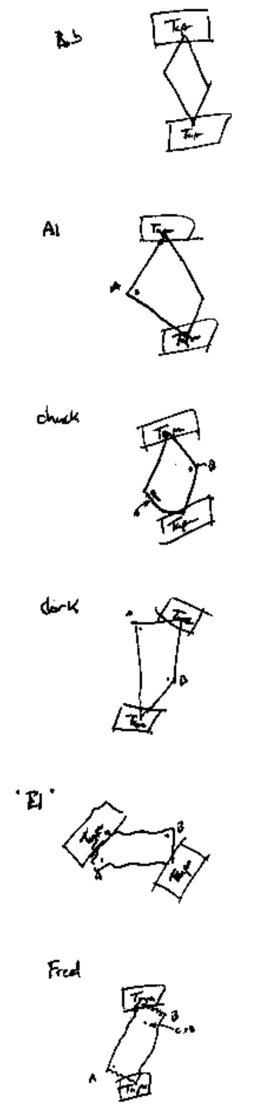

Figure 6 Placement of the tape on the wafers when spinning on the photoresist. Locations of build up are noted with dots on the wafer.

Section 4: Masking and developing the Photoresist

The reason for putting the photoresist on in the first place, was so that it could be removed in specific areas to allow us to make a p-well in the n-type wafer. To this end a ultraviolet light was used with a small mask made on an overhead. Our mask is in Fig 7. After printing the mask on a laser printer it was transferred to the overhead slide using a photocopier. This process created an interesting effect on the wafers in the end. When the mask was created it had small ‘pixels' on the edge of the region removed by the ultraviolet light. This can be seen partially in Fig. 8 where a small section of the mask has been expanded in size to show the pixels that are on the edges of the mask. This was enhanced through the printing and copying process.

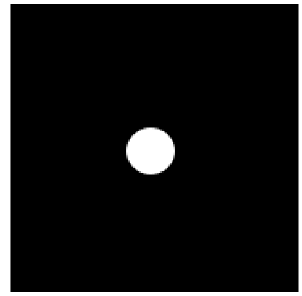

Figure 7 Mask used to remove photoresist

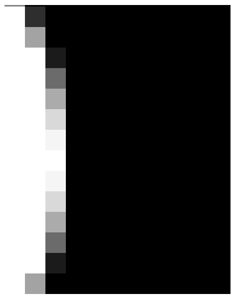

Figure 8 Zoomed in region of the mask from the ultraviolet light

The next step is to remove the photoresist that doesn't quite come off under the photoresist. To do this the wafers are placed in developer. The developer chemically reacts with the photoresist, decomposing it, getting further in the region which was already weakened by the ultraviolet light. In the first run through the exposure/developer process only one wafer came out with a recognizable ‘hole' in the photoresist. That wafer was Fred. The times for developing and exposing are in Table 5. The comments come from first reactions of the wafers, and later it

Table 5 Exposure and Development times for wafers on first run through process

Wafer Name	Exposure Time	Develop Time	Comment
Al	120 sec	60 sec +	too long
Bob	120 sec	20 sec	
Chuck	120 sec	10 sec	too long
Dork	120 sec	~5 or less	looks good
Eloise	120 sec	~5 sec	
Fred	120 sec	~4 sec	looks best

was decided that only Fred was good enough to continue on. All of the other wafers were cleaned off with developer and retried. It was discovered at this point that the developer concentration should have been diluted before being used in the lab. The correct concentration was a 4:1 mixture with water. The wafers were then respun, exposed and

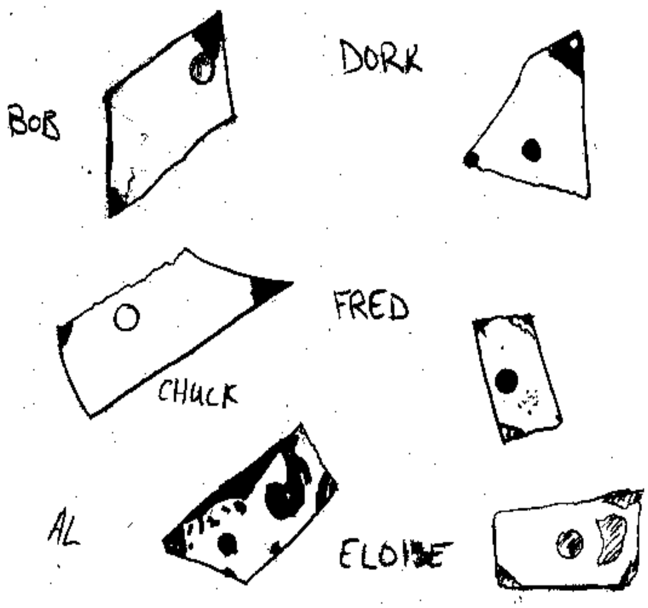

Figure 9 Sketches of the physical appearance of wafers after developing. Dark areas are where the oxide layer can be seen.

developed using a more standardized process. The spinning is noted in the previous section; they were exposed for two minutes; and they were developed until the person developing them could visibly see the whole in the photoresist. This developing time typically was around 45 seconds. After making these changes a good set of wafers was created. They were sketched as to note where the holes are, and what other areas of the wafer may have had enough of the photoresist removed to have etching occur there also.

Pictures of the Fred were also taken using a high powered microscope with a video camera attached to it. The camera was connected through a video capture card to a computer in the lab. This card was used to get the pictures shown in Appendix I. In those pictures the region that is covered in photoresist is visible, along with the area that has been eroded away using the ultraviolet light. Most of the pictures are of that region. Also many pictures so the photoresist region around the hole region which has been eaten way by the developer solution.

Section 5: Removing the masked oxide layer (Etching)

The next step is to remove the oxide layer in the region where the photoresist has been decayed by the ultraviolet light. To remove the oxide layer in this region hydrofluoric acid was used. Special precautions have to be used when using this acid. The person who was coming in closest contact with the acid covered themselves with a bib, lab coat, latex gloves, thick plastic gloves, and lab goggles. When the wafers are placed into the

$$4HF_{(aq)} + SiO_2 \rightarrow 2H_2O + 4F^{-}_{(aq)} + Si^{4+}_{(aq)}$$

hydrofluoric acid the reaction in the equation above takes place. Each of the wafers was put in the hydrofluoric acid for sixty seconds. After that it was rinsed in a water bath and left to dry in the hood. Pictures of the wafers were taken using the same microscope as in section 2, those pictures are in Appendix II. The edge that can be seen is the difference between the photoresist still on the wafer, and the bare silicon that has been exposed. Also, on the edges of the holes there is the 'pixelated' region that was discussed in section 2.

Section 6: Doping the well

To create the p-region in the n-type wafer boron was put on the outside of the wafer and baked in. To do this a boron paste was created using boric acid and water. The paste was make by placing a small spoon full of boric acid powder on a piece of glass, and then mixed with water until a paste was formed. The paste was then put on the hole region of the individual wafers. To cause the boron to move into the silicon structure further the wafer was placed in the same oven outlined in section 2. This time the wafers were put in the oven for 15 minutes at 1000^o C. When the wafers were removed from the oven they were covered in a brown shiny substance, borosilicate glass. The entire surface was not covered, mostly just the hole region and concentric circles around that region where the boron paste had spread from the original glob.

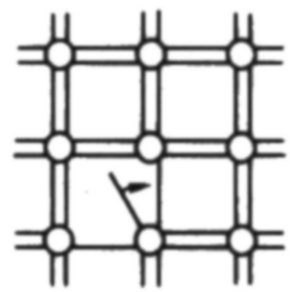

Figure 10 Diagram of the bonding structure of the silicon after the boron has been implanted.

Section 7: Removing the Borosilicate glass and oxide

To return the wafer to a usable state the borosilicate glass had to be removed from the surface. The protective oxide layer on the rest of the diode could be removed. To get to the oxide the photoresist was removed using acetone. The acetone was sprayed onto the wafer causing the photoresist to come off as the acetone evaporated. Then the wafers were again dipped in the hydrofluoric acid. The same procedure was that was used in section 5 was used here, except that the time was based on the appearance of the wafers. This caused the wafers to have the different times in the hydrofluoric acid as noted in table 6. At this point the wafers should theoretically be bare, but there are still artifacts left over from different stages of the process. Pictures of the wafers can be seen in Appendix III after the etching. In those pictures it can be seen how there are many discontinuities on the surface of the wafer. These discontinuities all have a potential to cause problems in the placement of contacts on the surface of the wafer.

Table 6 Table of the etching times to remove the borosilicate glass. Chuck was dropped in the hydrofluoric acid and the time was the length of time he was in the acid.

Wafer Name	Etching Time
Al	140 sec
Bob	105 sec
Chuck	215 sec
Eloise	140 sec
Fred	140 sec

Section 8: Applying the contacts

To place the contacts on the wafer a silver paste was used. The paste itself is a propriety formula purchased by Rose-Hulman. It consists of a two-part apoxy that must be mixed together in the right proportion to stick wires onto the semiconductor surface. To do this the wires were prepared first. To connect to the wafers the wire needed to be light enough that the apoxy could push the wire onto the surface of the wafer. To this end magnetic coil wire was used. The wafers were placed on a microscope slide and taped down to secure them. There were small areas left clear around the hole, and another section of the wafer to make the contacts. On Fred and Eloise there were no clear areas except for the hole. On these two wafers the second contact (to the n-type surface) was placed on the back of the wafer. To do this the wire was first attached to the microscope slide and then the wafer was attached on top of it. The silver paste was then placed on all of the surfaces with the wires next to them to attach the wires to the surface of the wafers. The silver past was allowed to dry in the lab over the weekend. The wafers on the slides were placed in compact disc cases to protect them. Larger gage wire was soldered to the magnet wire to make contacts outside of the case. The green wire was always attached to the p-type side of the junction.

Section 9: Testing the diode

To test the diodes they were taken to the circuits laboratory where a curve tracer was attached to the ends of the diodes. The results from the curve tracer are shown in Appendix IV. The reverse biases of the diodes were checked, but the results were not printed. On the reverse bias there was almost no current on the diodes that were checked. The graphs in Appendix IV show that all of our wafers had at least some curve in their I-V characteristic. The best exponential curves were in Fred and Eloise where the curve was quickly getting to an exponential state. Chuck's curve was also nice as it stayed the flattest for the longest amount of time.

Conclusion

For the total lack of a cleanroom, and the several unknowns in the process, the yield wasn't really that bad. There was only one wafer that was lost throughout the process, or before they made it to the testing phase. The I-V characteristics that the final results presented were less than ideal, but several of the wafers did show that a diode had been created. A lot of the problems come from the doping process where several impurities were not removed from the hole regions or the rest of the surface. This can be seen throughout the pictures in the appendices. These different artifacts caused the connections to be less that pure in the final stages of the production process. While the yields were less than ideal, much more can not be expected given the conditions of the production of the diodes.

Appendix I: Pictures of wafers after doping

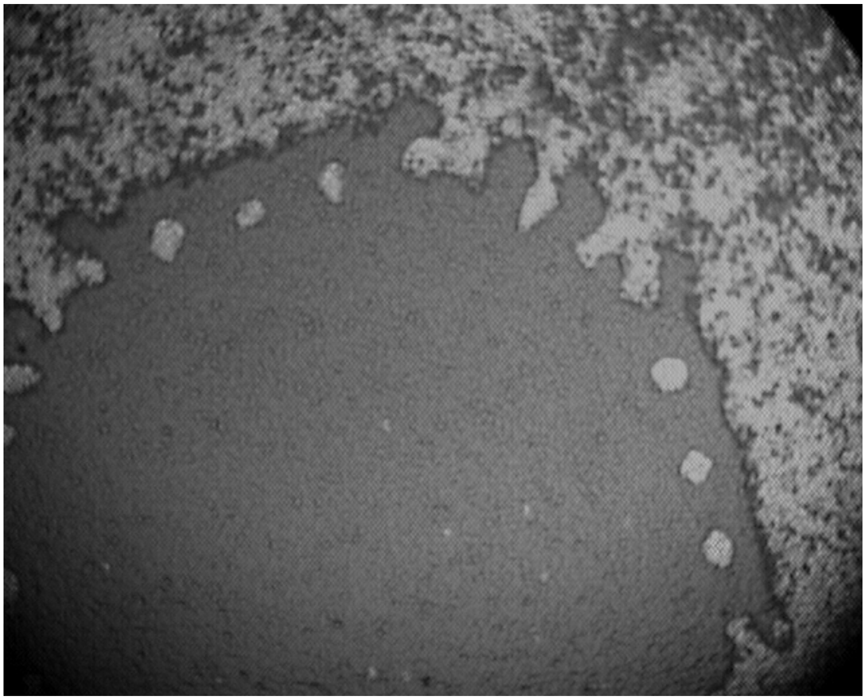

Figure 11 In this picture one can see a section of the hole that was removed in the photoresist on Fred. Notice the pixels that occur on the right side of the hole.

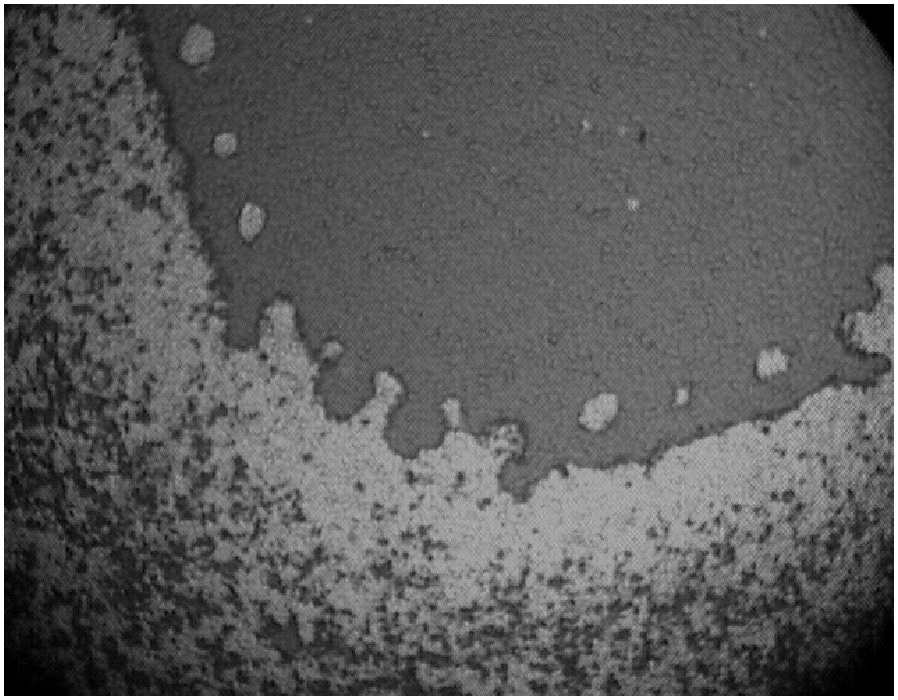

Figure 12 This is a picture on the other side of the hole created in the photoresist on Fred.

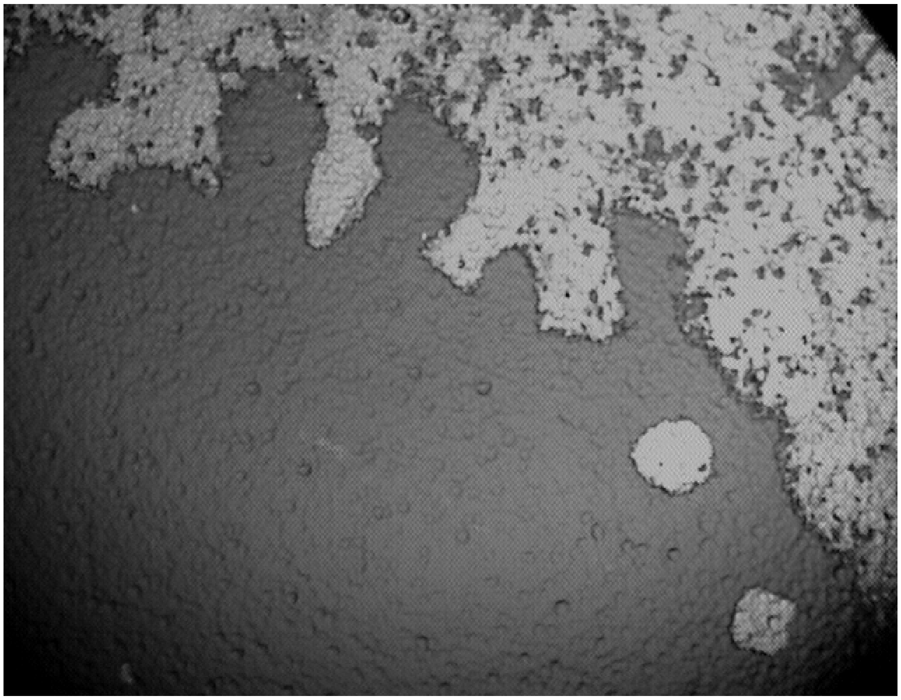

Figure 13 Here a stronger lens was used on the microscope to see more detail on the hole on Fred.

Figure 14 This picture is taken with even more magnification. Notice the divots that occur in all of the surfaces.

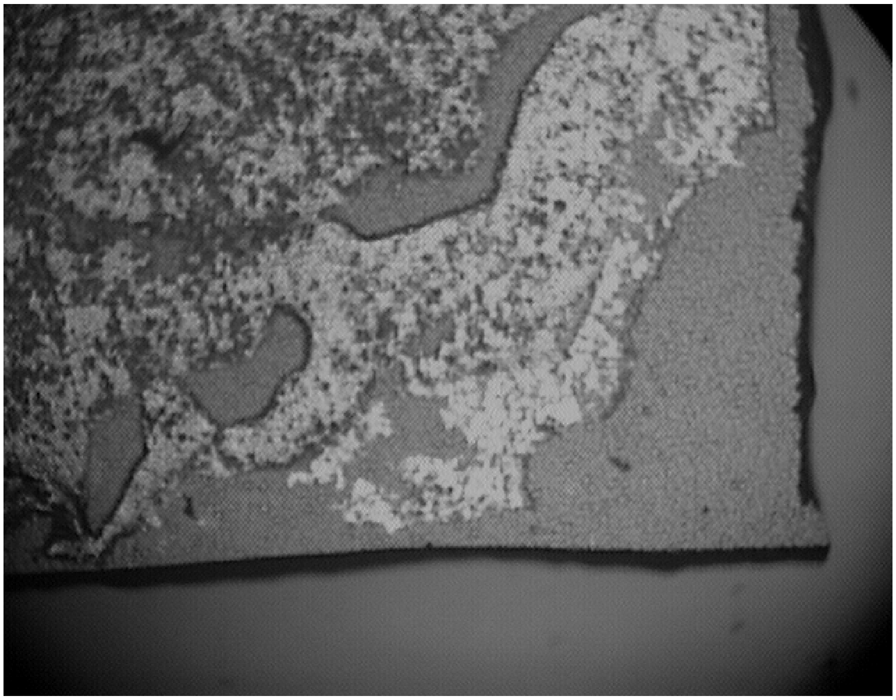

Figure 15 Pictures were also taken on the edge of Fred. Here the photoresist must have been thinner, causing it be removed in the developing process also.

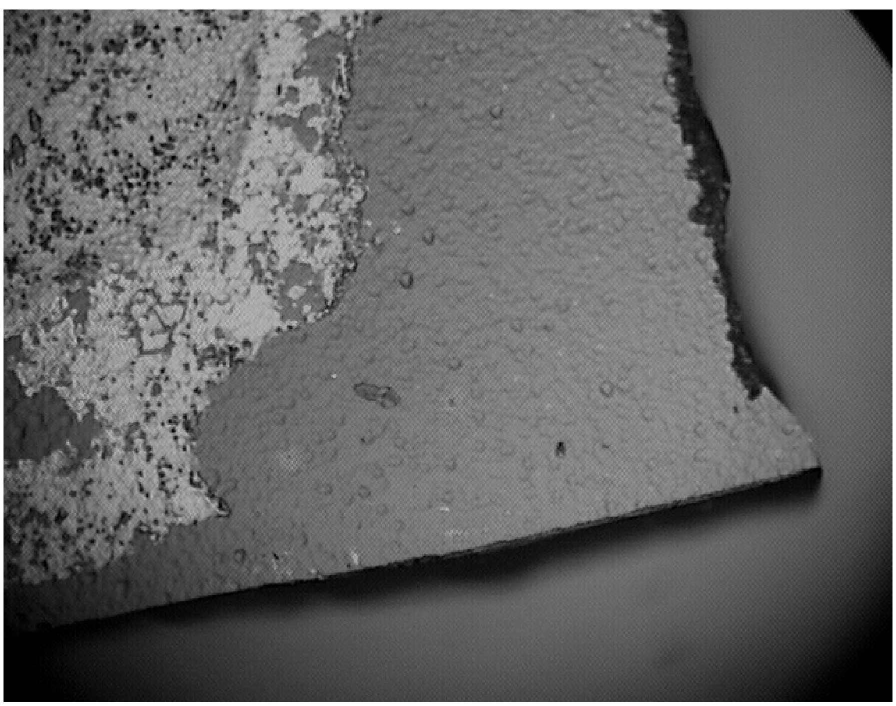

Figure 16 This picture is of the same edge, with more magnification.

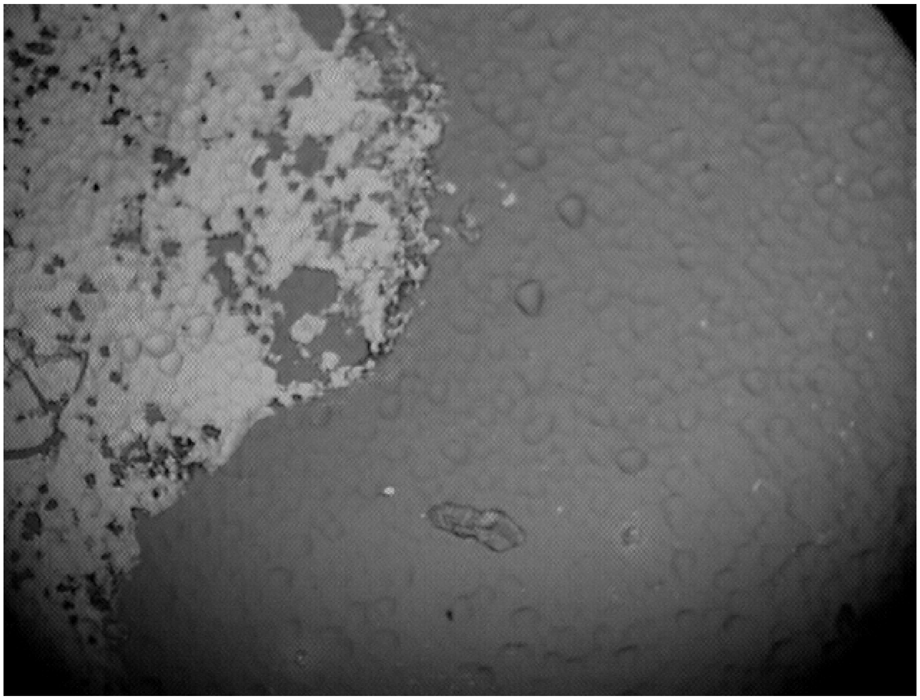

Figure 17 Here is the same corner of Fred taken with the most magnification. This picture shows the edge between the photoresist and the oxide layer.

Appendix II: Pictures of wafers after etching

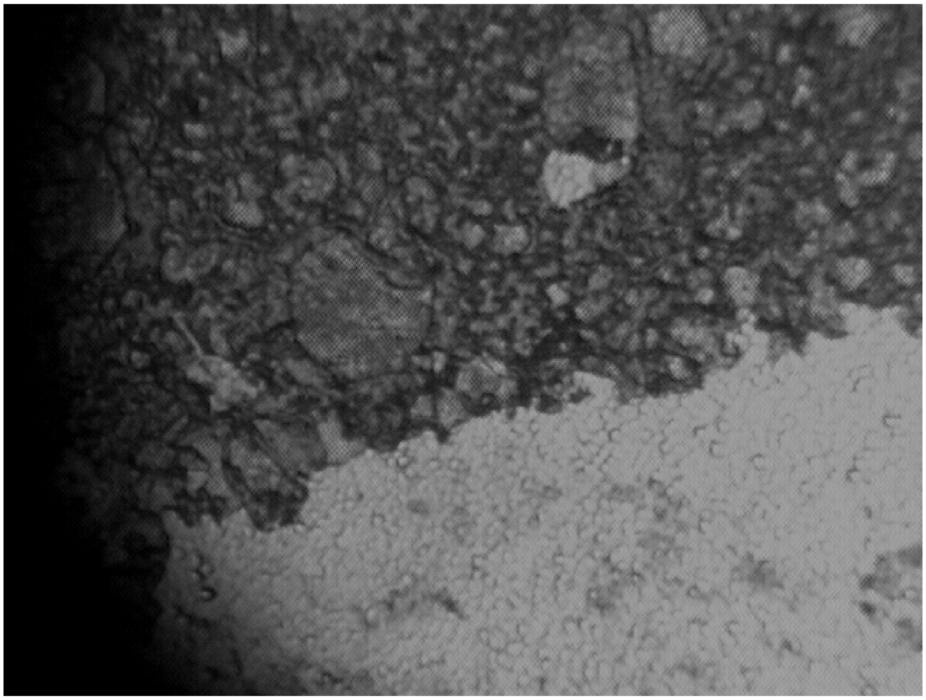

Figure 18 Here is a picture of Al after etching. There is still some photoresist in the hole.

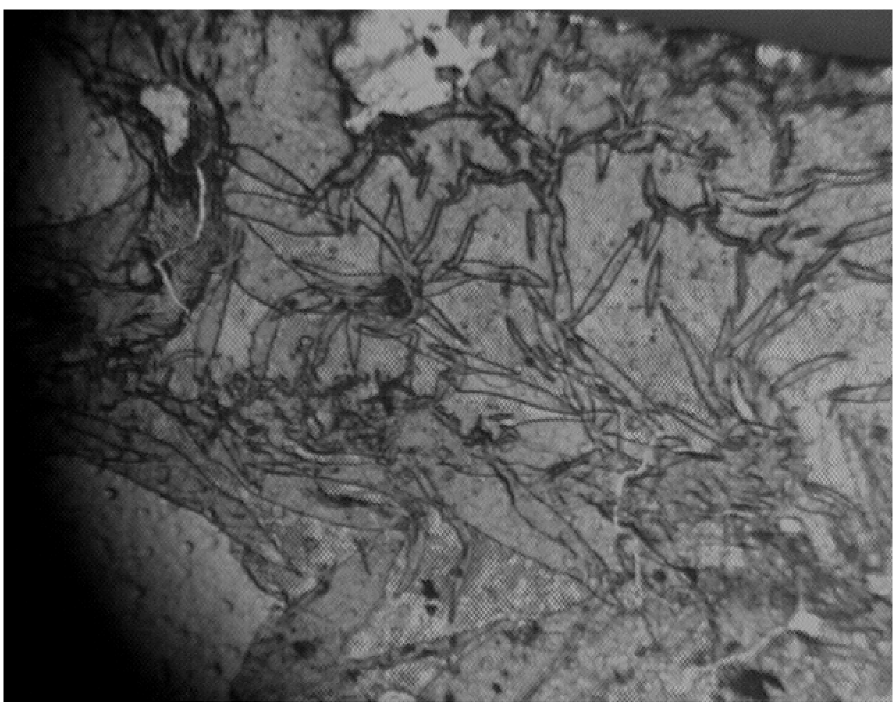

Figure 19 This picture is entitled 'funky stuff'. While looking around on the microscope this area was found. It is not know what this is, but it seems to be some artifact of the etching process.

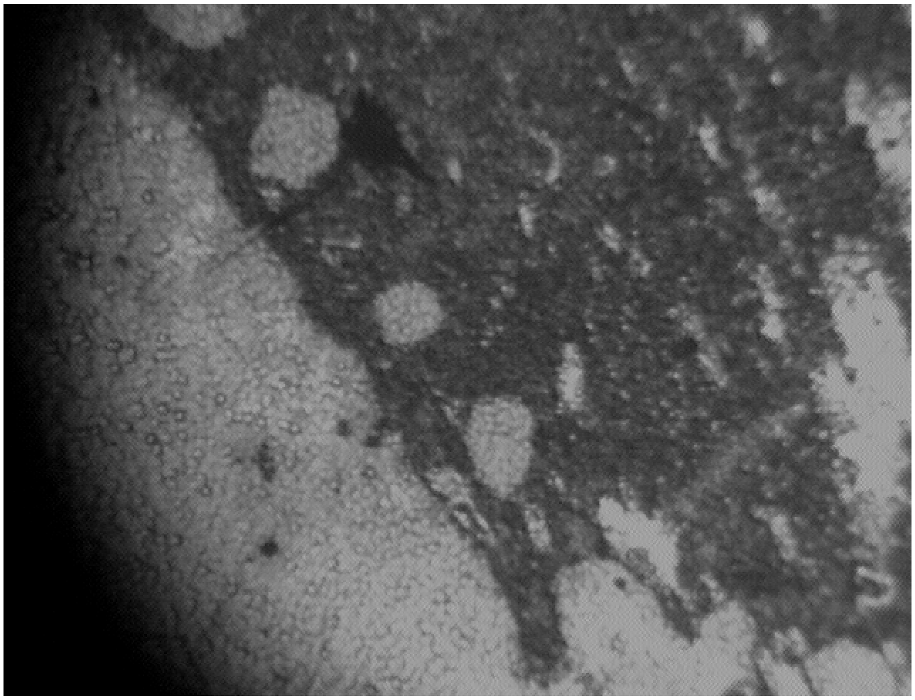

Figure 20 This is the hole region on Bob. Most of the photoresist and silicon dioxide is eaten away. There is still some photoresist in the region on the right side.

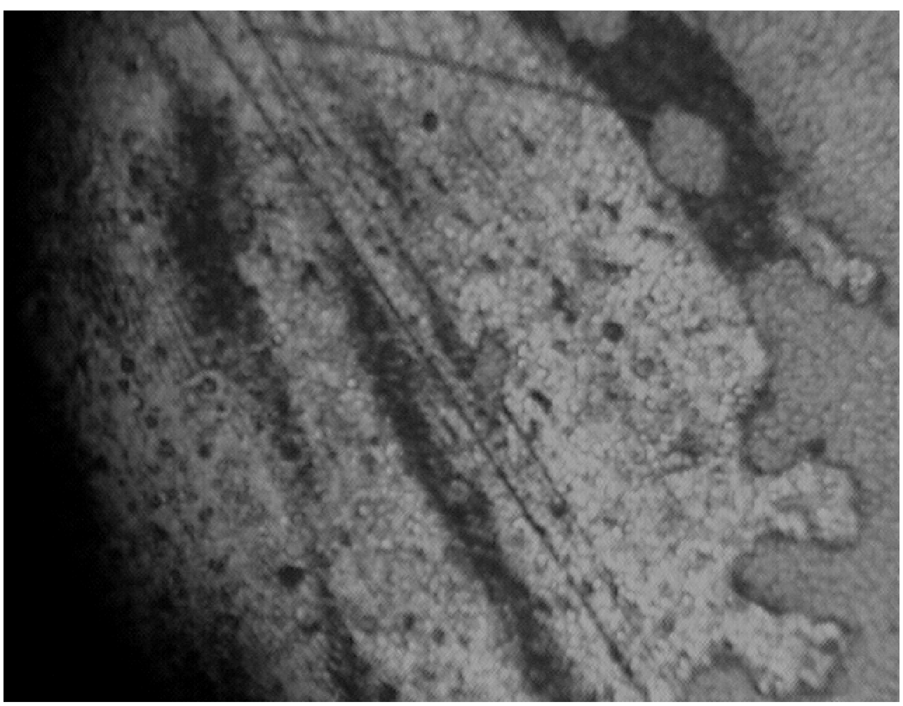

Figure 21 Here is a section of the hole in Bob. If you look at Fig. 9 you can see that part of the hole is shaded over with photoresist, this is that area.

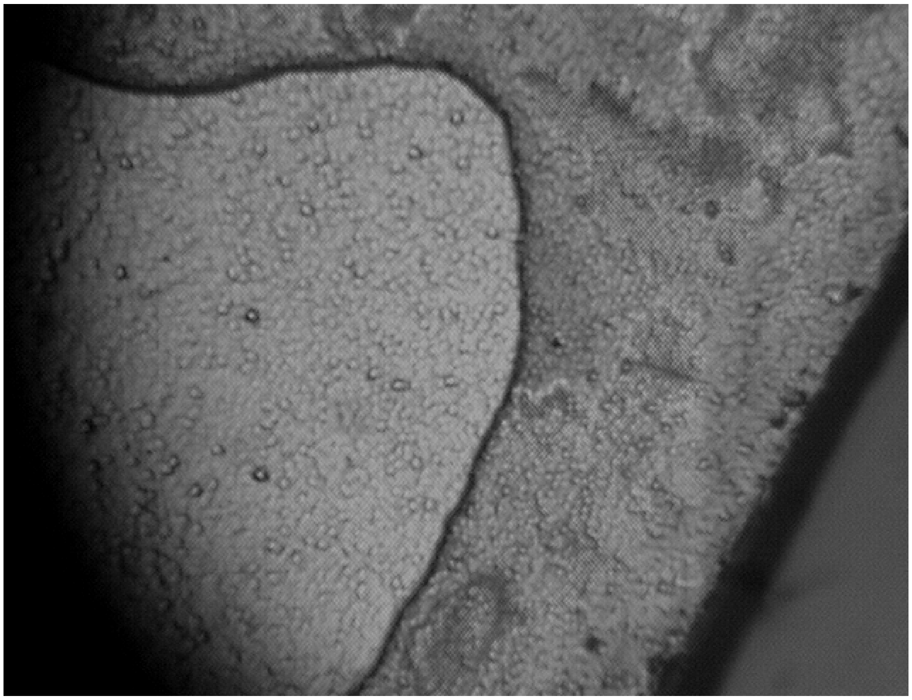

Figure 22 In this photo one can see the edge of the wafer. That is the dark line on the right hand side. This is a corner where the silicon dioxide was exposed.

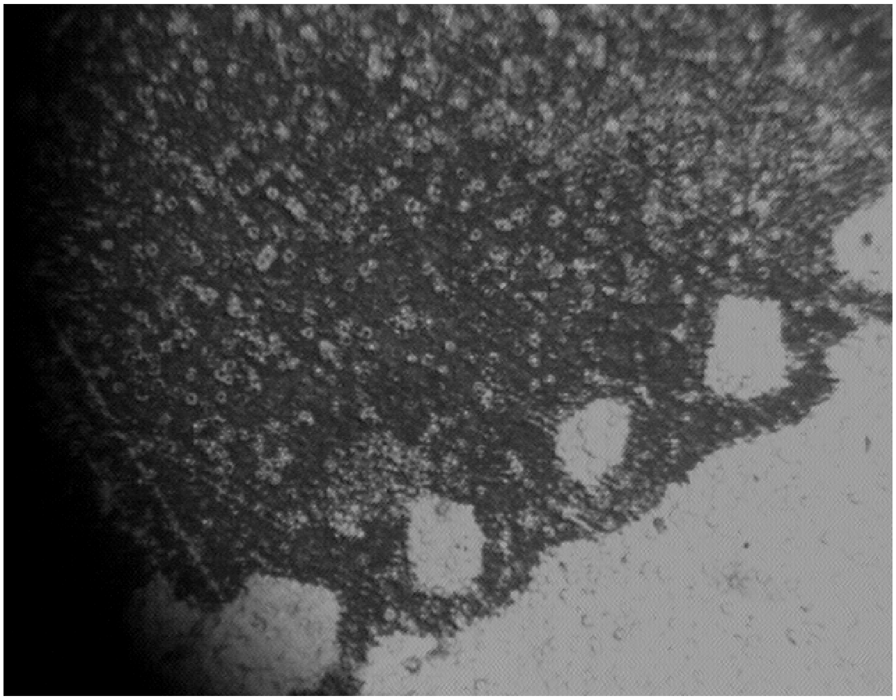

Figure 23 Here is a photo of the hole on Chuck. In the naked eye look at chuck it didn't look it he had a distinguished hole, but in this photo it looks like everything turned out okay.

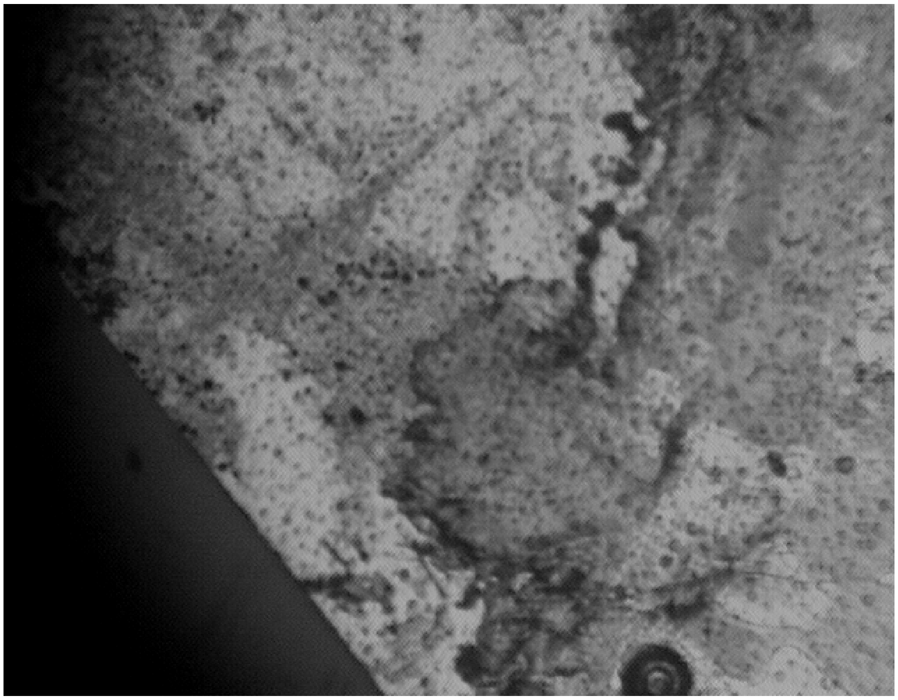

Figure 24 Here is a picture of the upper edge of Chuck. This area looks like it hasn't been etched, it might have been under the tongs in the hydrofluoric acid.

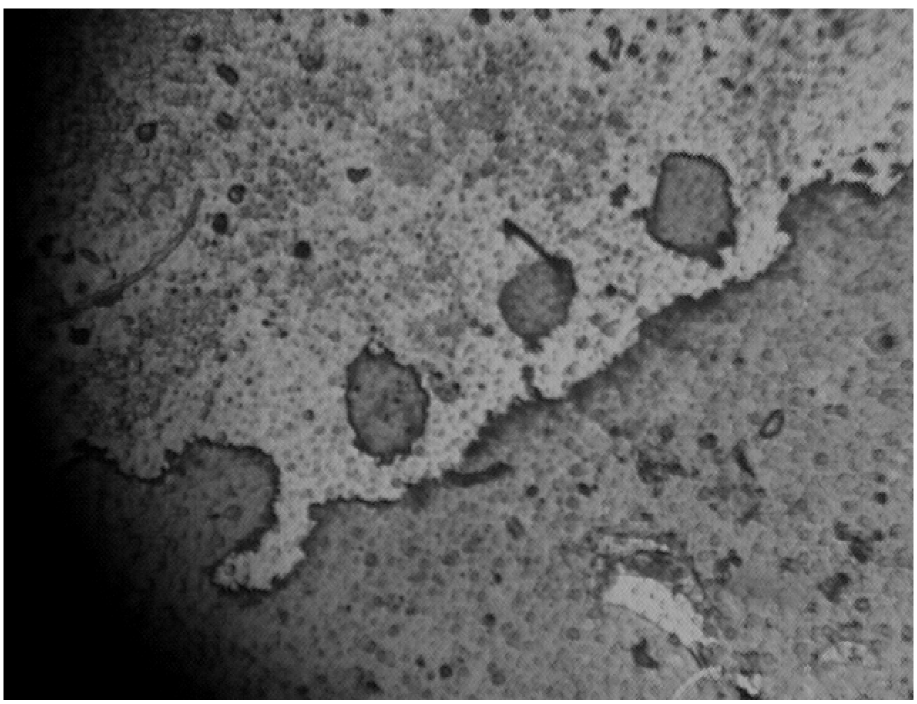

Figure 25 This photo looks at the hole region on Dork. Here the pixelated area can be seen clearly.

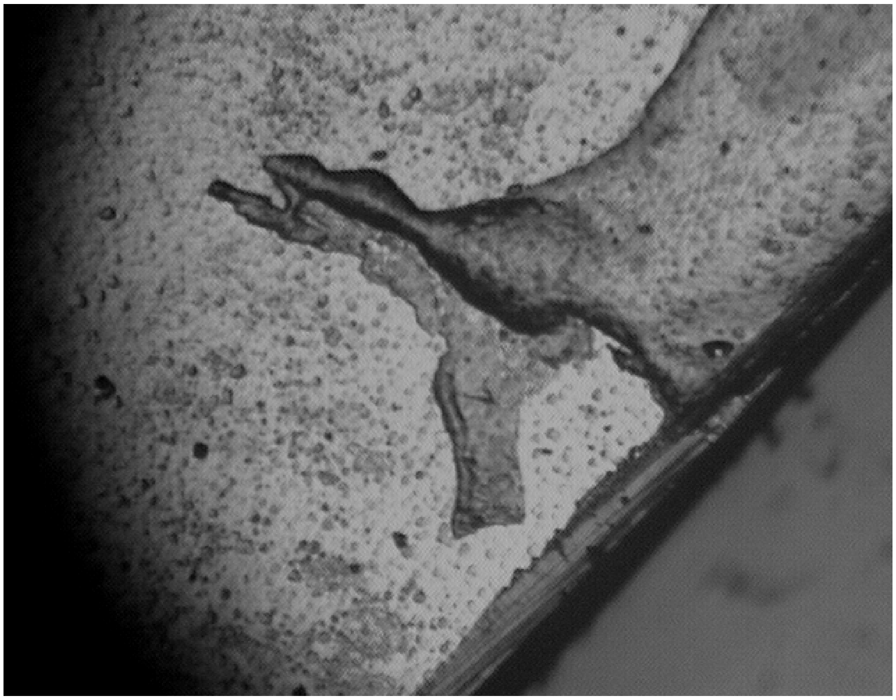

Figure 26 Also on Dork, this photo shows a corner of the wafer. The pattern that developed is interesting.

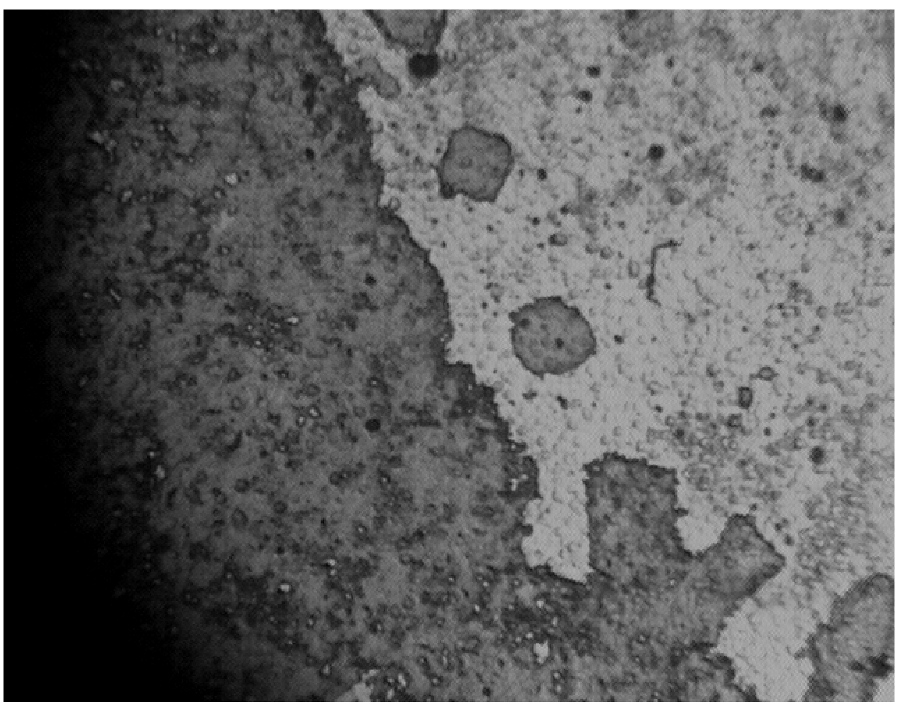

Figure 27 This photo is of the hole region on Eloise. A curiosity is the color inversion in the picture.

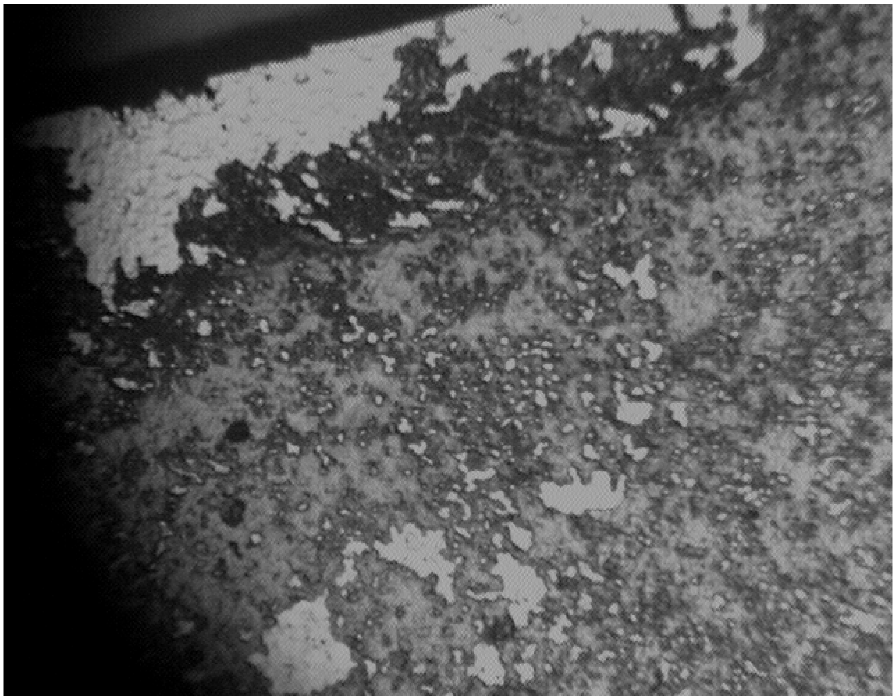

Figure 28 Here is a photo of the edge section of Eloise.

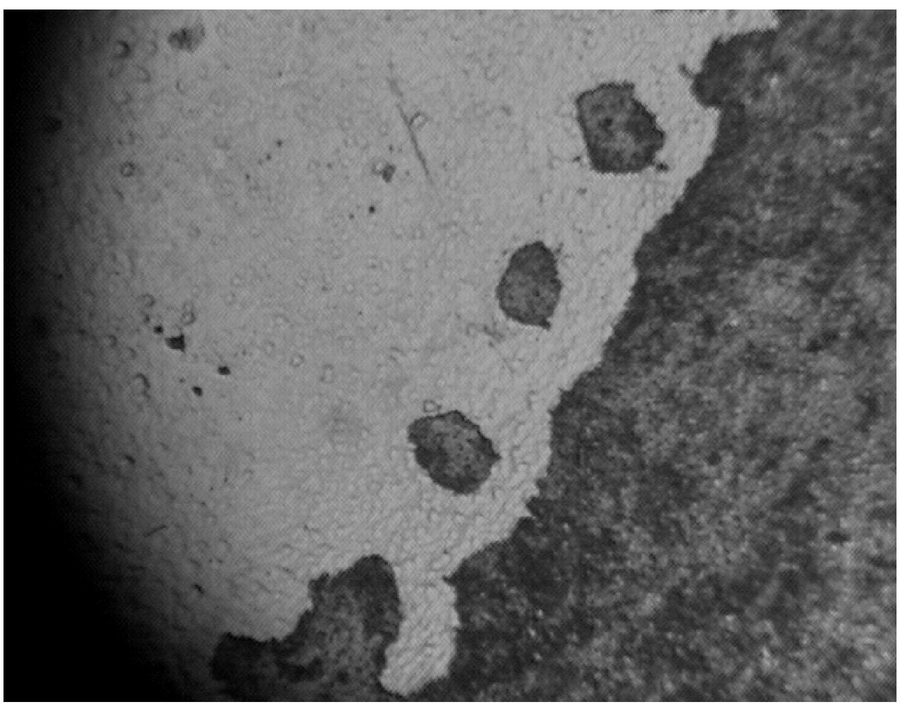

Figure 29 This is a photo of the hole section of Fred. Notice the very distinct line between the photoresist and the silicon. Also the pixels can be seen very well.

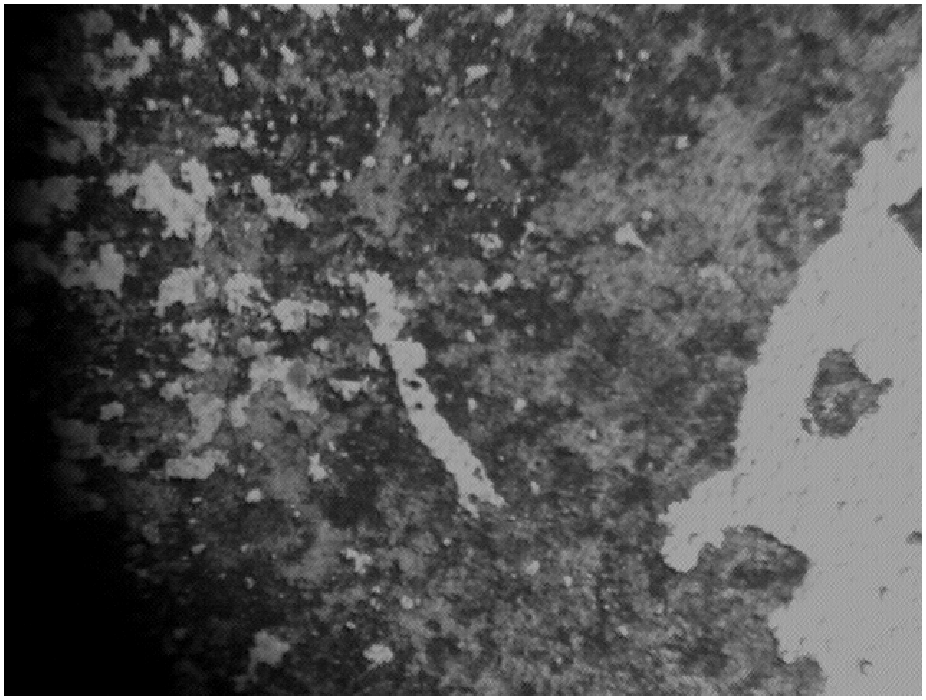

Figure 30 Here is a corner of Fred. This isn't nearly as pretty as the hole region was.

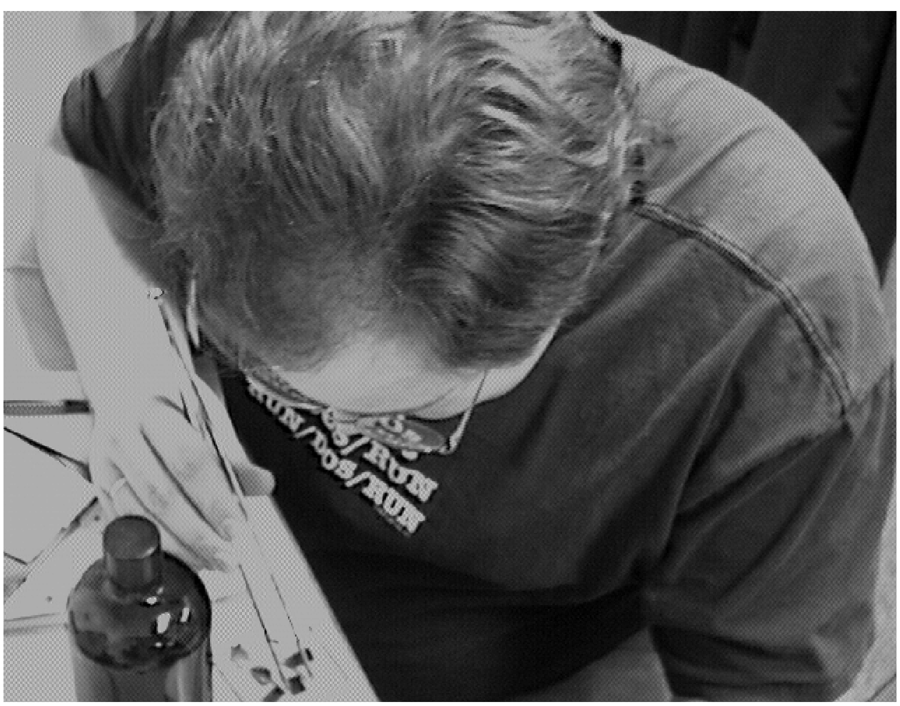

Figure 31 Here is Ted after etching. Notice the removal of the fuzzy stuff on the top of his head ☺

Appendix III: Pictures of wafers after second etching

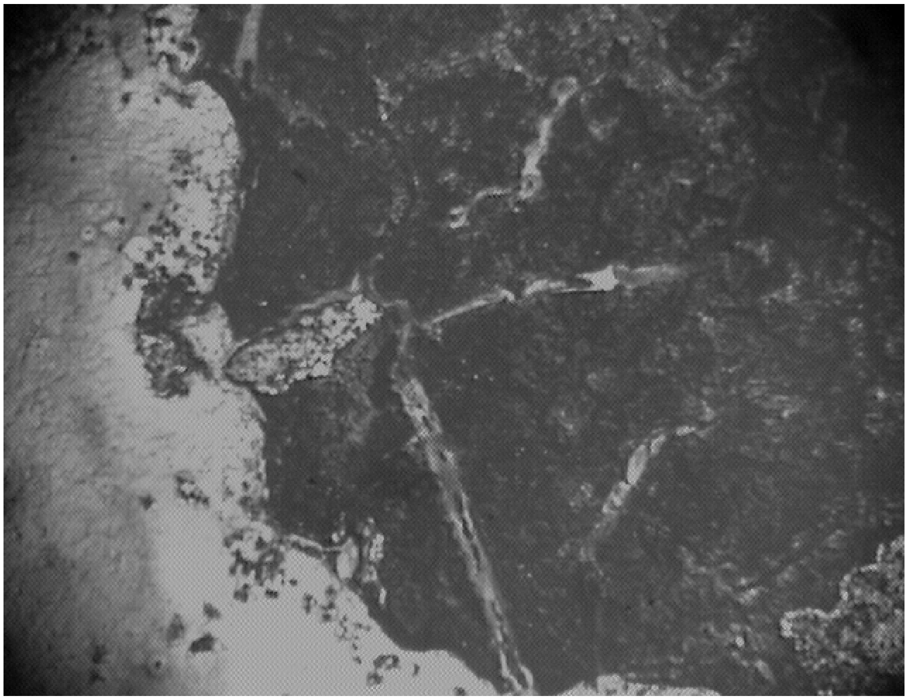

Figure 32 This photo is of the hole region on Al after the borosilicate glass has been etched off.

Figure 33 This is the same region shown in the above picture at a greater magnification.

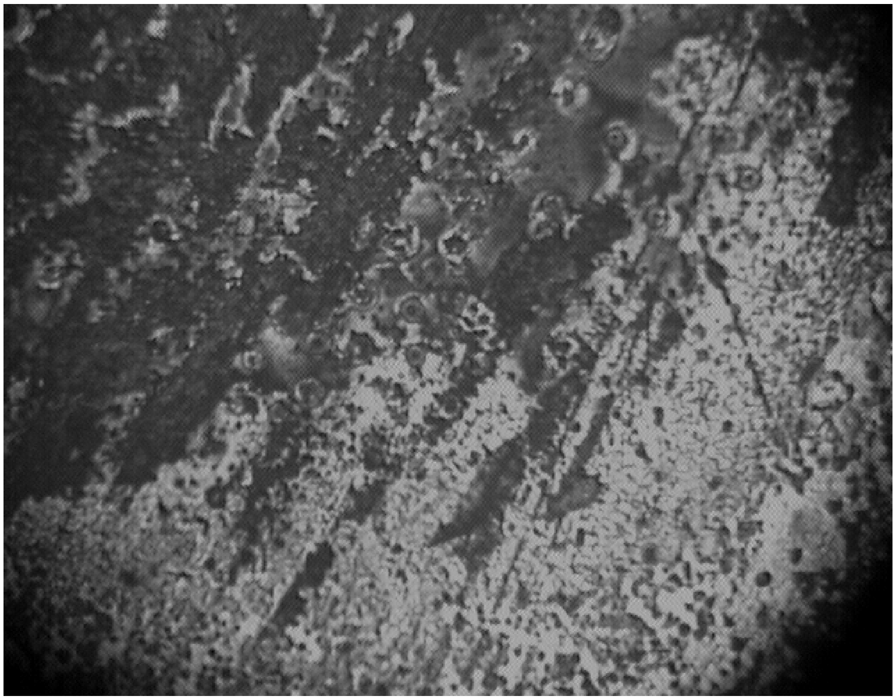

Figure 34 This is a photo of what was believed to be the hole region of Bob. The hole region was not evident at any place under magnification, this seemed the place with the largest exposed region.

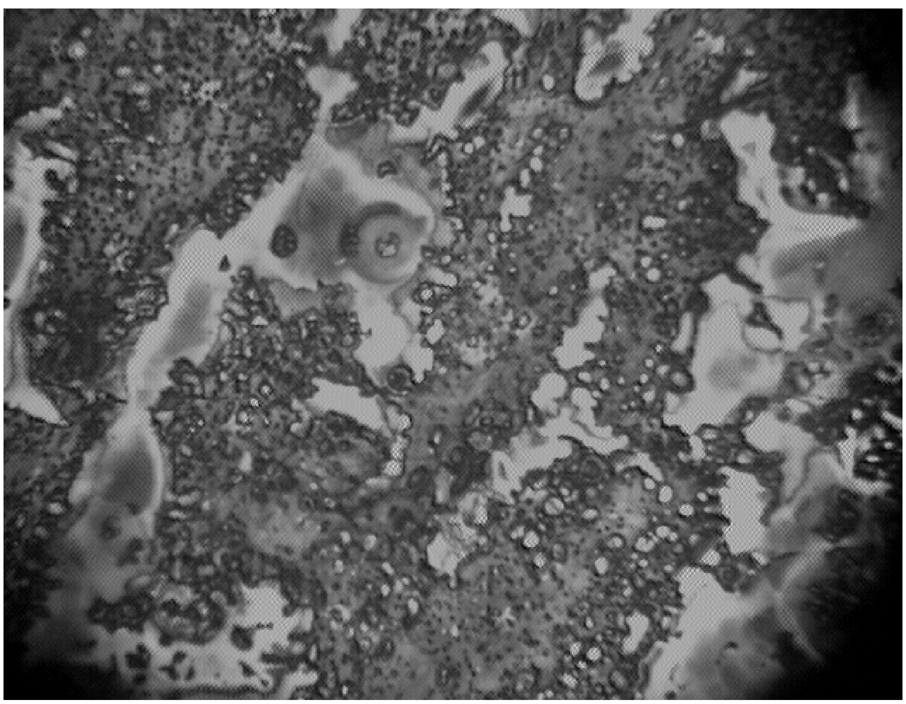

Figure 35 Here is a magnified version of the picture above. This region is not nearly as clean as the other photos.

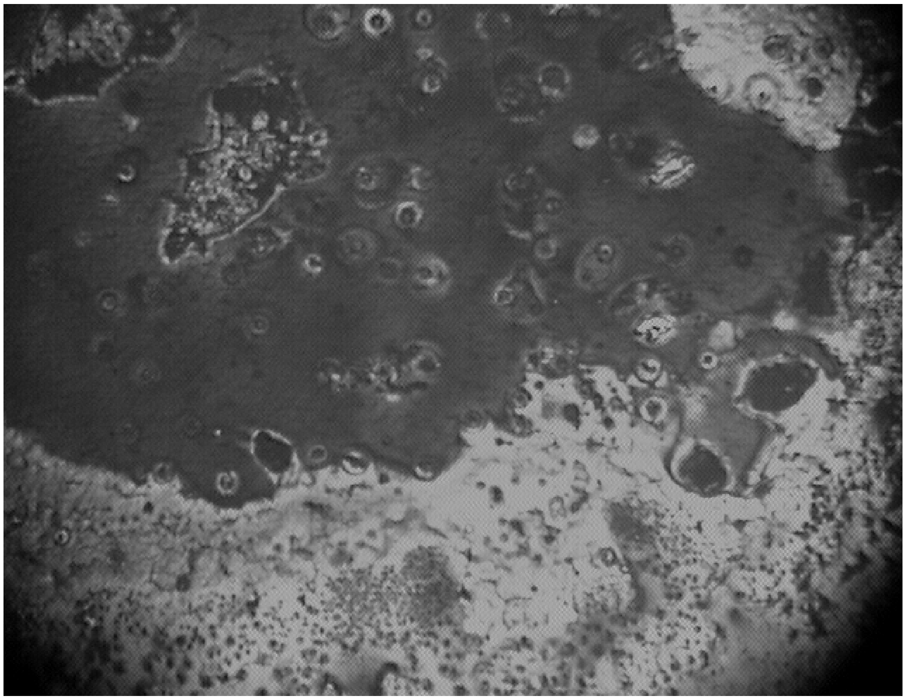

Figure 36 This is the hole region on Chuck. On the top is the doped region and the bottom is the silicon region.

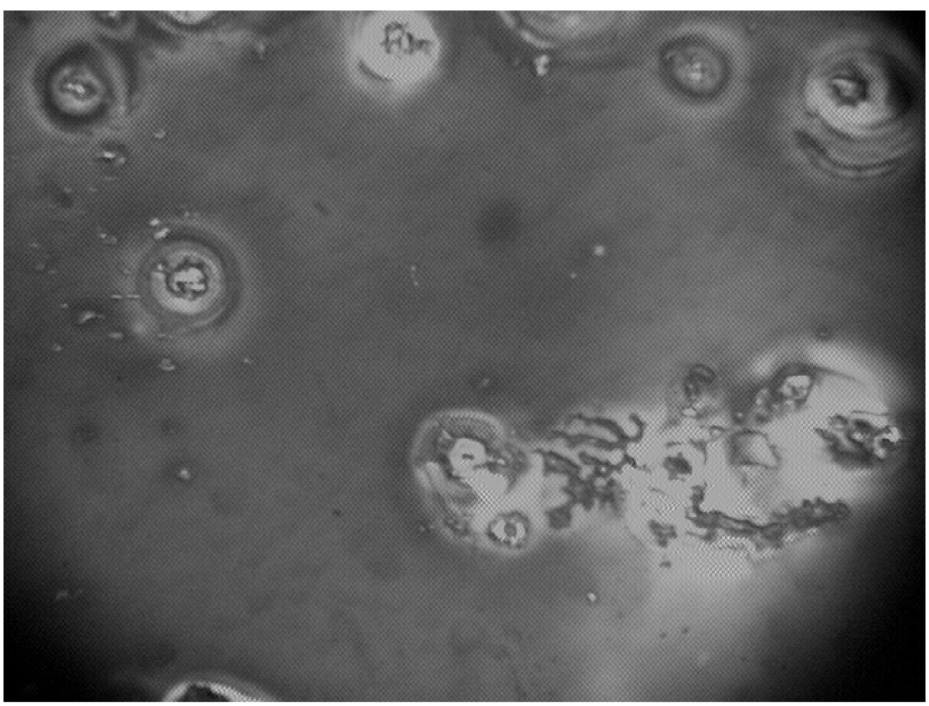

Figure 37 Here is a magnification of the previous image. This shows the small areas where there is not a clean well clearly.

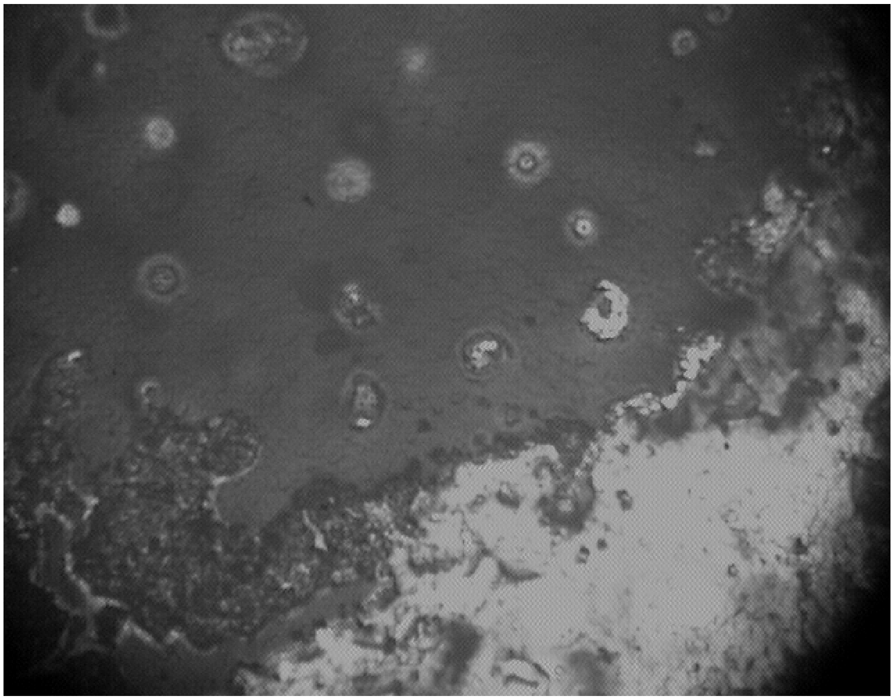

Figure 38 This is a picture of the hole region in Eloise. This is a much more distinct picture and you can see the doped and undoped regions.

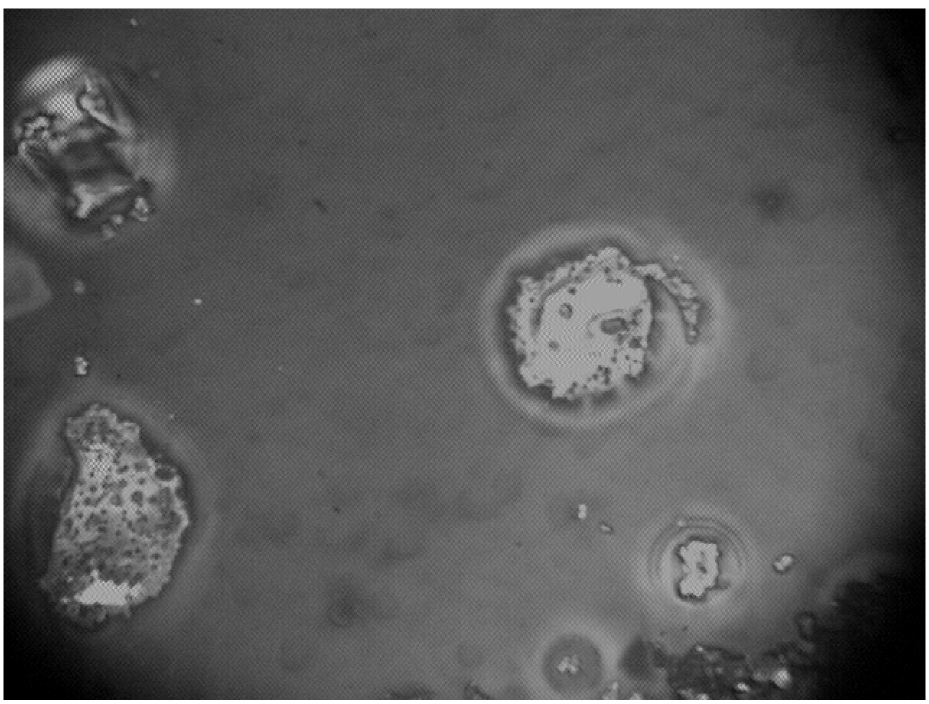

Figure 39 This is a magnification of the previous image. This shows a smooth doped region.

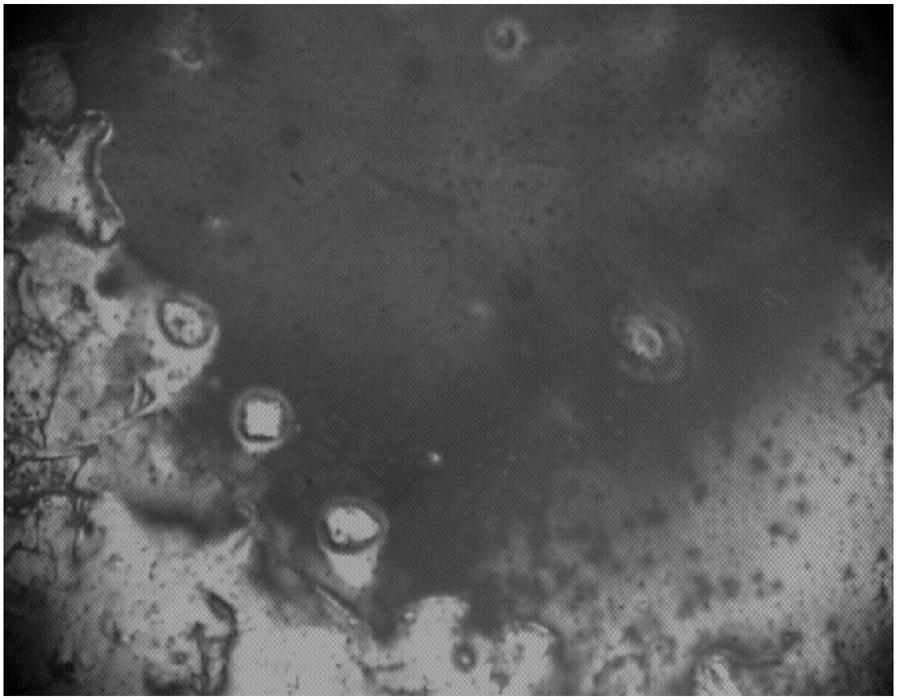

Figure 40 This photo is of the hole region on Fred. He has always been a star performer, and he doesn't disappoint here either. Notice the small number of flaws in the doped region and it's smootheness.

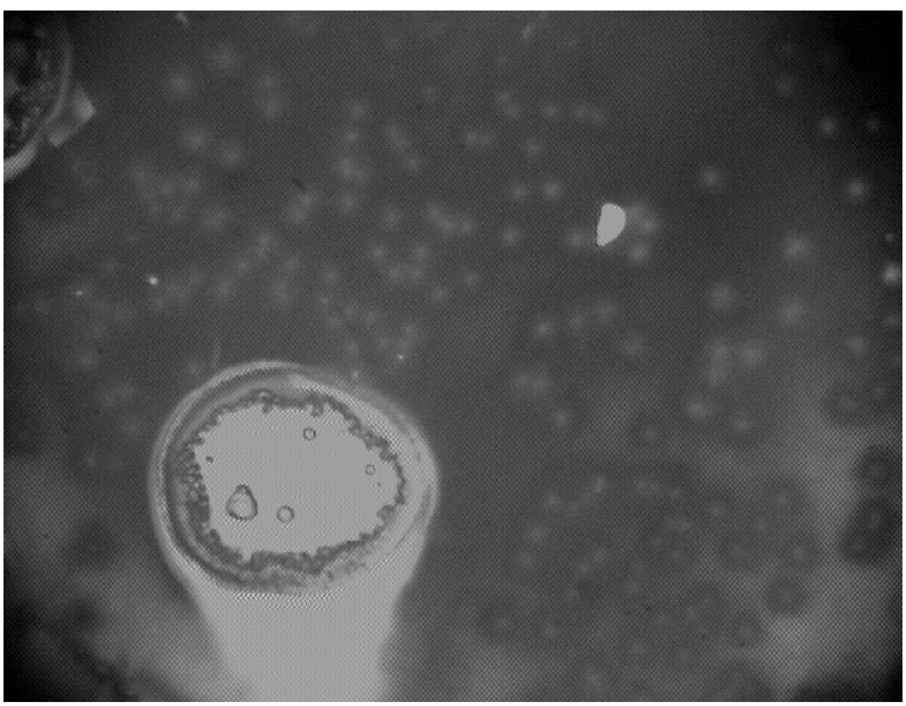

Figure 41 Here is a magnified look at Fred's hole. There is a good look at the doped region and also one of the pixels.

Appendix IV: I-V Characteristic Curves for wafers

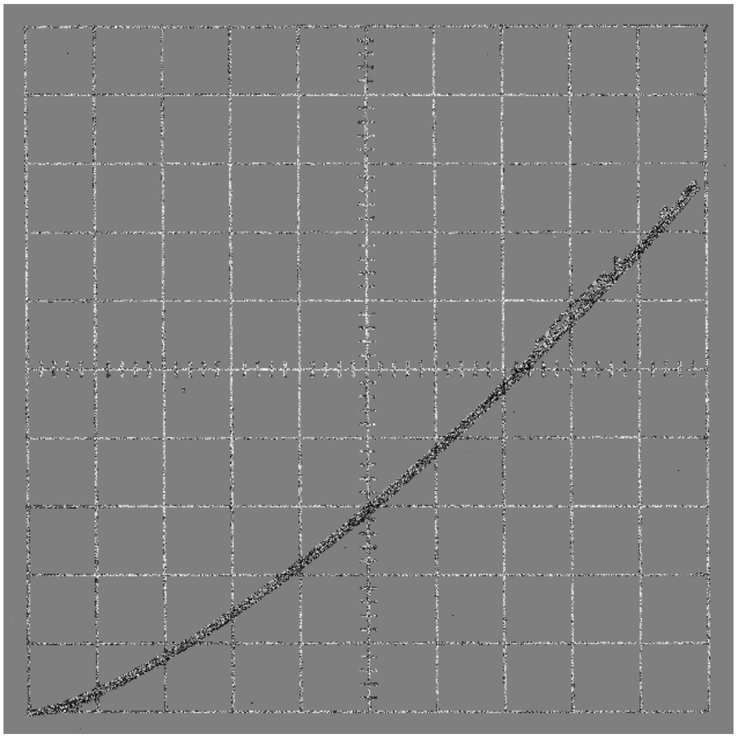

Figure 42 This graph shows the I-V Characteristic curve for Al. The x-axis is volts with each square being 500 mV. The y-axis is current with each square being 1 mA. Al's curve isn't very impressive at all. There is also some gitter at the top of the curve showing that Al wasn't even making that good of a resistor. There is still some curve though.

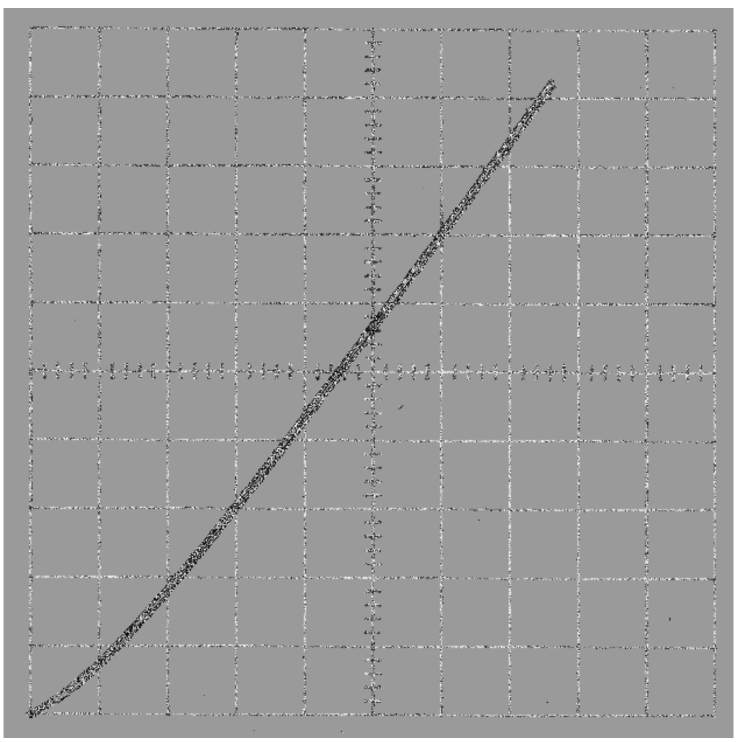

Figure 43 This graph shows the I-V Characteristic curve for Bob. The x-axis is volts with each square being 500 mV. The y-axis is current with each square being 1 mA. Bob looks like he is pretty much a resistor. The slop seems to be mostly linear.

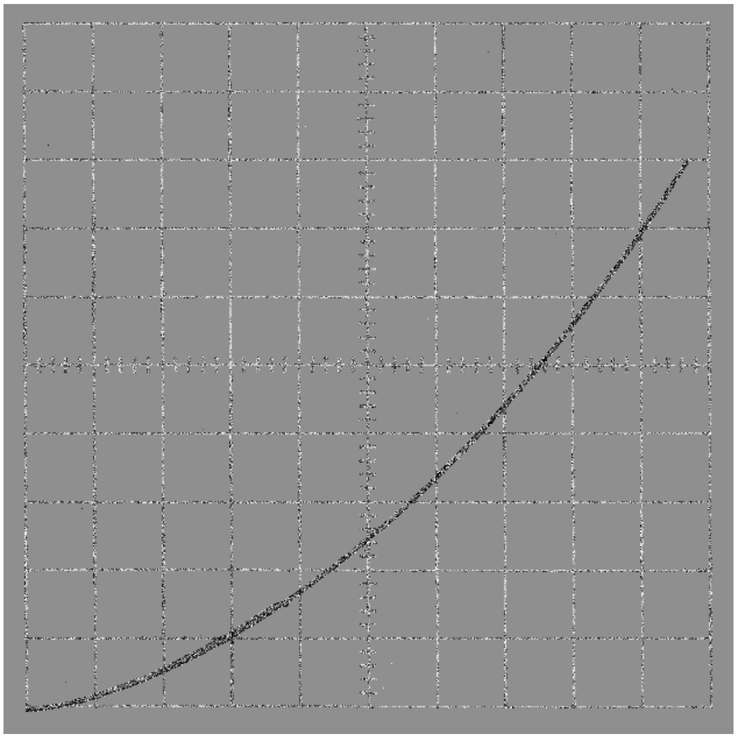

Figure 44 This graph shows the I-V Characteristic curve for Chuck. The x-axis is volts with each square being 500 mV. The y-axis is current with each square being 1 mA. Chuck did better than expected with a flatter region at the bottom, but the slot at the top of the curve isn't great.

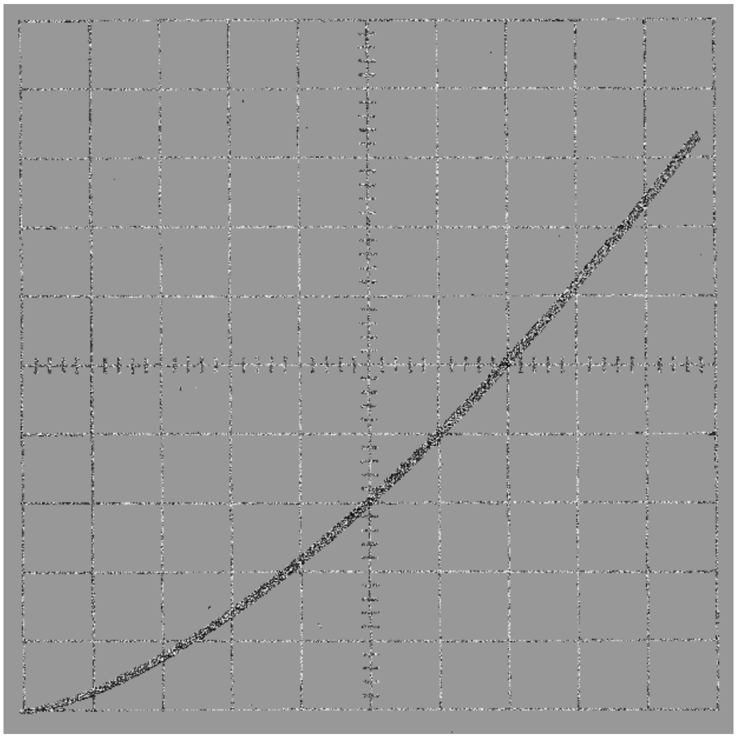

Figure 45 This graph shows the I-V Characteristic curve for Eloise. The x-axis is volts with each square being 500 mV. The y-axis is current with each square being 1 mA. Eloise performed okay, the curve doesn't have much of a slope in this low voltage region.

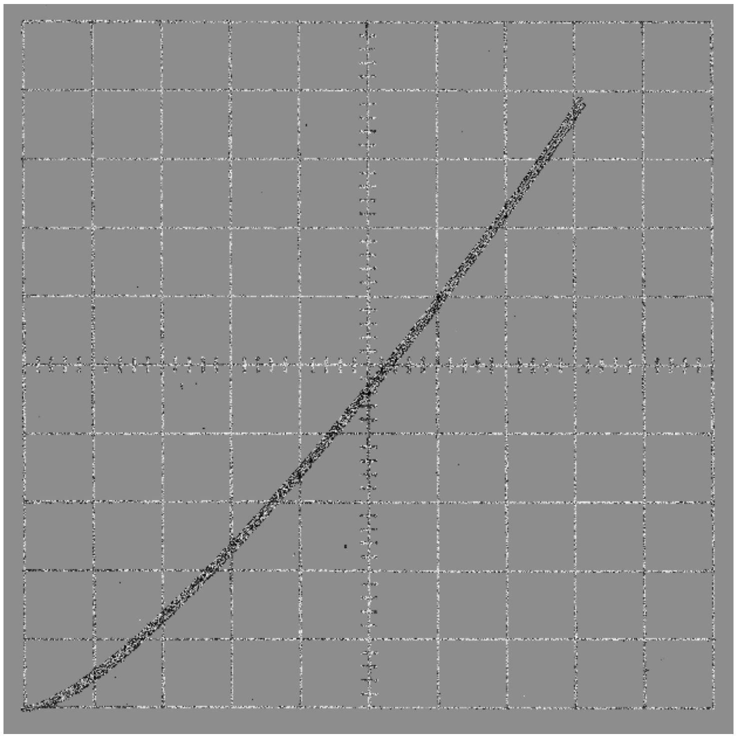

Figure 46 This graph shows the I-V Characteristic curve for Fred. The x-axis is volts with each square being 500 mV. The y-axis is current with each square being 1 mA. Again Fred is our star performer having the highest slope and most exponential curve of any of the wafers.