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Modified 24 May 1999 to add section 6 regarding further tests.

Note: This document is best accessed as a Word file, since the images are in color, and the original electronic version allows magnified views of the images.

## Zygo Optical Profilometer Measurements of Soft Nickel Coupon: Sample Tests for Mach-6 Quiet-Flow Nozzle Throat

### 1. Introduction

A soft nickel coupon was fabricated to test the performance of GAR's soft electroformed nickel, which was used by DEI on the Langley Mach-6 quiet nozzle, and is an option for use on Purdue's Mach-6 quiet nozzle. The total cost of the throat section is about \$100K, and the largest flaw in the finish controls the tunnel performance. Our concern is our ability to maintain the low initial roughness, despite operating temperatures of about 400-550F. Performance degradation was earlier observed by Frank Chen at Langley, for unknown reasons. The vendor, DEI, sent me a specimen of the subcontractor's (GAR) standard soft sulfamate nickel bath, nominally the same kind of bath used for Langley's Mach 6 electroformed nozzle. DEI determined the Rockwell hardness as Rc21,22,23 in 3 tests.

The 2x2x1/8-inch specimen had a mirror finish with some scratches. In the first test, it was placed in the oven at about 450F for 24 hours, then allowed to air cool. The oven is electrically heated and open to the lab air. The surface became somewhat cloudy. The mirror surface turned purple blue near some of the edges. The color is reminiscent of steel that has been heated. Wiping the somewhat cloudy surface with the soft cloth used for shipment by DEI restored the full mirror surface.

It seemed possible that we were seeing some oxidation of the mirror surface, even at these temperatures, which are low compared to those studied in the literature. It may be that very small levels of oxidation are noticeable due to the mirror surface. The results certainly seemed reminiscent of those mentioned by Frank Chen at LaRC regarding the LaRC M6. It is not clear that the cloudiness has any real effect on the surface roughness. It also seemed possible that the cloudiness was due to oil vapor or dirt from the lab. However, it took a bit of effort to wipe it off, which tended to suggest it was not only dust.

In a second test, I heated the 2x2 inch soft nickel coupon for 2 more days at 425F. It air cooled over the weekend. The surface was again cloudy, although you could still see yourself in it. I wiped it hard with the soft cloth from DEI, there was little apparent effect, it was still cloudy. I wiped half of the surface with some Happich Simichrome Polish. This made a mirror surface of that side, although it seemed also to add some very fine scratches. I wiped both sides with acetone and the soft cloth, and there was little apparent effect, besides cleaning up the residual polishing compound.

## 2. Initial Hypothesis

The cloudy finish was thought to be caused by oxidation of an initially mirror-like surface (electroformed on glass). The shiny side is the result of briefly polishing the cloudy surface (and apparently introducing some fine scratches).

We needed to determine the following: 1) Is it really oxidation that makes the surface cloudy? 2) Does this oxidation degrade the surface roughness significantly, or does it merely cause a monolayer of nickel oxide? The target roughness is less than 1 microinch rms, with peak roughnesses of less than about 50 microinches. Although the specimen is not polished to this quality, can we determine if the oxidation will be a significant issue at this level?

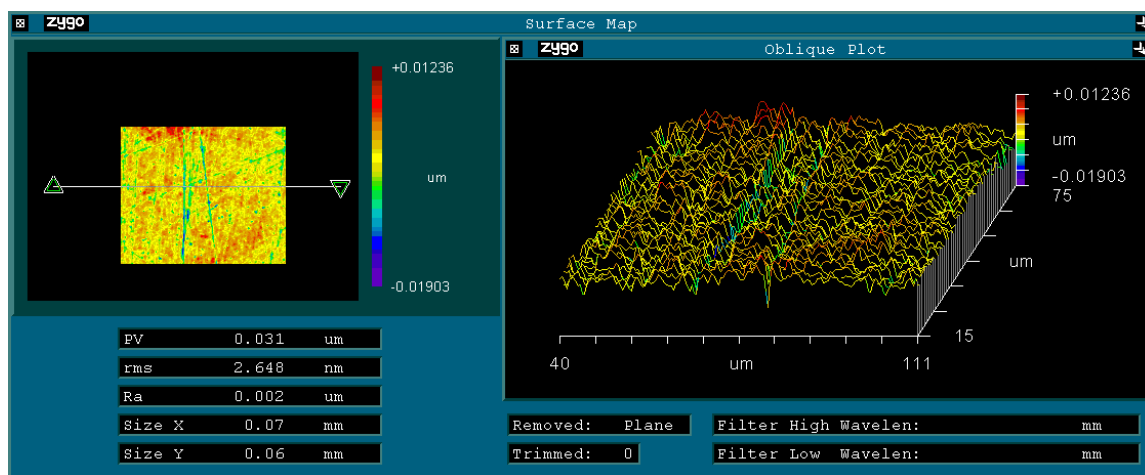
## 3. Optical Profilometer Measurements

Chris Tieche carried out measurements on the specimen using the Zygo optical profilometer at Purdue, and provided a set of .bmp files, along with a handwritten summary. This section presents his results. Chris is a graduate student in tribology who works under the direction of Prof. Tom Farris.

Chris took 5 measurements for each surface using the optical profilometer. Since getting raw data out of this instrument has not been successful, he supplied some data that represents the material in the output files. Although these were supplied as .eps files initially, they turned out to be .bmp format.

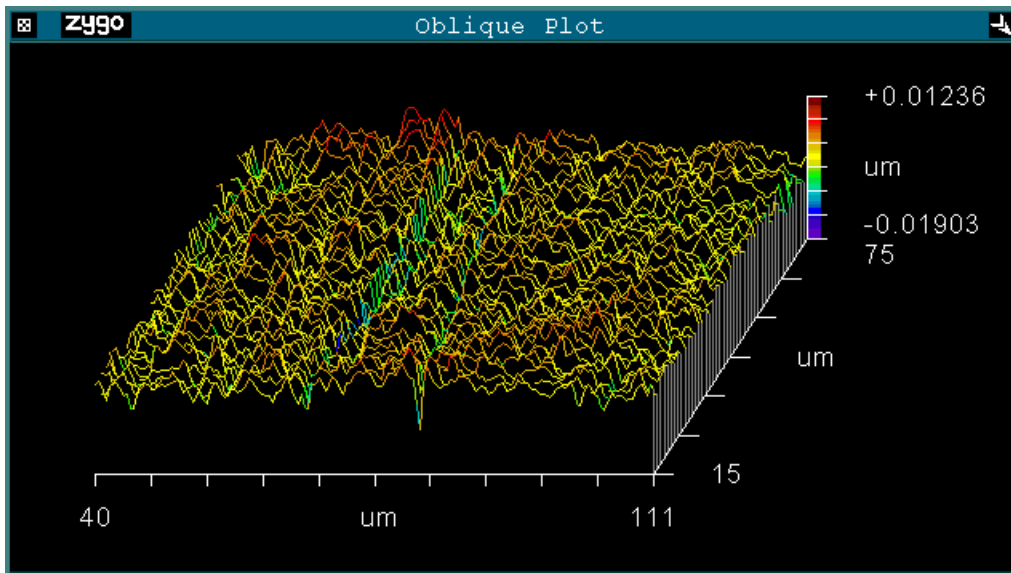
### 3.1. Measurements on the Polished Area

Figure 1 shows a top view of the area selected for the first measurement. The rms roughness is 2.6 nm and the Ra roughness is 0.002 microns (0.1 microinches). No scratches were evident on the surface.



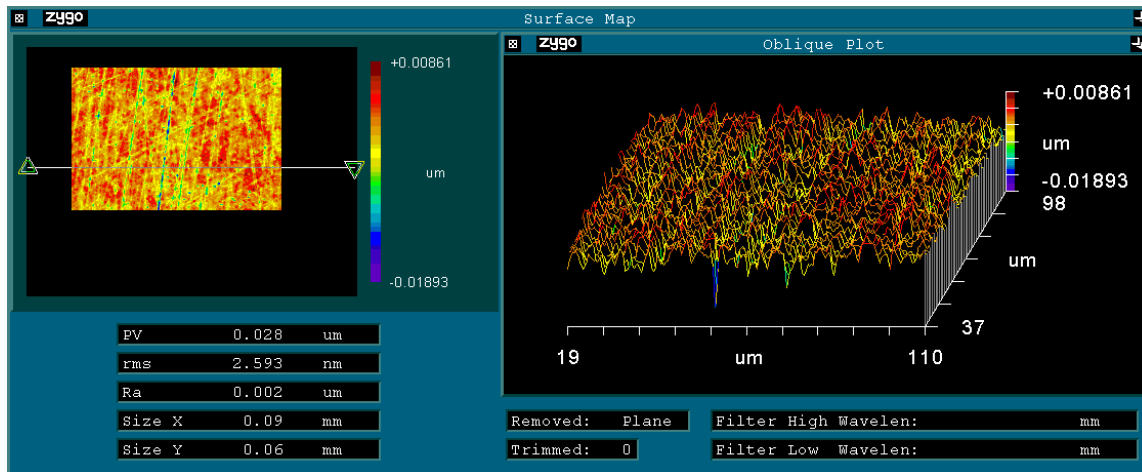
**Figure 1: Top View of Polished Area, Portion 1**

Figure 2 shows an oblique closeup view of the same area. These are available for each view but will mostly be omitted since they duplicate a portion of the image in Fig. 1.



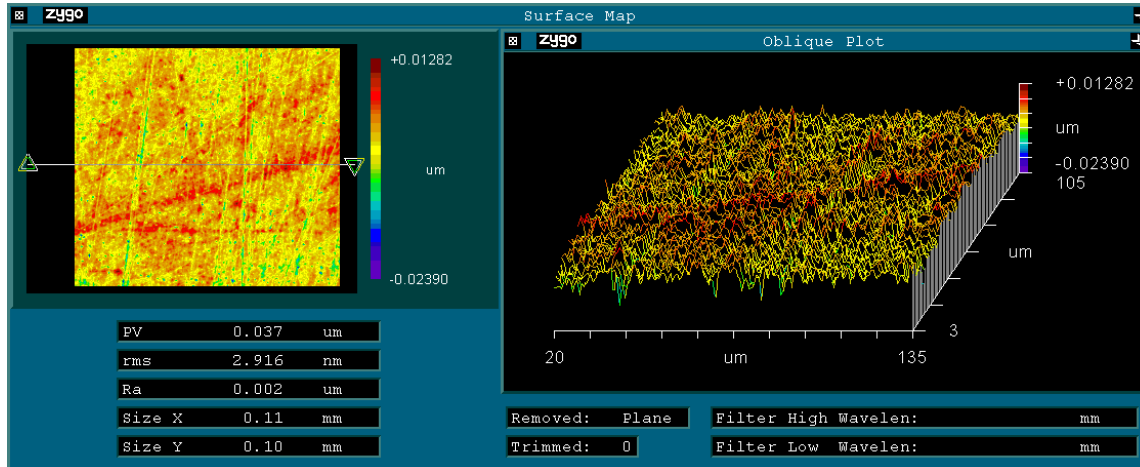
**Figure 2: Oblique View of Polished Area, Portion 1**

Figure 3 shows an image similar to Fig. 1, but from a different portion of the polished surface. Here, the rms roughness is 2.6 nm, again, and Ra=0.002 microns (almost the same value, a slightly different definition).



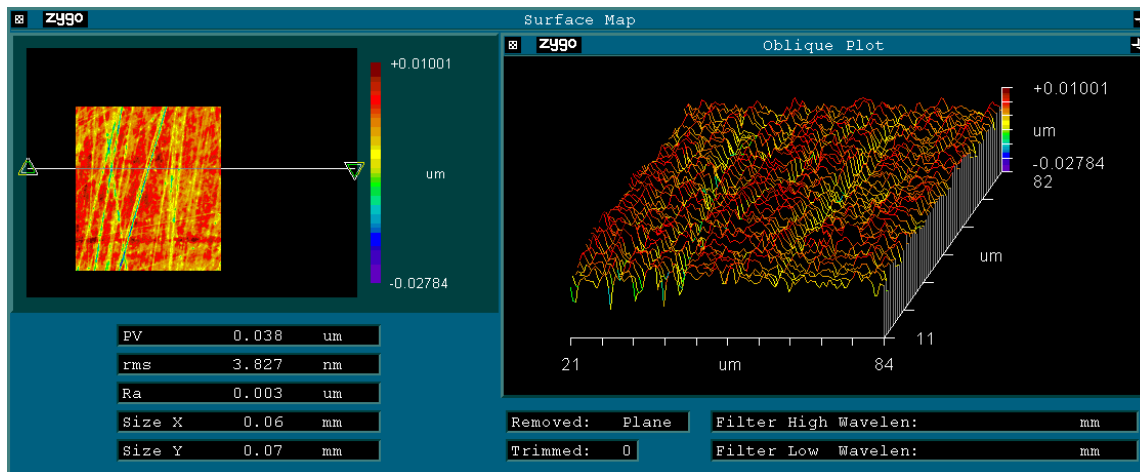
**Figure 3: Polished Area, Portion 2**

A larger area was scanned for portion 3, so that scratches were integrated into Ra and the rms roughness. The rms is now slightly increased to 2.9 nm, as shown in Figure 4. Some scratches can be seen in the figure. However, the roughness is still within about 0.02 microns peak, or about 0.8 microinches.



**Figure 4: Polished Area, Larger Area Scan, Portion 3**

Figure 5 shows another part of the polished surface, including several relatively deep scratches, about 0.025 microns deep (about 1 microinch).



**Figure 5: Polished Area, Portion 4, Some 'Deep' Scratches**

Finally, Figure 6 shows the fifth image of the polished area. In this figure, a still larger area is imaged. The rms roughness has increased to 3.3 nm in this image, still about 0.13 microinches. The maximum deviations are still about 0.03 microns, or about 1 microinch, a very high quality finish.

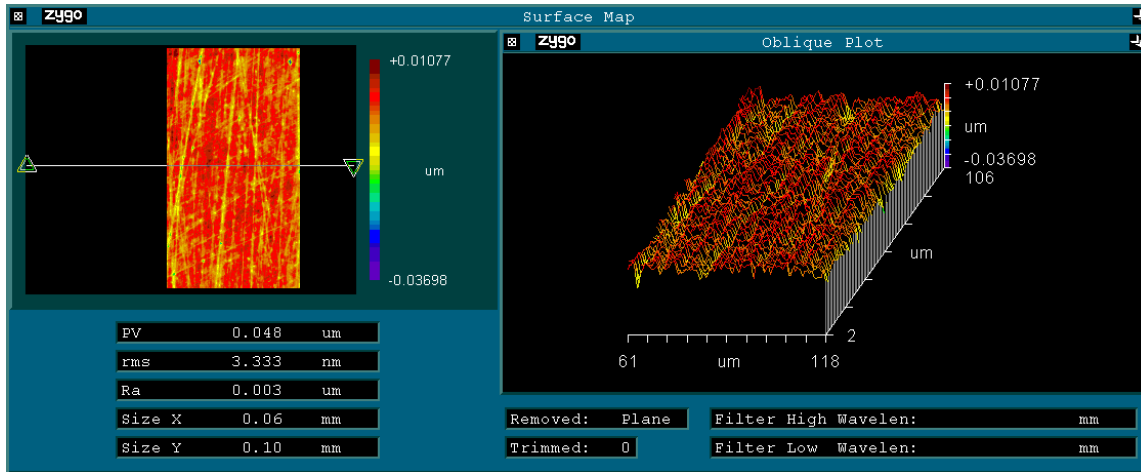


Figure 6: Polished Area, Portion 5, Larger Area Scan

### 3.2. “Oxidized” Surface

The next set of images were obtained on the “oxidized” surface. This is the area that was not polished. It was initially mirror-like, but became cloudy looking to the eye after being heated. It was never polished. Figures 7-11 show the 5 portions imaged by Chris.

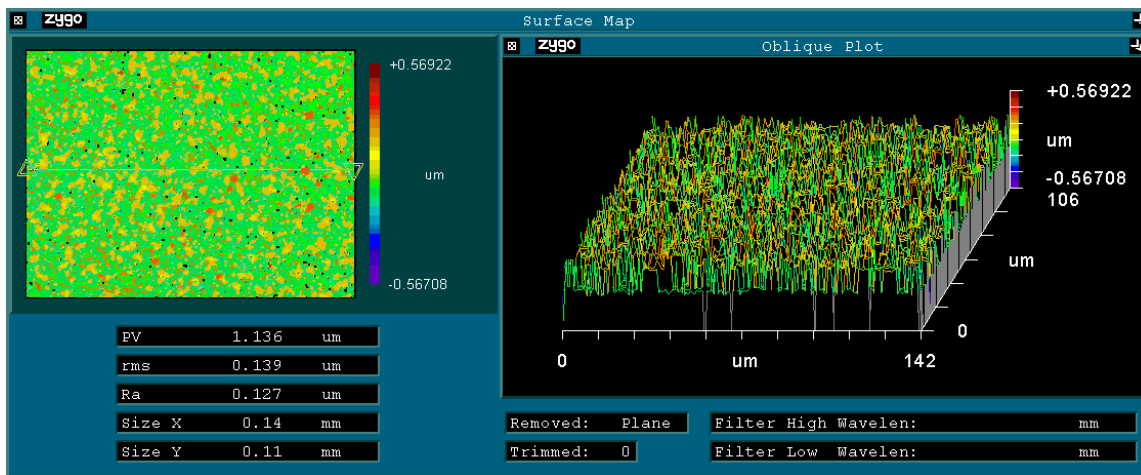


Figure 7: Unpolished Area, Portion 1

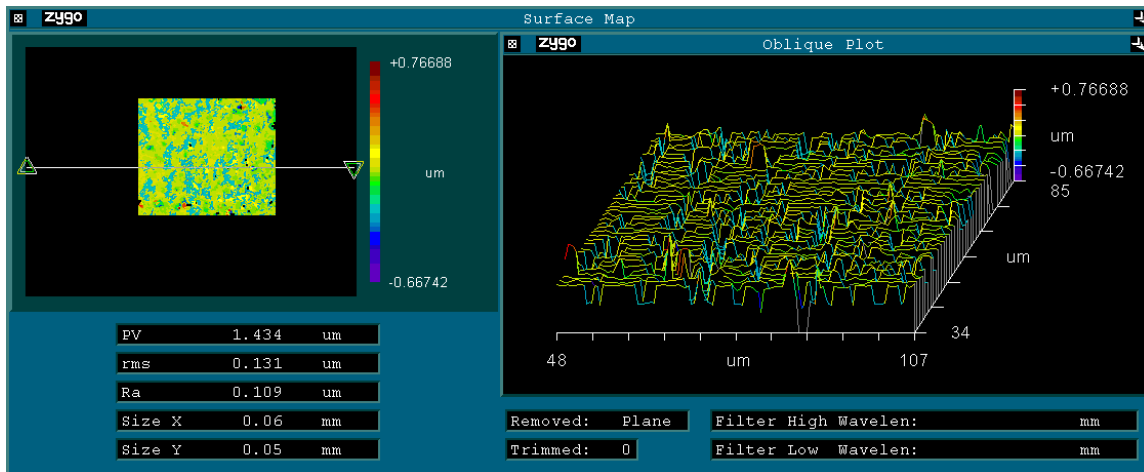


Figure 8: Unpolished Surface, Portion 2

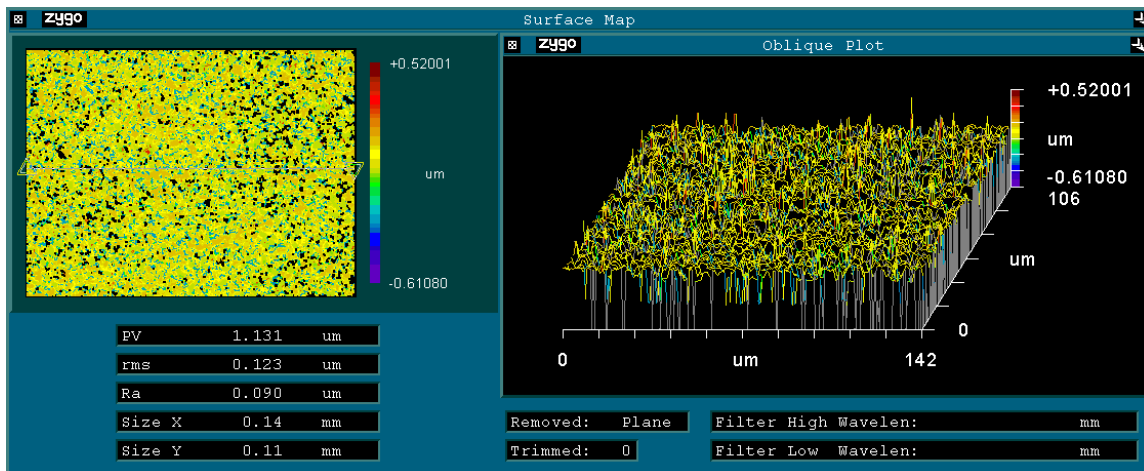


Figure 9: Unpolished Area, Portion 3

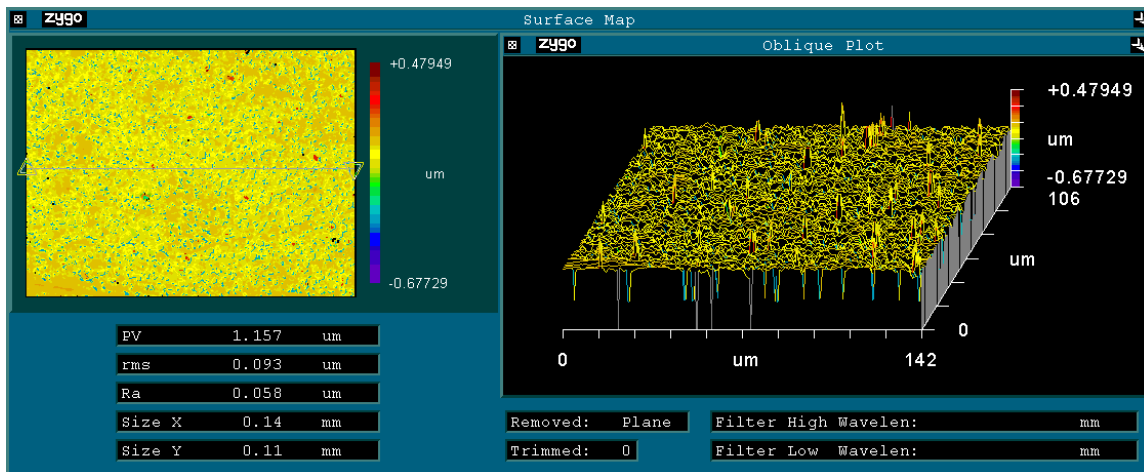
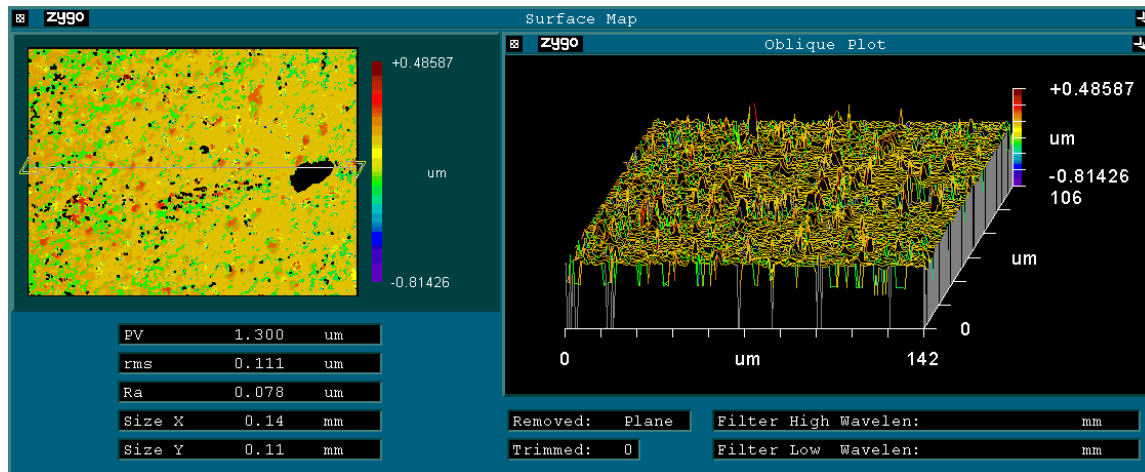


Figure 10: Unpolished Area, Portion 4



**Figure 11: Unpolished Area, Portion 5, Large Cavity on Right-Hand Side**

The rms roughness ranges from 0.09 to 0.14 micrometers, or from 3.5 to 5.5 microinches. The peak deviations are roughly 0.8 microns, or about 31 microinches, a value that is very significant in terms of generating throat roughness that would adversely impact quiet-flow performance. These roughness values are much higher than the ones for the polished surface. Unfortunately, no Zygo data was taken before the specimens were first put in the oven, so we cannot verify directly that the temperature generated the roughness from what appeared to be an initially mirror smooth specimen. However, we have verified that once the nickel is polished, after initial heating, it remains smooth after further heating.

#### 4. Interim Conclusions and Further Work

These measurements show that the cloudy surface has significant roughness, much larger than the mirrored surface. It may be that the initial mirror surface on the part had low roughness. In this case, the cloudiness that developed indicates the development of roughness due to heating, perhaps because of corrosion or oxidation of the initial surface. This surface is a peculiar layer in the electroform, since it is the portion initially deposited on the glass.

When this cloudy surface was polished, very lightly, a mirror surface was again visible. This mirror surface has very low roughness, and maintains its low roughness after considerable additional time in the oven. This suggests that the initial surface present after the electroforming is peculiarly sensitive to heating, in a way that the lower layers of material are not.

The following tests should probably be performed:

- 1) Test the hard nickel and layered hard/soft nickel coupons
- 2) Measure the roughness of a soft nickel coupon as it comes initially from the electroformer. See if the soft nickel problem repeats.
- 3) Leave the soft nickel coupon in the oven for many days, see if a deterioration in the polished surface is evident.

X-ray measurements were performed on the soft nickel coupon, these found only nickel, but they cannot tell if there was a surface layer of oxide. After these measurements were obtained, the specimen was placed back in the oven at 218C (424F) for 4 days. Scanning electron microscope

images of the surface are being obtained today. They will be followed by an additional round of Zygo images, to see if any further degradation is visible.

If the above hypotheses are verified, we may need to polish the nickel surface after removal of the mandrel, and after an initial temperature test.

## 5. Additional Information Regarding Sample Preparation

After the above information was communicated to DEI, Larry DeMeno checked with GAR. It appears that GAR deposited a thin layer of silver ("1-2 mil") on the glass substrate before electroforming the sample. It seems likely that this silver layer oxidized or corroded during heating, causing the cloudy surface. Alternatively, the alkaline solution used by GAR to remove the silver may have left a surface layer on the nickel which then pitted. No release agent was used. GAR did not understand the critical nature of the coupons.

The hard nickel cannot be plated on a flat piece of glass, since it peels away due to surface tension. Only the soft nickel can be done this way. A test involving plating on a stainless layer is probably in order, but it remains to be determined whether this should involve a flat sample (for easy Zygo measurements) or a mandrel sample (for tests of peeling, etc, and for the hard nickel, but hard to make surface measurements then).

## 6. Second Set of Tests on Soft Nickel Coupon

The soft nickel coupon was placed back in the oven on 5/14/99 and left there for 3 days 21 hours. The oven temperature was 217-218C or about 425F. The sample was then removed and allowed to cool, after which it was cut into two 1x2 inch pieces, each containing about half mirrored side and half cloudy side. A line was scribed across one piece between the mirrored and cloudy sides. This piece was delivered to Mark McCormick in MSE for scanning electron microscope imaging. These images are not shown here since the Zygo images are more instructive. The piece was then delivered to Chris Tieche for additional Zygo measurements.

*"I have measured both sides of the soft nickel. Since RMS and Ra are in the bmps, I'll only quote you the max deviation here. "s" is for the smooth side, "r" is for the rough. "map" means the surface maps containing both the top view and the oblique view, and "sp" is the surface profile along the guide line visible in the top view. 10x is the objective lens power. The sizes for all of these pictures are 0.36x0.27mm.*

<i>file</i>	<i>maxdev</i>	
<i>s...1</i>	<i>15nm</i>	
<i>s...2</i>	<i>20nm</i>	
<i>s...3</i>	<i>30nm</i>	<i>350nm pit pictured</i>
<i>s...4</i>	<i>30nm</i>	<i>700nm pit</i>
<i>s...5</i>	<i>25nm</i>	<i>I forgot to save the picture itself, but RMS=3.513, Ra=2.303</i>
<i>r...1</i>	<i>1000nm</i>	<i>pit at left</i>
<i>r...2</i>	<i>1000nm</i>	
<i>r...3</i>	<i>1000nm</i>	<i>several 1um pits at lower right</i>



r...4            1000nm  
 r...5            700nm

*The rough pictures have some voids in them because the surface is so rough that the zygo can't get reflected light from them. RMS and Ra are not affected by the voids however, and the pictures still look pretty good. Also, I have noticed that rough side is not of uniform roughness, but changes, not only in the degree of severity, but also in the general profile -- some of the profiles look "square-wavish" while others are more random looking."*

Figures 12-19 show the images corresponding to this data, for the smooth side. The 5<sup>th</sup> area imaged was not recorded, so no files are available to show here.

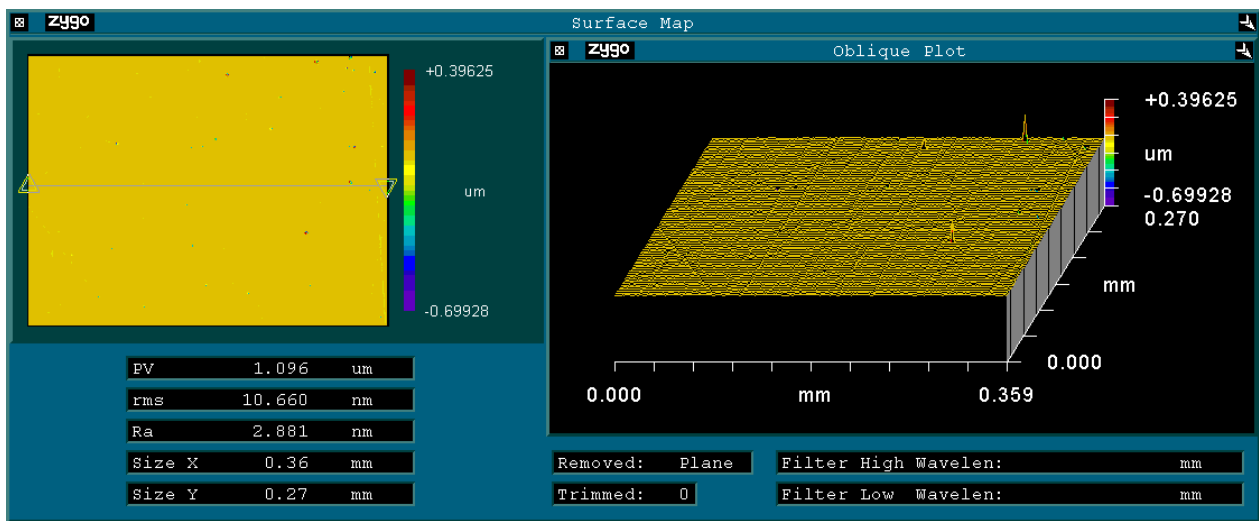


Figure 12: 1st Portion, Smooth Side

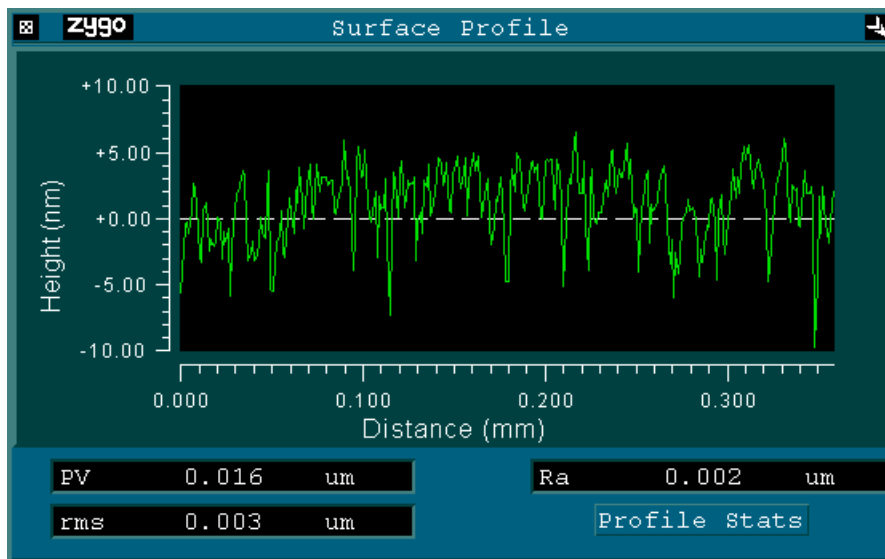


Figure 13: 1st Portion, Smooth Side, Selected Profile

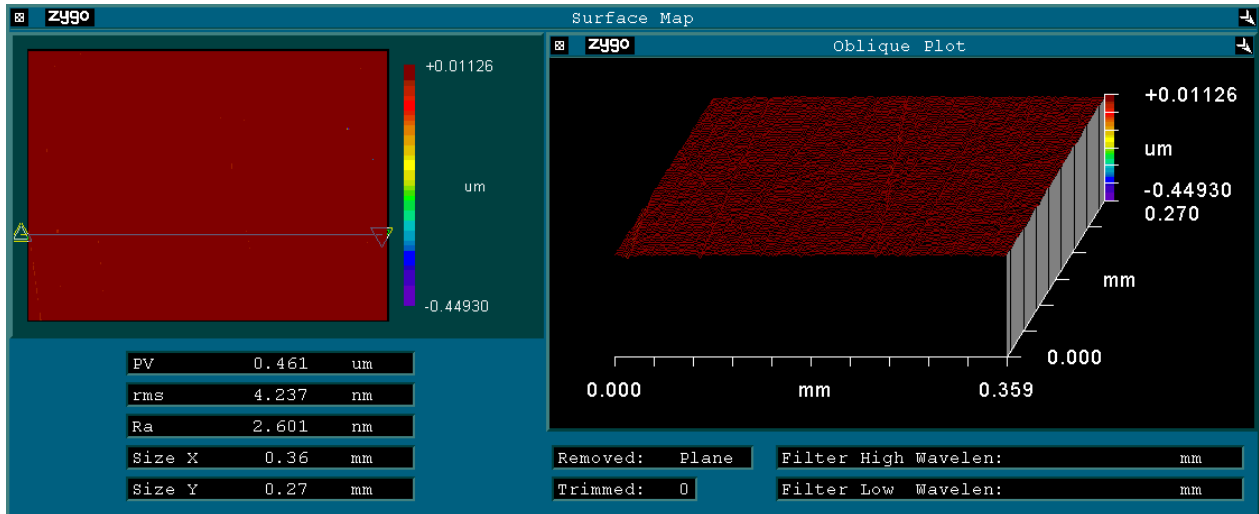


Figure 14: 2nd Portion, Smooth Side

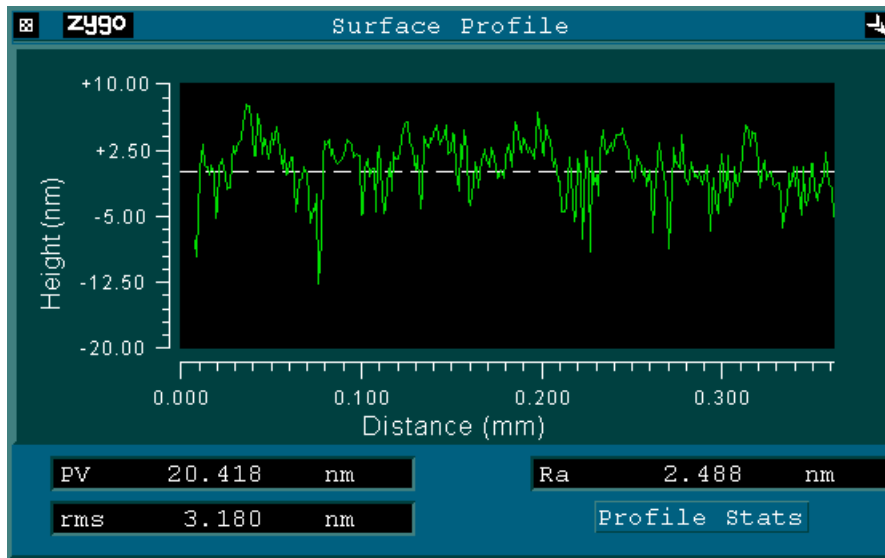


Figure 15: 2nd Portion, Smooth Side, Selected Profile

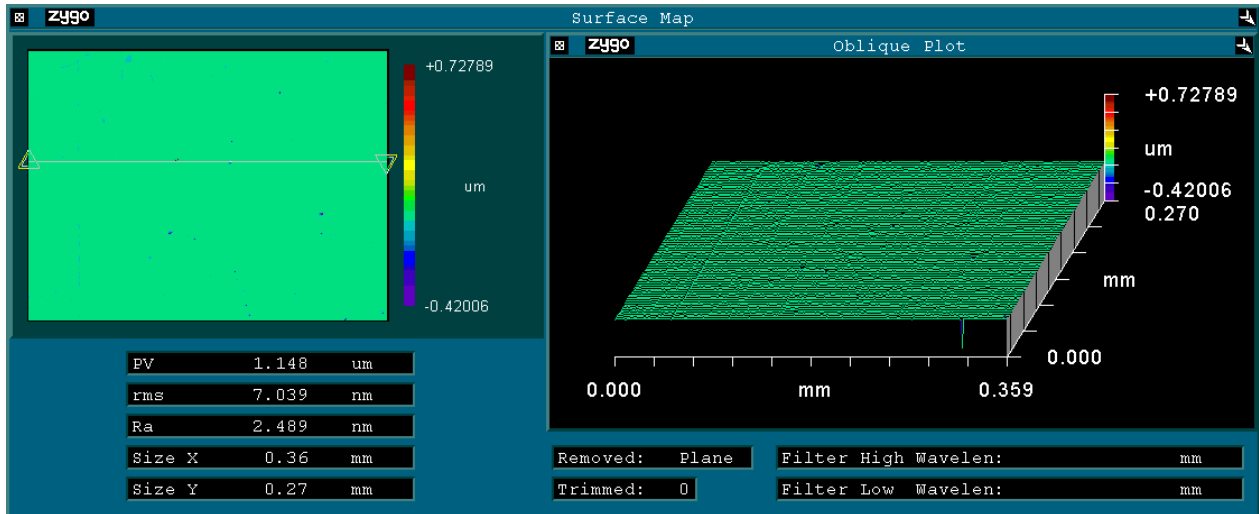


Figure 16: 3rd Portion, Smooth Side

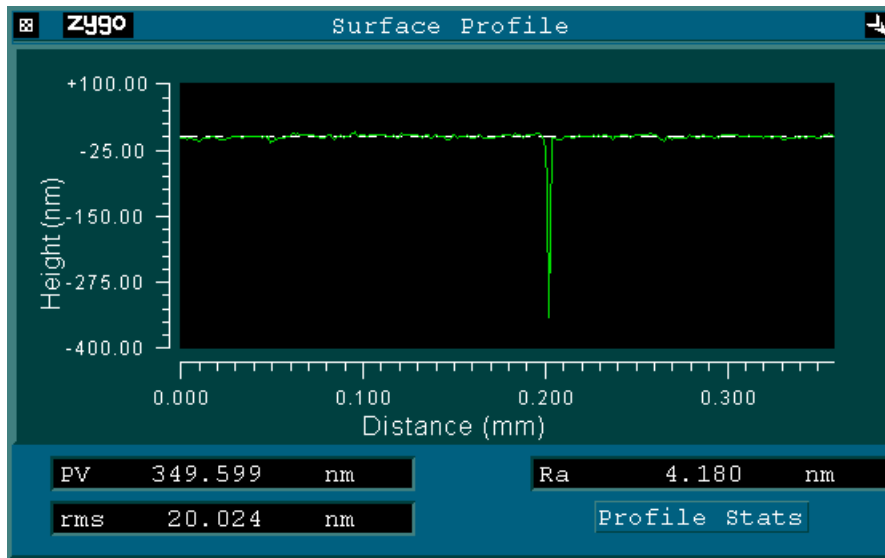


Figure 17: 3rd Portion, Smooth Side, Selected Profile

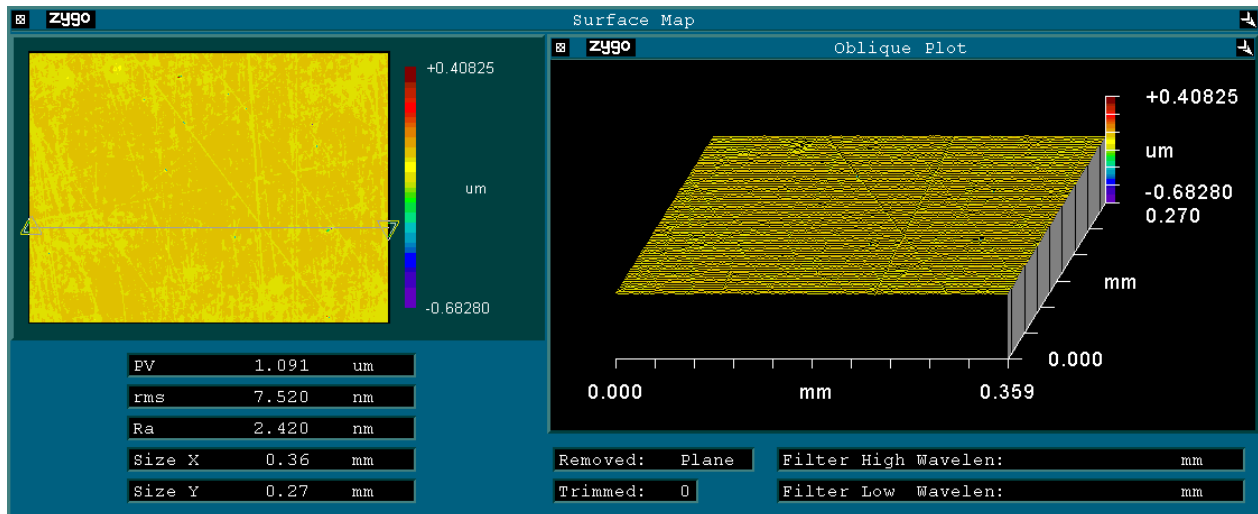


Figure 18: 4th Portion, Smooth Side

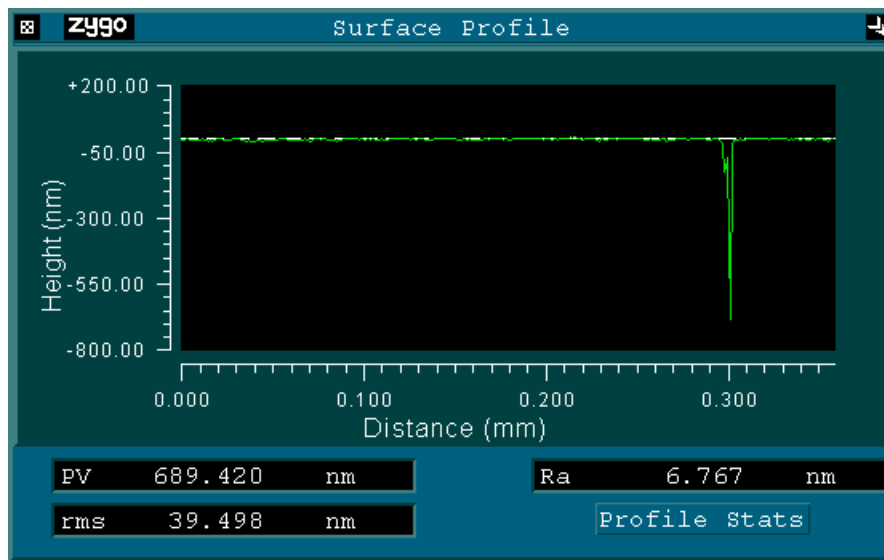


Figure 19: 4th Portion, Smooth Side, Selected Profile

The rms values of roughness for the 5 portions are 10.6, 4.2, 7.0, 7.5, and 3.5 nm. Before this second heating cycle, the rms values for 5 (different) portions were 2.6, 2.6, 2.9, 3.3, and 3.8 nm. However, those earlier measurements were for smaller areas, 0.06 x 0.10 mm, so they probably have smaller rms values, since they won't include as many large scratches. The values of Ra before the 2<sup>nd</sup> heating cycle were 0.002, 0.002, 0.002, 0.003, and 0.003 micrometers, or 2-3 nm. After the 2<sup>nd</sup> heating cycle, the values were 2.8, 2.6, 2.5, 2.4, and 2.3 nm. This suggests that the 2<sup>nd</sup> heating cycle did not degrade the polished surface. I conclude that **the nickel itself is not degraded by the 425F temperature, only the initial surface layer was a problem.**

The smooth electroform still contains a number of large pits. These remain a concern, although the Ra values are low.

Figures 20-29 show the data for the rough side.

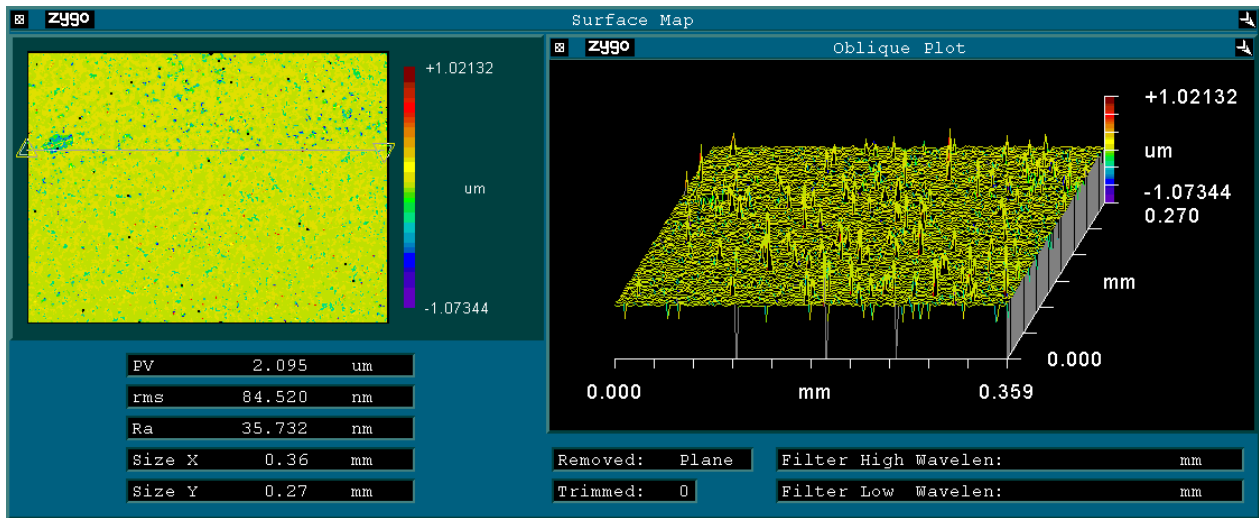


Figure 20: 1st Portion, Rough Side

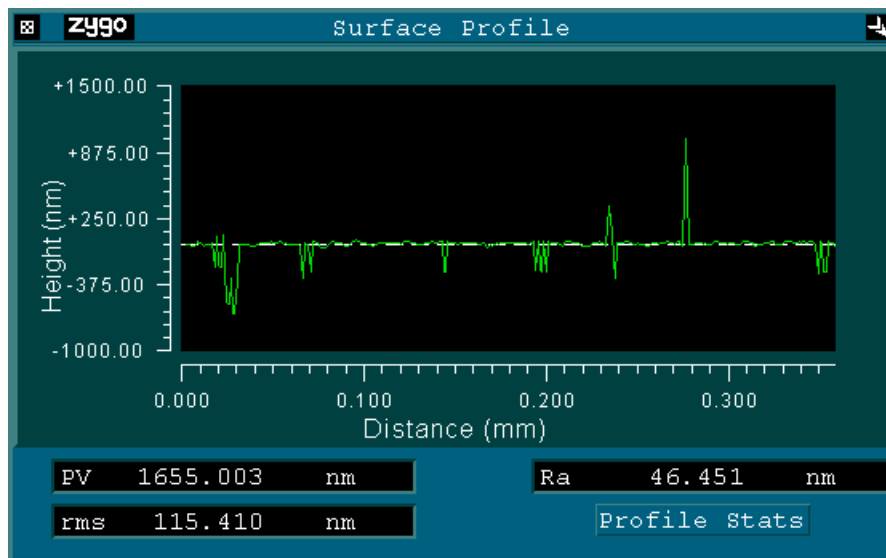


Figure 21: 1st Portion, Rough Side, Selected Profile

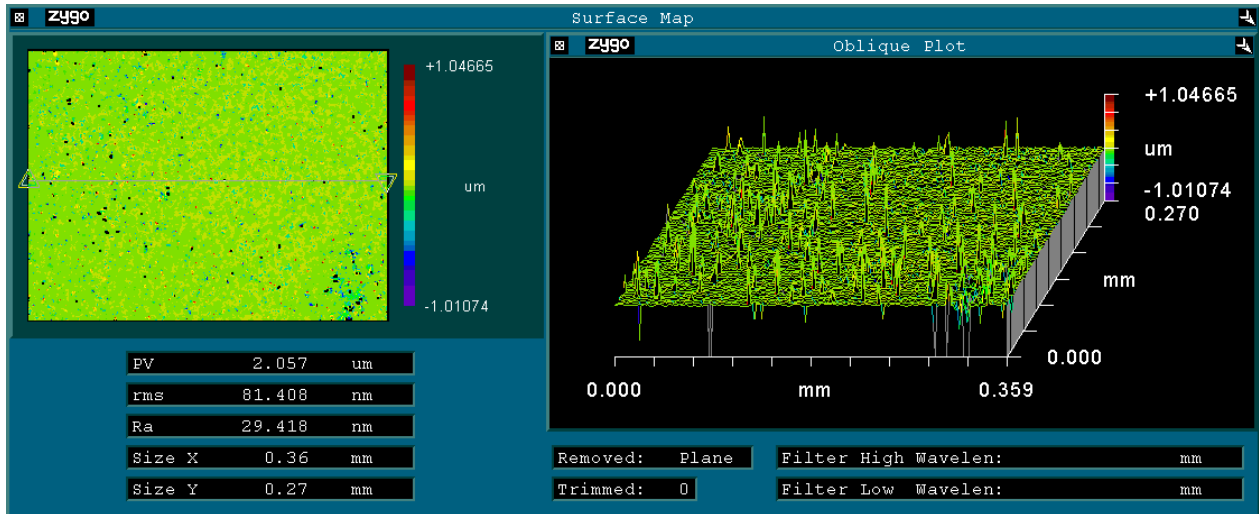


Figure 22: 2nd Portion, Rough Side

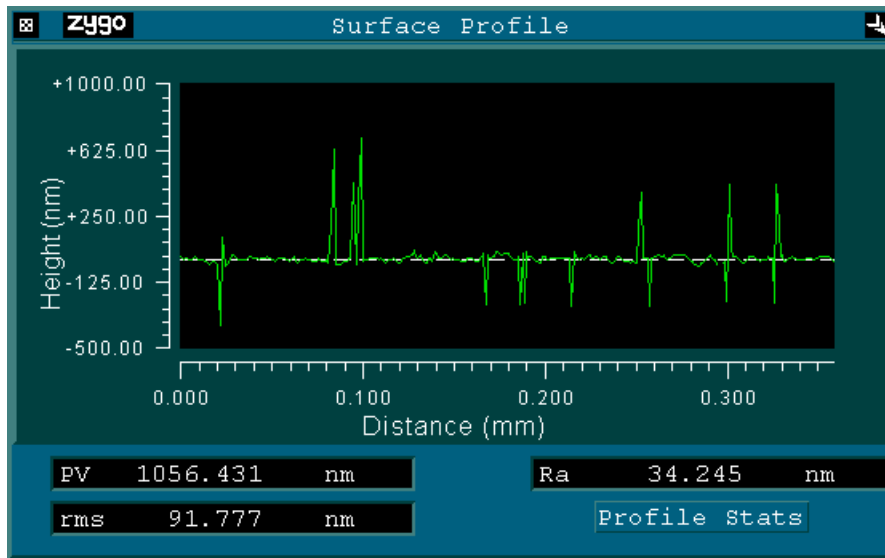


Figure 23: 2nd Portion, Rough Side, Selected Profile

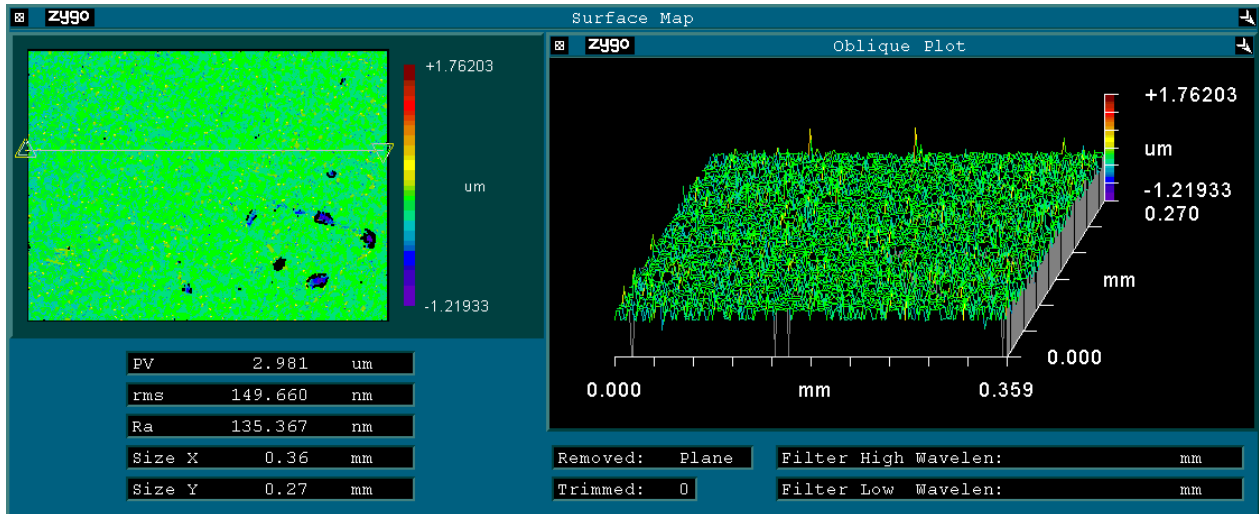


Figure 24: 3rd Portion, Rough Side

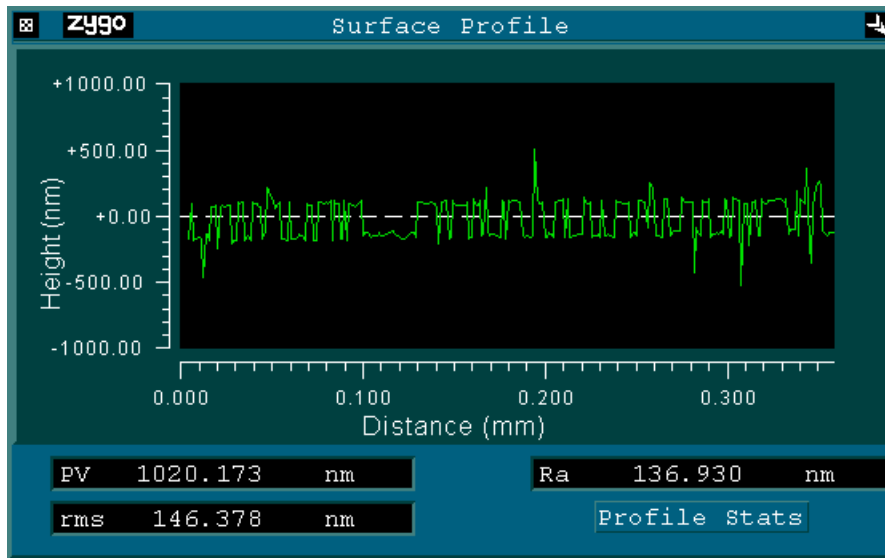


Figure 25: 3rd Portion, Rough Side, Selected Profile

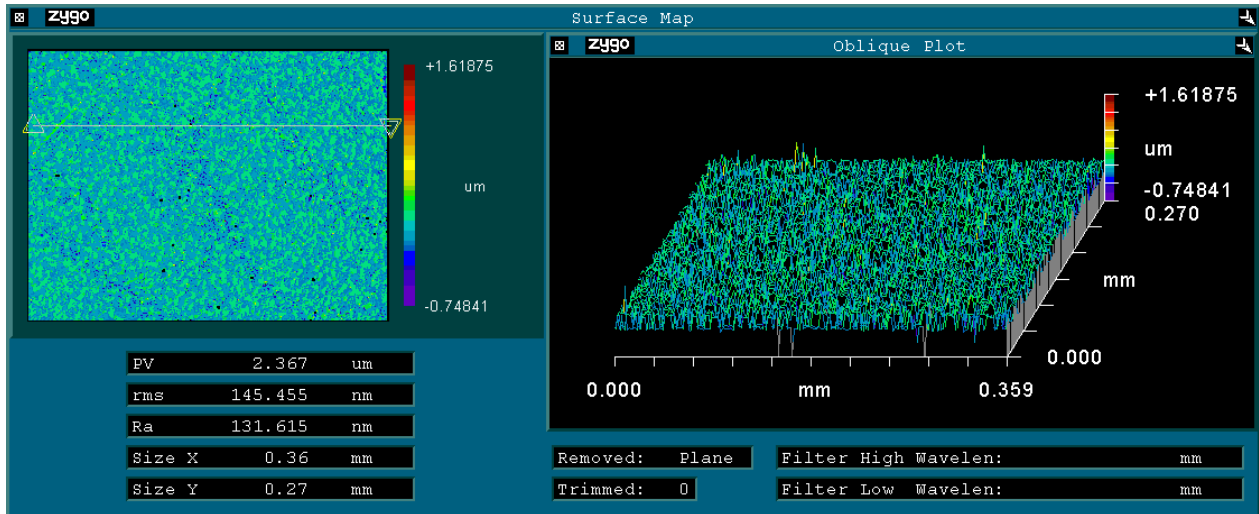


Figure 26: 4th Portion, Rough Side

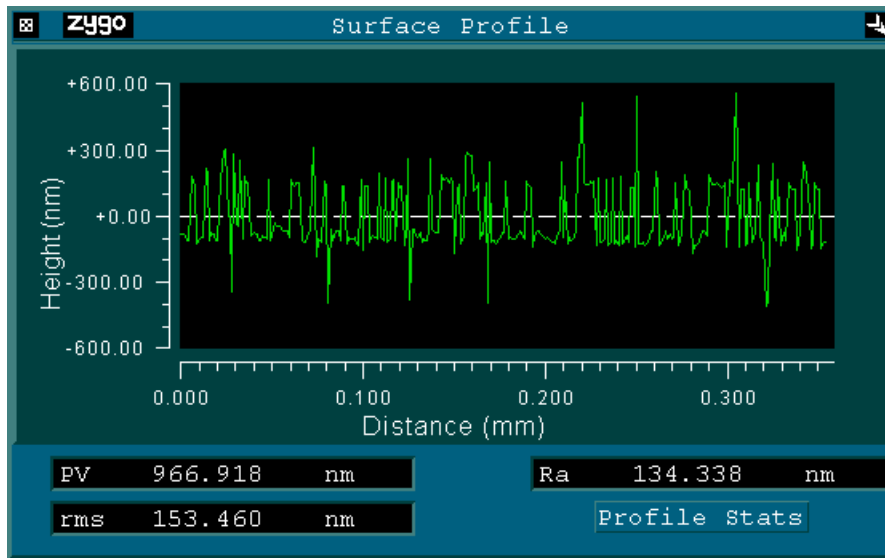


Figure 27: 4th Portion, Rough Side, Selected Profile



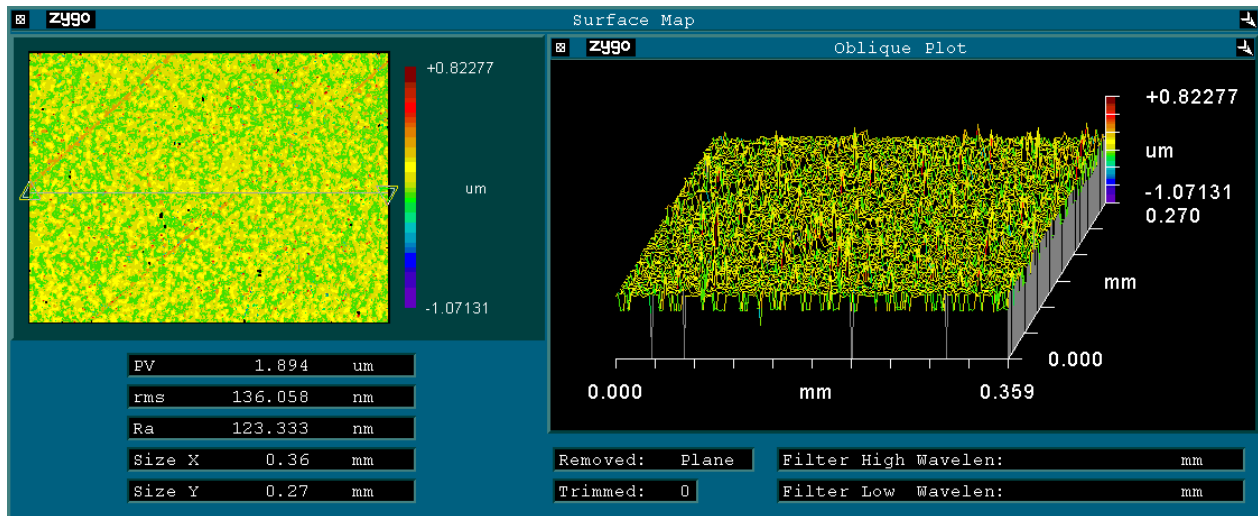


Figure 28: 5th Portion, Rough Side

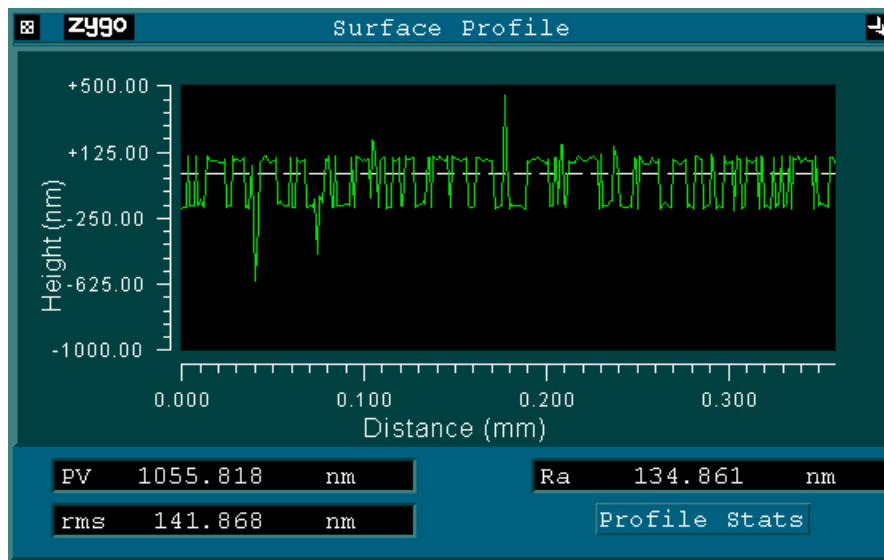


Figure 29: 5th Portion, Rough Side, Selected Profile

The values of Ra on the cloudy or rough surface before this 2<sup>nd</sup> heating were 0.13, 0.11, 0.09, 0.06, and 0.08 micrometers, or 80-130 nm. After the 2<sup>nd</sup> heating, the values were 36, 29, 135, 131, 123 nm. It appears that the 2<sup>nd</sup> heating did not noticeably further degrade the cloudy surface either. The square-wave-like profiles may be the remains of the silver layer. If so, they are about 300 nm or 10 microns thick. In any case, the cloudy surface would be unacceptable for a quiet nozzle throat.

## 7. Additional Interim Conclusions

The cloudy surface is completely unacceptable for a quiet nozzle throat. Even the polished smooth surface contains a number of large pits, which might cause serious problems, although they are of less concern than similar spikes would be. Tests on a more representative electroform are needed.

Test 3 from the list in section 4 has been performed. The sample does not degrade further under additional heating. It appears that the initial surface layer was the problem. Although the special processing performed on this coupon by GAR may well have caused the problem, we have decided to build a small tapered-cylinder mandrel using the same material and same processes to be used on the actual nozzle mandrel. This test mandrel will be used by GAR to electroform a test nozzle section using the same processes planned for the actual nozzle. A surface prepared by the same means to be used on the actual nozzle can then be tested, to increase confidence and reduce risk.

Tests 1 and 2 cannot be performed at present. The hard nickel coupon received first from the vendor had a badly flawed surface. It was tested in the oven and no degradation was observable in the good parts of the surface, after 1 day in the oven, as reported elsewhere. It may be used for fracture-toughness tests. The first 6x6 coupon was also flawed, although again the mirrored hard surface did not degrade visibly after a day in the oven. It was used to test whether the hard/soft layered nickel would bend due to thermal effects when heated. As reported elsewhere, any such bending was less than 0.002 inches in amplitude, and therefore negligible.

Another 2x2 hard nickel coupon is to be delivered by GAR, along with another 6x6 layered coupon. These will be used to make a preliminary determination of our interest in using the hard nickel layering. The baseline nickel selection will then be electroformed onto the test mandrel as a final test before electroforming the nozzle mandrel.

