

## Surface Characterization in Engineering Curricula

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## **Surface Characterization in the Engineering Curriculum**

### **Introduction**

Every material, part, component, device, and system has surfaces and interfaces. The surface and interface properties (e.g., roughness, structure, optical reflection, emissivity, and cleanliness) often play a crucial role in the performance of many technologies. Despite its practical importance, surface characterization is a comparatively neglected subject in engineering curricula. Further, characterization of surfaces is an excellent vehicle for teaching metrology, statistical analysis, and quality assurance. Machined metals, 3D-printed plastics, and semiconductor materials for solar cells in various stages of production provide interesting and informative case studies for surface characterization. We have developed a suite of laboratory modules for surface characterization using stylus profilometry, depth gauge measurements, laser and LED light scattering, image processing, thermal imaging with infrared cameras, atomic force microscopy, and white light interferometry. Students learn the metrology and parameterization of surfaces, the techniques to measure and characterize surfaces, the advantages and disadvantages of various methods with regard to accuracy, information content, cost, time, contact vs non-contact, and localized vs global measurements, and how to determine best methods for research, process development, prototyping, and quality assurance. With these experimental modules, students will learn the cost of measurements in terms time, capital equipment, sample invasiveness, complexity, and the return with regard to accuracy, repeatability, and informational value.

### **Applications for Instruction and Course Work**

There are many metrics describing the geometry and other features of surfaces and also many options for characterizing surfaces based on optics (color, specular and diffuse reflection, scattering), mechanics (1- or 2-D topography, sliding friction), and surface chemistry (e.g., wetting). Each of the techniques or methodologies described below have been used by us in engineering courses and laboratories, either as demonstrations or student hands-on work for undergraduate Engineering Technology programs. These courses include: 1) undergraduate Engineering Materials, 2) CNC machining operations, 3) Rapid Prototyping, 4) Introduction to Nanotechnology, 5) Robotics, 6) Quality Assurance, and 7) Renewable Energy Engineering. As laboratory exercises, each instrument can be learned in a time frame of 30-60 minutes. With the exception of the AFM, most of these instruments range in cost from \$100 to \$1000, and can be readily interfaced with a laptop computer. We emphasize concepts related to correlating and collaborating measurements by different techniques. Further, many of these techniques can be done by remote access over the internet, for the benefit of on-line students. For instance, the techniques can be demonstrated with videos (recorded or live), and data sets can be analyzed by students as part of their on-line coursework. In the case of image capture (with digital CCDs and thermal cameras), the students can direct the camera and work on transmitted or archived images to assess surface properties. For quality assurance courses, we implement a Lean Six Sigma DMAIC (Define, Measure, Analyze, Improve, and Control) approach to formulating a strategy and protocol for measurement and analysis of samples. Data for statistical comparisons, such as Gage Reproducibility and Repeatability (GR&R) with different measurement systems

and users, are readily generated. More simple studies, such as correlating surface roughness values between different measurement methods (e.g., laser light scattering vs. surface profiling) serve as realistic case studies in statistical analysis. For our robotics and quality control courses, we adapt these techniques in the context of machine vision in manufacturing. For example, laser light scattering and image capture have been incorporated into a conveyor belt system for silicon wafers, machined metal parts, and 3d-printed parts. Parts meeting surface quality criteria can be sorted from a production lot loaded on the conveyor belt. We focused on three types of samples (machined metal parts, 3d-printed plastics, and silicon wafers in various stages of solar cell fabrication) due to their commercial or technology importance, availability, and richness in variations which can be assessed by the techniques described below.

### **Survey of Surface Characterization Methods**

Commonly used methods of surface characterization in industry and R&D include:

- 1. Atomic Force Microscope (AFM).** An AFM scans the surface with a finely-tipped probe generating a signal proportional to height deviations. Educational versions of these instruments (~\$15,000) are now available. They scan very small areas of the sample, but give a 2-dimensional profile of the surface at very high resolution (< 10 nm). Although AFM measurements can be tedious, a few representative samples can provide a “gold standard” for comparison of other methods.
- 2. White Light Interferometry.** White light interferometry is a non-contact technique for surface height measurement surface profiles ranging between tens of nanometers to several centimeters. These are relatively expensive instruments (~\$40,000), but are often available on campuses and can be used for benchmarking samples and calibration of other methods.
- 3. Surface profiling with a stylus.** These instruments are moderately priced (~\$3000) and are quite commonly used in machine shops. They generate a one-dimensional scan of the surface, and are good for introducing many of the quantitative measures of surface roughness.
- 4. Laser Light Scattering.** These are specialty instruments (\$ several thousand dollars) and work by using a detector array to determine the spatial distribution of light scattered from a laser directed at the sample surface. Smooth surfaces reflect specularly; rough surfaces scatter light. The distribution of scattered light can be correlated with roughness.
- 5. LED light scattering.** These are low cost instruments (~\$300) that measure the diffuse light component of a collimated low-angle incident beam produced by an LED. The diffuse light component is an indicator of surface roughness.
- 6. Glossimeters.** Glossimeters similar to LED light scattering instruments but use multiple LEDs/detector pairs at three incident angles to assess the reflection and specular /diffuse components of reflection. These low cost (~\$300) instruments are commonly used in the paper, paint, and textile industries.
- 7. CCD Camera.** CCD cameras can capture images of surfaces and create 2-dimensional matrices of pixel intensities. Pixel intensity histograms can be correlated with surface roughness.

- 8. Depth gauge profiling.** Digital depth gauges (~\$500) measure the penetration of a probe into surface depressions. Penetration depth can be correlated with surface roughness.
- 9. Fluorometry.** Fluorescence dyes sprayed on the surface can be rinsed off. Surfaces with rough features will retain traces of dye, which can be imaged with low-cost handheld USB fluorescent microscopes (~\$500). These images can be digitized and quantified.
- 10. Thermal infrared images.** Thermal camera images can readily provide a two-dimensional temperature profile ( $\pm 1$  °C). Since the temperature detected is dependent on surface emissivity, the uniformity of the surface geometry can be estimated. Thermal cameras range in price from less than \$1000 to \$15,000, depending on temperature resolution and image size.
- 11. Capillary wetting.** Drops of various liquids (water, alcohol, hexane) on the surface will exhibit a characteristic wetting angle which is a function of surface tensions and is affected by surface roughness and purity. The drop profile and wetting angle can be imaged and quantified with a CCD camera.

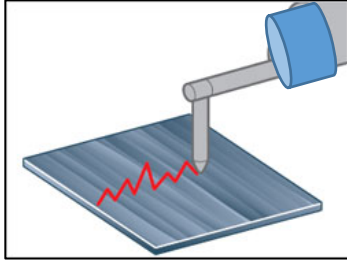
## **Procedures and Laboratory Activities**

Machined surfaces, polycrystalline silicon wafers and 3d-printed parts were used to gather data using different surface roughness measurement techniques. A correlation curve was generated against different practices. These plots were later compared and a linear trend line was created to better understand the paramount surface roughness measurement technique.

The main goal of this report is to distinguish between the different surface roughness measurement techniques. Using different samples, these techniques were compared on the basis of accuracy of readings, time taken to record the data and other miscellaneous factors which include damaging the surface during contact, and the cost of equipment. The different techniques that have been used to measure surface roughness are the stylus profilometer, LED light scatter, Laser Check, Gloss meter, image processing through MATLAB™, surface analyst, white light interferometer and the atomic force microscope.

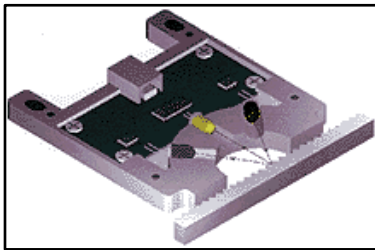
## **Surface Characterization Methods**

The stylus profilometer is a type of surface contact technique that uses a diamond stylus that will horizontally traverse over a surface. It is moderately priced and is one of the most widely used practices. One of the disadvantages of this technique could be that it leaves a scratch on the surface.



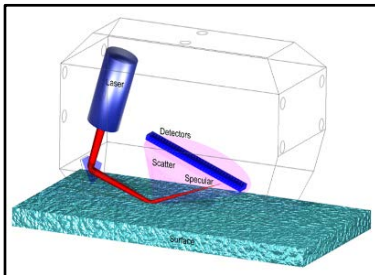
**Figure 1:** Profilometer principle.

The LED light scatter instrument is a non-contact low cost optical technique. When light is flashed, reflection and the scattered light is measured. This data is used to compute the surface roughness. One of the disadvantages of this technique would be that inconsistent readings can be seen when performing this on shiny surfaces.



**Figure 2:** LED light scatter sensor (D522 LED Surface Roughness Sensor, Hohner, Inc.)

The gloss meter is used to measure the specular reflection gloss of a surface. This can be related to the surface roughness. A beam of light is incident on the surface that is being measured and the amount of reflected light at different angles is measured. The area of the surface should be large enough for surface roughness measurement. Inconsistent readings can be seen when performing this on shiny surfaces.



**Figure 3:** Laser Check® (Schmitt Industries)



**Figure 4:** Gloss Meter (M&A Instruments, ETB-0833)

The LaserCheck® works on the same principle as the LED light scatter technique. Instead of using a flash of light, a laser with a particular voltage is projected on the surface and there are twenty detectors that measure the reflected laser voltage. This reflection scatter values give the surface roughness values of the specimen. Inconsistency is seen when the surface has a higher luster value.



**Figure 5:** Atomic Force Microscope (ezAFM, NanoMagnetics).

The atomic force microscope can be used to measure the topography and magnetic features of materials in nanometer scale. This is considered a gold standard for surface roughness measurement of a particular sample. A small cantilever goes over the surface to get accurate readings. The downsides of using this unit is its elevated cost and time needed to take measurements.



**Figure 6:** White Light Interferometer Surface Profiler (Zygo NewView 6000)

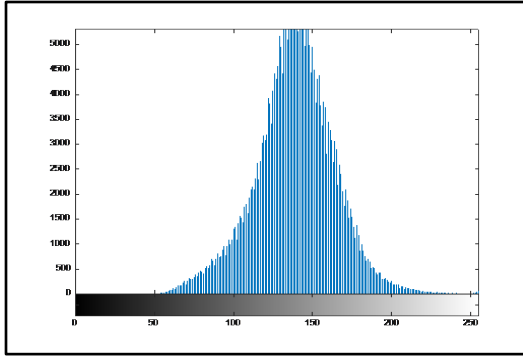
A white light interferometer is a non-contact optical technique that measures the surface height with different surface profiles on 3d structures. The instrument setup is really expensive but is an effective instrument to benchmark samples.

Droplet of water is released onto the sample that is being measured. The surface will demonstrate a wetting angle which is related to surface tensions. This is affected by surface roughness. The droplet on the surface is imaged and the wetting angle is determined.



**Figure 7:** Surface Analyst Wetting Angle Meter (Brighton Industries)

MATLAB™ image processing of surface images can be used to estimate surface roughness values. Once an image is taken, it is converted to grey scale. Grey-level histograms of the image of the surface is created. The standard deviation of this bell curve is calculated and correlates with surface roughness..

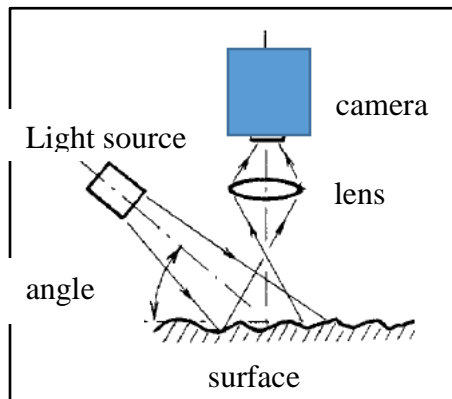


**Figure 8:** Grey-level histogram of surface image.

## Measurements and Discussion

### MATLAB™ Image Processing

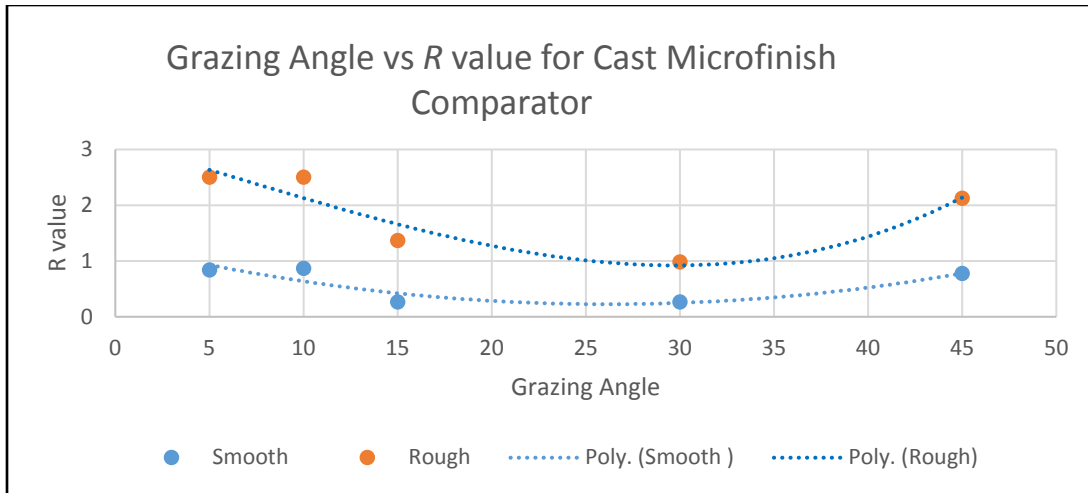
The essential parameter for MATLAB™ image processing is the quality of the image. It is essential that a light source is incident on the surface at an optimal angle from the horizontal. The perfect grazing angle will establish a good quality image with the shadows representing rougher areas of a particular surface. A hand held portable microscope was used to take the images. The figure below shows the setup that was used to get the images.



**Figure 9:** Setup for Image processing

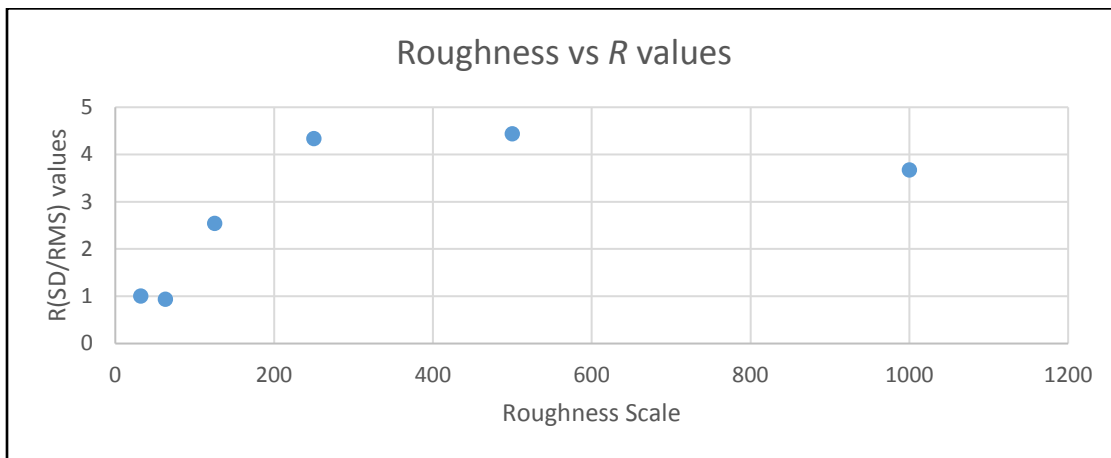
The  $R$  (standard deviation/root mean square) value is computed<sup>[1]</sup>. At first, a cast micro-finish comparator was used to generate the plot for the angle of illumination with  $R$  values using MATLAB™.





**Figure 10:** Computing the ideal grazing angle.

The largest difference in the  $R$  values came when the grazing angle was small. For best sensitivity for the system, the ideal grazing angle for other experiments was chosen to be five degrees. The same comparator was used to get the relationship between the optical roughness parameter  $R$  with the RMS value specified in the specimen. It can be seen that as the roughness of the surface increases the  $R$  (optical parameter) also increases. This proves that  $R$  value from image processing can be an effective technique to measure the surface roughness. When a shot blasting comparator was used to generate the same relation between the  $R$  value and the roughness scale value specified in the specimen, a similar trend like above was observed.



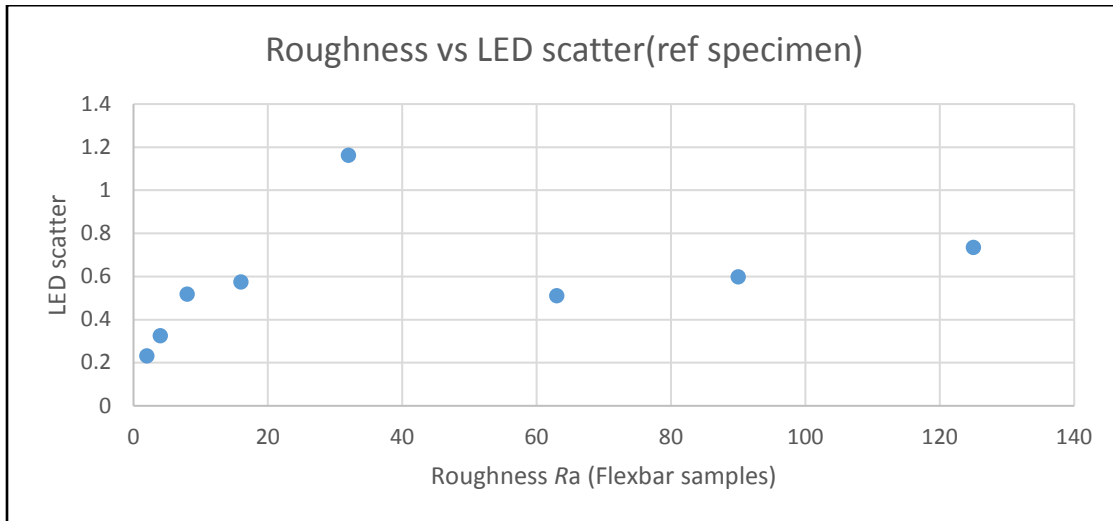
**Figure 11:** Roughness Scale vs  $R$  value for machined sample.

Although, after a certain roughness scale measurement, the trend seems to be inconsistent. As the roughness scale value goes beyond 500, the  $R$  parameter starts to decrease and shows an inversely proportional relation. This shows that the image processing technique could be only effective to a particular range of surface roughness. In this particular range of specimen, this technique could be used to distinguish between different surfaces.

A direct proportion is observed in all cases which shows that as a rougher specimen is used, higher values of  $R$  are observed. This shows that the image processing is a good measurement practice and is good to measure plastic parts and machined parts.

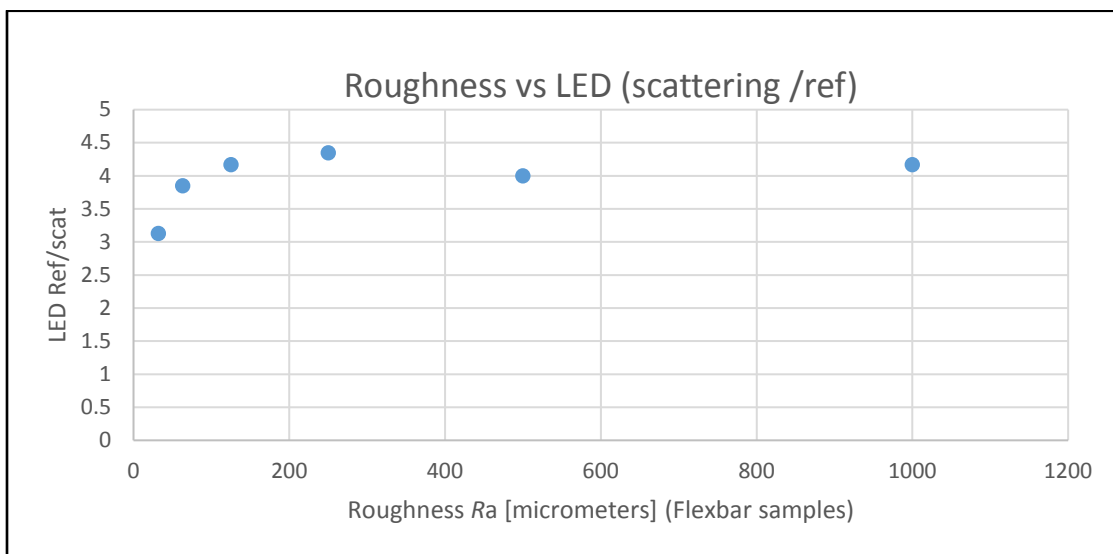
### Surface Roughness by LED Light Scattering

The M-15 visual tactual reference specimens (Flexbar, Inc) are used to establish a relationship between the LED light scatter value and the roughness values specified on the sample.  $R_a$  values are in micrometers.



**Figure 12:** Roughness Scale vs LED light scatter for reference machined samples.

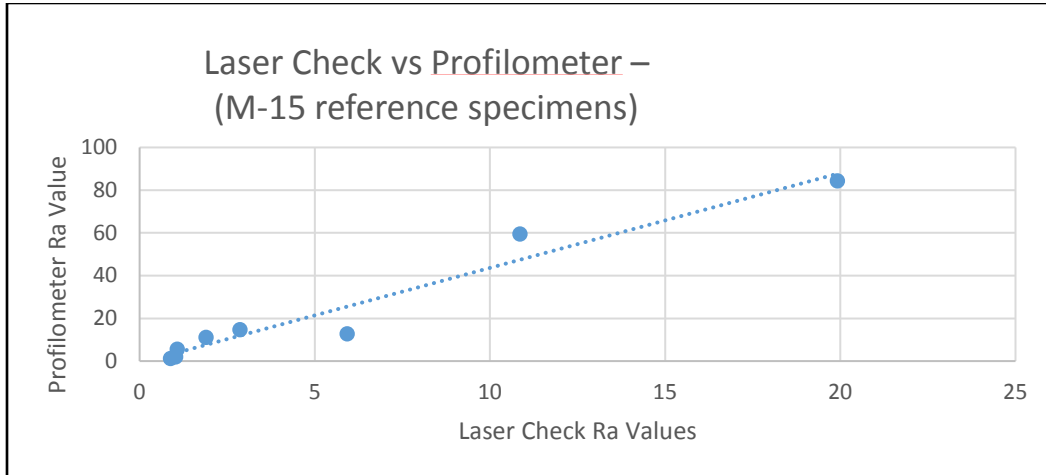
It can be seen that as the roughness of the surface increases, the LED light value also increases with some discrepancies. A linear relation was observed when the shot blasting comparator was used to get the relationship between the LED light scatter value and the roughness values specified on the sample.



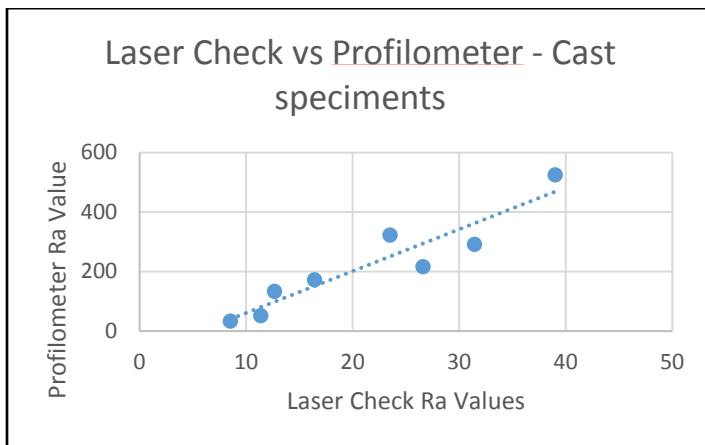
**Figure 13:** Roughness vs LED for machined sample

## Surface Roughness by Laser Light Scattering

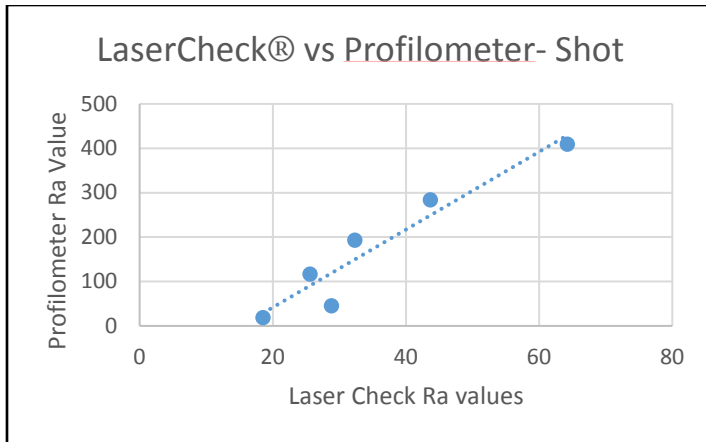
Using M-15 visual tactual reference specimen, cast and shot surfaces are tested to find a relationship between the laser scattering values and the stylus profilometer values. As we deemed the profilometer as a fairly accurate in these roughness measurements, it was used to compare with the laser scattering data. Different machined samples were tested to record the data for the following processes.



**Figure 14:** Lasercheck® vs profilometer for M-15 machined specimen



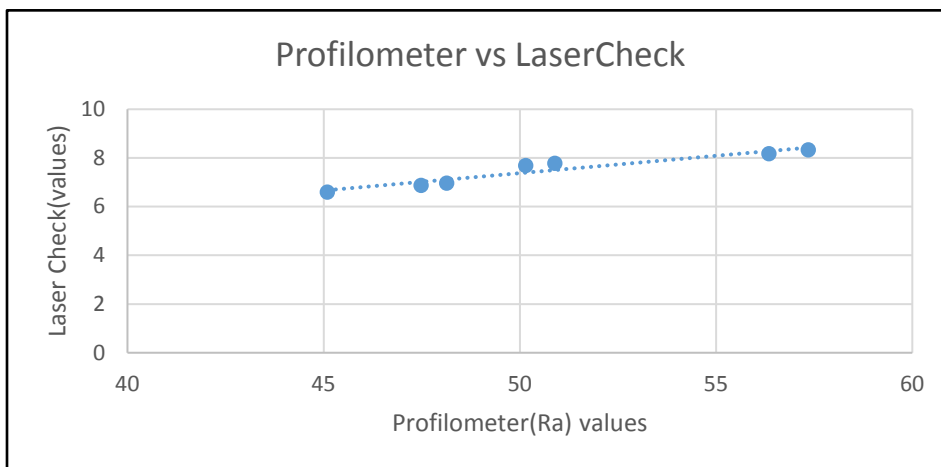
**Figure 15:** Lasercheck® vs profilometer for cast finish



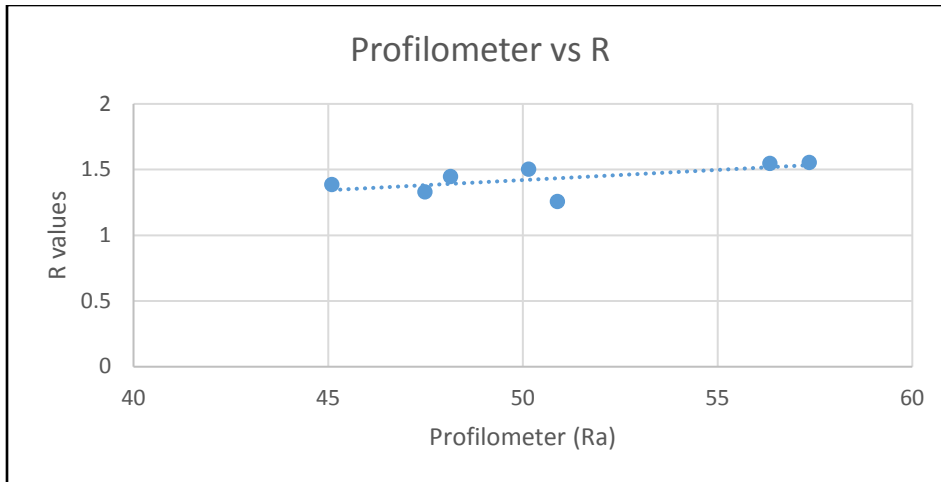
**Figure 16:** Laser check vs profilometer for shot finish

The plots show that laser check and the profilometer values are directly proportional to each other. As the profilometer values increase, the laser check also gives incremental values. This proves that the laser check can be an effective technique to distinguish between smooth and rough values.

Seven different samples of polycrystalline silicon wafers were used to categorize the different surface roughness techniques. At first, the laser check and the profilometer were used to generate the surface roughness values. These values were then compared to the R value from the image processing tool from MATLAB.



**Figure 17:** Profilometer vs Laser Check for polycrystalline wafers

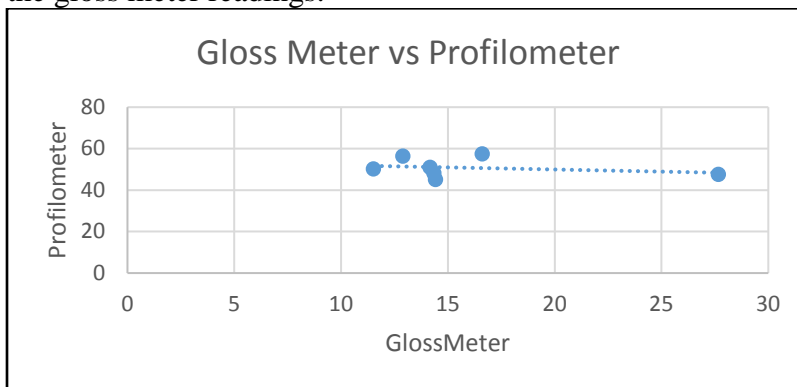


**Figure 18:** Profilometer vs  $R$  values for polycrystalline wafers

Both the plots above showed a linear trend line. Profilometer is considered an accurate way of measuring the surface roughness. When a rougher sample was taken, higher  $R$  values were recorded. This proves that the image processing tool could be an effective practice to measure the roughness of a particular surface. A similar trend was observed when the profilometer readings were compared to the laser check readings. When a rougher surface was used to gather data, a higher laser check value was recorded. This showed us that the laser check and image processing give fairly consistent data when compared to the profilometer.

### Gloss Meter

The next step was to compare the gloss meter readings with that of the profilometer, laser check and the  $R$  values from the image processing. An inversely proportional trend was observed with the gloss meter readings.

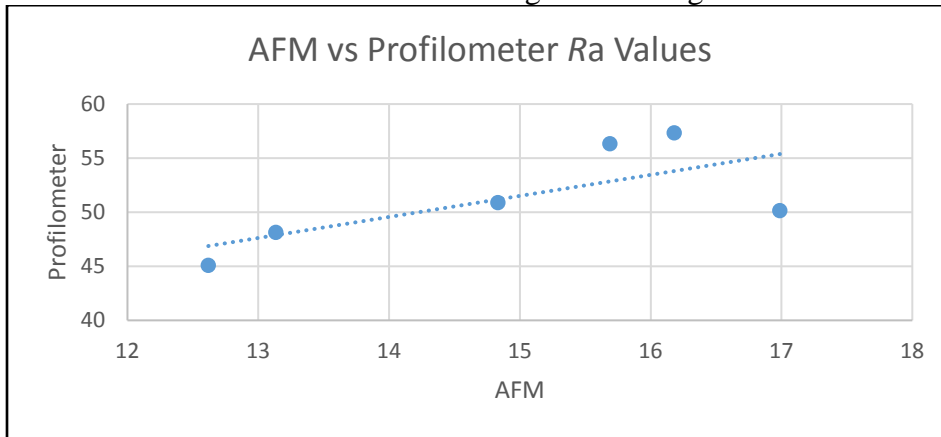


**Figure 19:** Gloss Meter vs Profilometer for polycrystalline wafers.

The gloss meter showed a weak inverse proportionality to roughness as measured by the surface profilometer, and there was also significant scatter in the measurements. This showed that gloss meter is not a very effective technique to measure the surface roughness for the type of samples studied.

## Atomic Force Microscope (AFM) Measurements

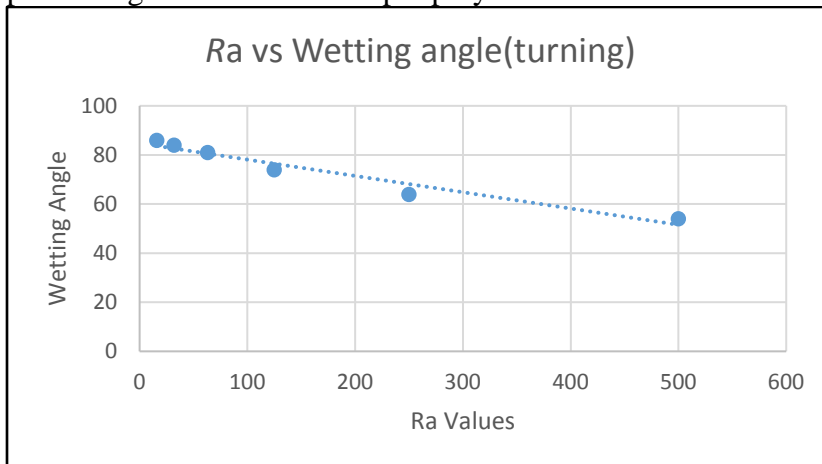
The atomic force microscope was used to collect the surface roughness data from these polycrystalline silicon wafers. The AFM values with the profilometer values were plotted against each other which lead to a directly proportional linear trend. This shows that as the surface is rougher, higher roughness values from AFM are observed. Due to its consistency in the data points when compared to profilometer, it can be said the atomic force microscope is a good instrument to benchmark the surface roughness as a 'gold standard' to compare other techniques.



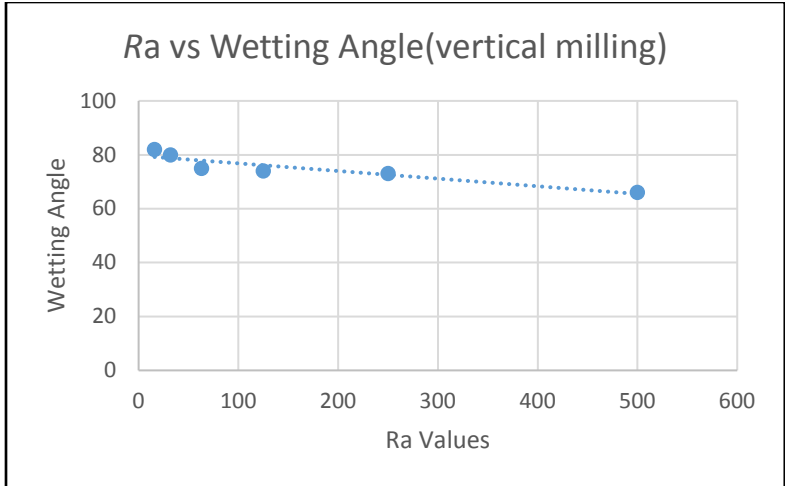
**Figure 20:** AFM vs Profilometer Polycrystalline wafers

## Surface Analyst (Wetting Angles)

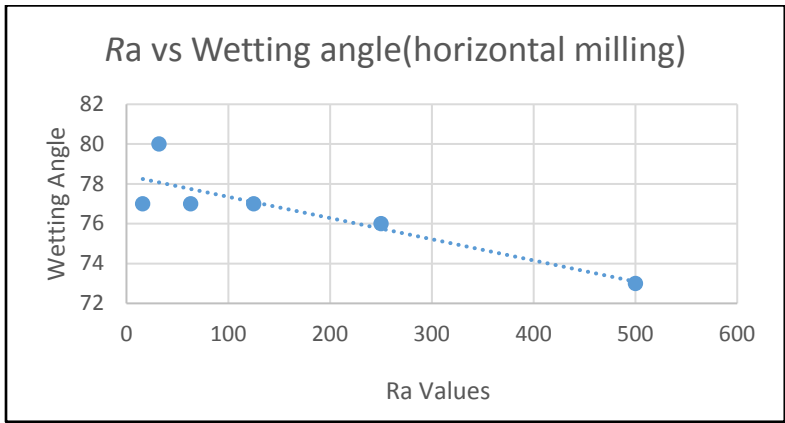
The SurfaceAnalyst® is a handheld instrument that measures wetting angles by digital image processing of water microdrops sprayed on a surface.



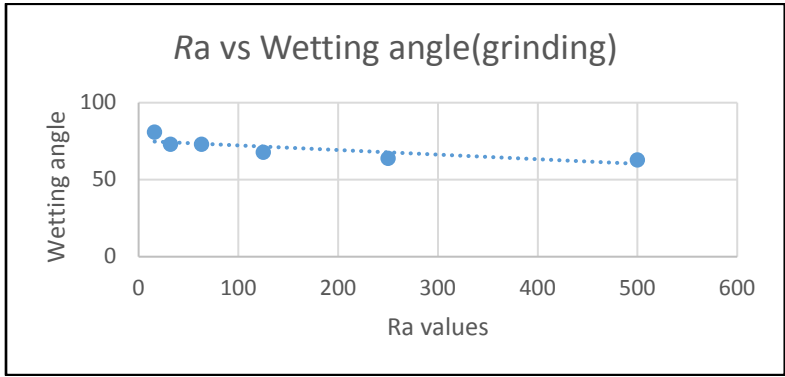
**Figure 21:** Ra values vs wetting angle for turning



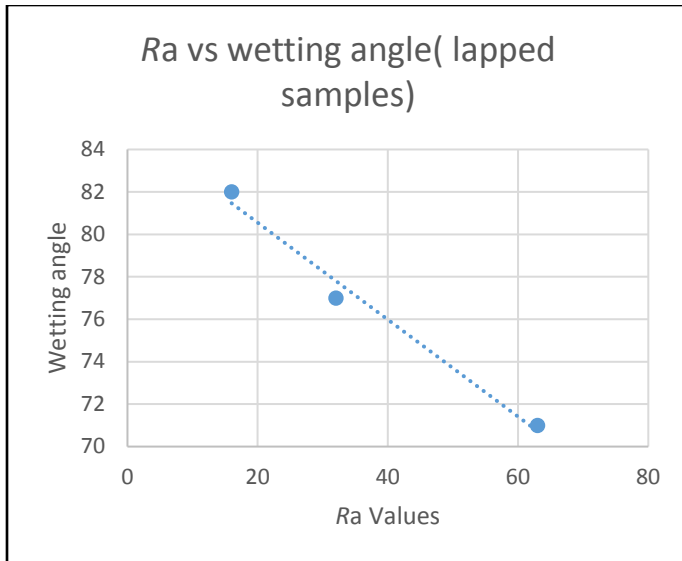
**Figure 22:** Ra values vs wetting angle for vertical milling



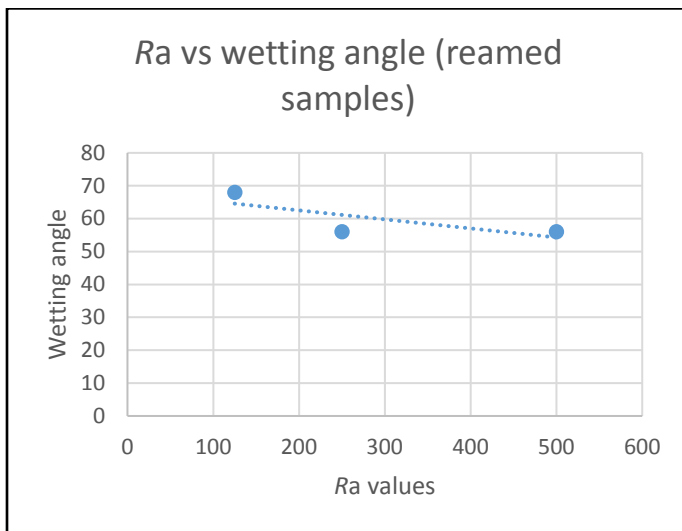
**Figure 23:** Ra values vs wetting angle for horizontal milling



**Figure 24:** Ra values vs wetting angle for grinding.



**Figure 25:** Ra values vs wetting angle for flat lapping



**Figure 26:** Ra values vs wetting angle for reaming

The surface analyst wetting angle instrument has been used to generate the above plots. The wetting angle data was plotted with the arithmetic mean roughness values for different manufacturing processes. It could be seen from the plots that all the plots are inversely proportional. As the Ra values increase the wetting angle decreases. So as the wetting angle increases, the surface is smoother. The wetting angle approach worked consistently for machined parts. Unreliable data was observed when wetting angle practice was implemented on polycrystalline wafers and 3d printed samples.



## Summary and Discussion

These laboratory modules have been successfully incorporated into the several Engineering technology courses. The laboratory exercises demonstrated high reliability for generating meaningful and consistent data for students to perform useful analyses of surface roughness. In addition to introducing students to concepts of surface characteristics and measurements, students gained experience in the approaches to metrology and the nature of surfaces with respect to optical and chemical properties. We believe the main educational utility of this approach is the feasibility of characterizing a material by many diverse techniques, and letting students reconcile differences between methodologies, as well as gain an appreciation for ideas relating to benchmarking, gold standards and references, corroborating and complementing studies, and assessing techniques for information value, ease of use, cost, and throughput. The fact that we were able to measure and characterize samples by six or more different techniques provides almost unique opportunities for teaching fundamental concepts of measurements and metrology to students.

We have described and validated a suite of laboratory modules which can be selected and utilized for educational purposes in a wide range of engineering courses. The modules give students hands on experience with simple and more sophisticated instrumentation for optical, mechanical, and chemical characterization of surfaces. Future work will expand the range of materials, include white light interferometry, continue robotics implementation for more of the methods, and include adding sandblasting, coatings, and solvent treatments to evaluate methods of improving surface characteristics. We are investigating the application of new nanomaterial hydrophobic coatings, fluorescence evaluation of cleaning steps, and correlating solar cell performance with surface roughness of silicon. In summary, this report serves to draw attention of educators to the fertile and important areas of surface characterization for low-cost, instructive, and commercially relevant experimentation for engineering, science, and technology students.

## References

1. F. Luk, W. North, V Huynh [1989]. "Measurement of surface roughness by a mission vision system" *J. Physics E: Scientific Instruments* **22**, 12 977-980.
2. Mauk, M. G., & Chiou, R., & Bayas, C. R. (2016, June), *Machine Vision for Solar Cell Inspection* Paper presented at 2016 ASEE Annual Conference & Exposition, New Orleans, Louisiana. 10.18260/p.27325