

# Surface Metrology and the National Science Foundation

An Interactive Qualifying Project  
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by

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## **Abstract**

This project evaluates the use of Surface Metrology—the study of surface texture—in research publications funded by the National Science Foundation. It involves a critique on these research publications based on how they characterize surfaces towards fulfilling their research objectives. By conducting a comparative analysis of various measurement instruments and parameters, this project seeks to improve the use of Surface Metrology in research and industry.

## **1. Introduction**

### **1.1. Objective**

The objective of this project is to promote the use of better Surface Metrology techniques in industry and NSF funded research. There is a high likelihood that the NSF is not funding advanced Surface Metrology methods (Brown, 2014). As part of this project, this proposition will be investigated in related research publications.

### **1.2. Rationale**

Controlling surface quality is an important aspect of the manufacturing process because surfaces are the starting point of several engineering component failures, including wear, cracking, and corrosion. The desired performance of manufactured parts related to functions, such as, friction, cohesion, and adhesion, is also dependent on surface quality. (Blunt et al., 2005)

Predicting the mechanical behavior of machined parts with different types of surfaces can help select the right type of surface finish (rougher surfaces, for instance are desired for better adhesion). Finding a way to quantify surface texture is therefore important in order to find correlations with the abovementioned functions and, thereby, evaluate how a surface influences a part's performance.

Surface Metrology—the study of surface texture—presently encompasses a variety of instruments, techniques and measurement parameters. However, many quality control professionals to date have largely used the same Surface Metrology technique since the 1940s, which the conception of the field can be traced back to (Mathia et al., 2011).

Using traditional methods of studying surfaces (such as Ra-average roughness), which only account for height information at a single scale of measurement, is insufficient to find reliable functional correlations (Berglund et al., 2010). Therefore, evaluating funded projects is important to create an informed comparative platform by which researchers and the NSF can improve their use of Surface Metrology.

### **1.3. State-of-the-art**

There are two aspects of this project—to evaluate funded science publications and to conduct a comparative analysis on various Surface Metrology techniques.

An evaluation on the methods used in funded research has been conducted by Ioannidis (2014). The report highlighted some prevalent shortcomings in funded research, including statistical errors and non-repeatability of findings. Primarily focusing on the medical sciences, the author made proposals on how current research methods could be redesigned in order to produce better results and make the most out of research resources, which mostly come from the funding agency National Institutes of Health (NIH).

Vorburger et al. (2007) have made comparative studies on various surface metrology techniques including stylus profilometry, optical profilometry, and interferometry. By measuring the same surface using different instruments, the researchers studied discrepancies between roughness results. In addition, variations between results obtained from 2D and 3D measurement parameters were also analyzed. Their analysis found that, for nanoscales, there were significant discrepancies of up to 75% between 2D and 3D measurement results obtained from optical and stylus profilometers, respectively. The measurement results for 2D measurement parameters showed reasonable

agreement across different measurement instruments. Phase-shifting interferometry and stylus profilometry yielded similar average roughness (Ra) results, which is a 2D parameter.

Bergland et al. (2010) have traced variations between 32 different Surface Metrology parameters when correlating friction with surface topography. Their study showed that the functional correlation between friction and surface topography is dependent on the parameter used, as well as, the scale of measurement. For example, several classes of conventional parameters (including height, spatial, and material ratio parameters) did not correlate well with friction. On the other hand, 3D hybrid parameters, such as, Sdq (the root mean square surface slope) and Sdr (the developed surface area ratio) correlated well with friction.

#### **1.4. Approach**

This project will conduct a comparative study in order to evaluate the use of surface metrology in NSF funded projects. Critiques on funded research projects, like that of Ioannidis (2014), have mostly focused on general aspects of research practice such as statistical errors and the influence of scientific bias. It doesn't have a critique on the technology used. Multi-scale analysis—the study of surface texture as a function of measurement scale—will be included in our comparative study. While there are several comparative studies on the subject, multi-scale analysis has not been used as an additional perspective to aid comparisons.

## 2. Background

### 2.1. National Science Foundation

The National Science foundation is the US government agency primarily responsible for funding research in colleges and universities. Using financial grants, which secure resources for research, the organization incentivizes research in various fields of study. Close to a quarter of all research proposals (11,000 out of 40,000 per year on average) receive funding (NSF, 2013).

All R & D expenditures are in the upwards of \$60 billion dollars, of which \$40 billion accounts for basic research. Total federal funding for Mechanical Engineering is around \$2 billion. Surface Metrology is within the category of Mechanical and Metallurgical Engineering. WPI has also been a significant recipient of NSF grants for research and development. Its rank in the amount of budget received has been on average 220<sup>th</sup> out of all US institutions and 71<sup>st</sup> out of private institutions in the US. The NSF Statistics for WPI are tabulated below.

Table 1: NSF Funding and Expenditure Statistics for WPI (NSF, 2013)

Data year	Total federal obligations			Total R&D expenditures		
	Rank	Percentile	Institutions ranked	Ranks	Percentile	Institutions ranked
2013				243	38.3	643
2012	211	20.5	1067	269	41.9	649
2011	231	21.2	1127	265	29.8	910
2010	247	21.2	1209	272	37.3	741
2009	253	22.2	1177	269	38.6	707
2008	246	22.2	1146	265	39	689
2007	251	21.4	1213	268	40.6	668
2006	261	22.1	1219	259	39.9	657
2005	281	23.8	1216	263	41	650
2004	306	25.4	1237	263	42.1	632
2003	311	27.7	1149			

The main recipients of NSF grants are public and private academic institutions. All R&D projects that are undertaken by universities and colleges draw nearly 70% of their funds from the

federal budget. The other contributors to R&D include the State administration, academic institutions themselves, and industry.

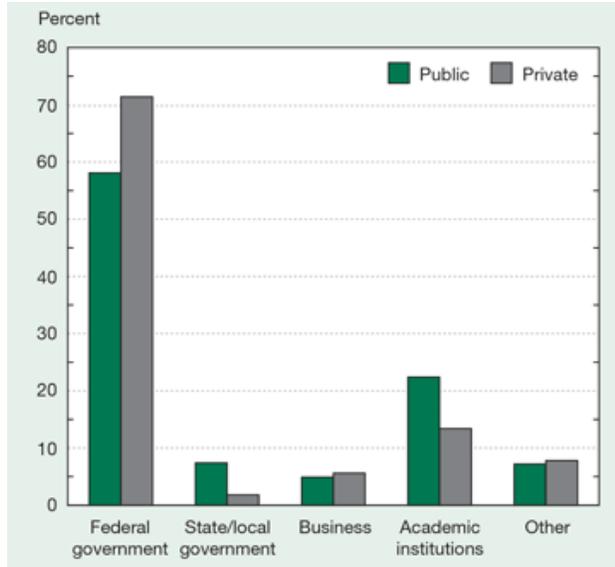


Figure 1: Sources of Science and Engineering funding for public and private academic institutions (NSF, 2013)

As a relatively new field, there is limited statistical data on how much funding has been granted specifically for Surface Metrology research. However, there are some NSF funded projects that make use of Surface Metrology with other research goals in mind. The focus of this project was to analyze these research projects and the respective techniques used to inspect surface roughness.

## 2.2. Stylus Profilometry

Stylus profilometry is the most widely used Surface Metrology technique to date. A stylus tip is linearly moved over the sample and the electronically recorded vertical displacements create the roughness profile.

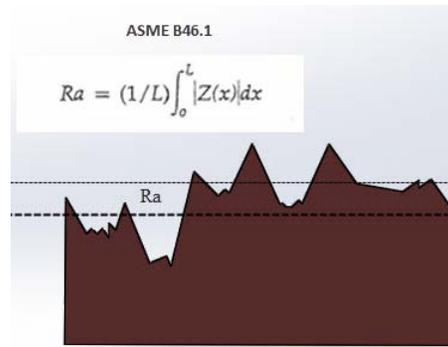


Figure 2: Average Roughness (Ra)

There must be direct contact between the tip and the surface in order to generate the profile, whereupon the profile is quantified using 2 dimensional roughness parameters. These parameters have been standardized by ISO 4287 and ASME B46.1. As part of this project, the roughness parameters of these two standards have been tabulated (see Appendix A). The most commonly used parameter is the average roughness value Ra. As defined in ASME B46.1, the Ra is the arithmetic average of the absolute values of profile deviations within the evaluation length (L).

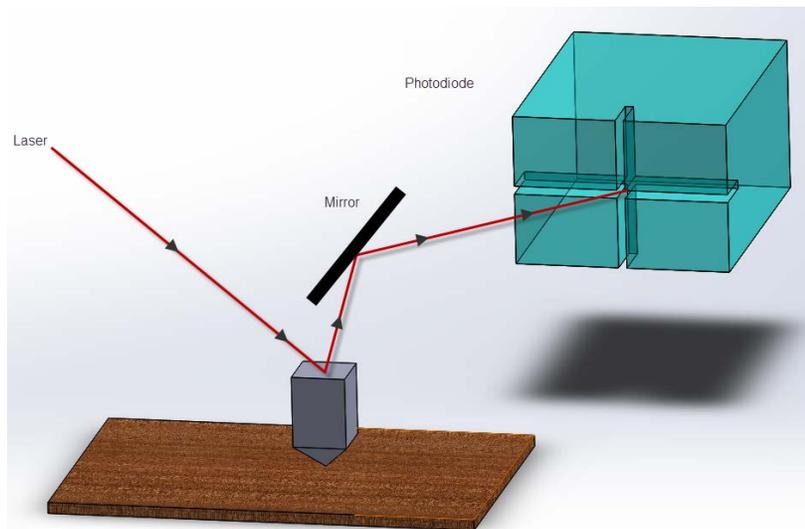
Since this technique was one of the earliest roughness analysis techniques, accumulated industrial data consists primarily of 2D parameters. It is currently the least demanding method for acquiring measurements in terms of cost. The self-reliance of 2D measuring instruments is also another reason why they are still widely used in industry. They can easily be built into manufacturing machines for immediate surface quality inspection. (Frade et al., 2013)

### 2.2.1. Atomic Force Microscopy

AFM is treated as a branch of stylus profilometry because it utilizes a similar principle. A cantilever, where the tip is located, scans the surface and generates the roughness profile. The tip

however, is much smaller than that of Stylus Profilometry, with its diameter often less than 100 nanometers. AFM can therefore capture much smaller features on the surface (Capella et al., 2005).

Atomic force makes the cantilever bend so as not to damage the surface by imposing an unaltered downward force. The vertical motion of the cantilever is captured by a photodiode. The basic schematic is shown below.



*Figure 3: Simplified Schematic of AFM*

There are three AFM methods for acquiring surface roughness: contact mode, non-contact mode and tapping mode. In contact mode, the tip is dragged over the surface with the cantilever deflection kept constant. Here, the process is influenced by frictional and adhesive forces, and the images generated may, therefore, be distorted.

In non-contact mode, the Van der Waals forces are utilized to generate the profile. Using a certain resonance, the cantilever is hovered (while oscillating) nanometers above the surface. These forces decrease the frequency of this resonance which can be derived into information about the surface texture. Highest peaks would cause the highest reduction in the frequency and vice versa for lower peaks.

In tapping mode, a lower amplitude of resonance is used. This characteristic gives the tapping mode a higher frequency than the non-contact method. The surface is contacted (tapped) at small intervals, thereby avoiding the effects of friction and adhesion. This method is often seen as a combination of contact and non-contact, making it the more optimal technique for conducting AFM. Its high lateral resolution mean is ideal for measuring sub-micron features.

## **2.3. Optical Profilometry**

### **2.3.1. Confocal Microscopy**

Confocal microscopy is a non-contact Surface Metrology technique that uses a light source and focusing numerical apertures to generate 3D surface profiles (Bezák et al., 2013). The image is generated using photomultipliers. The aforementioned apertures are used to focus the microscope in relation to the z-direction range of the surface. A light source, typically laser, passes through the apertures and the varying signal intensities generate a 3D surface profile as a large accumulation of statistical data points.

The major advantage of confocal microscopes is the fact that no contact with the surface is required to create the 3D profile. This characteristic makes confocal microscopes non-destructive to the surface. The images can also be generated at much higher speeds than in the case of stylus profilers. 3D roughness parameters have been standardized in ISO 25178 and ASME B46.1 (See Appendix A).

Using the OLS400 microscope, which is available in WPI's Surface Metrology Laboratory, the differences between surface characteristics can be closely examined. Figure 5 shows three surfaces

of metal foils and their surface roughness values as quantified with 3D ISO standards. The area studied is  $250 \mu\text{m}^2$ . The average z-axis range is  $3 \mu\text{m}$ .

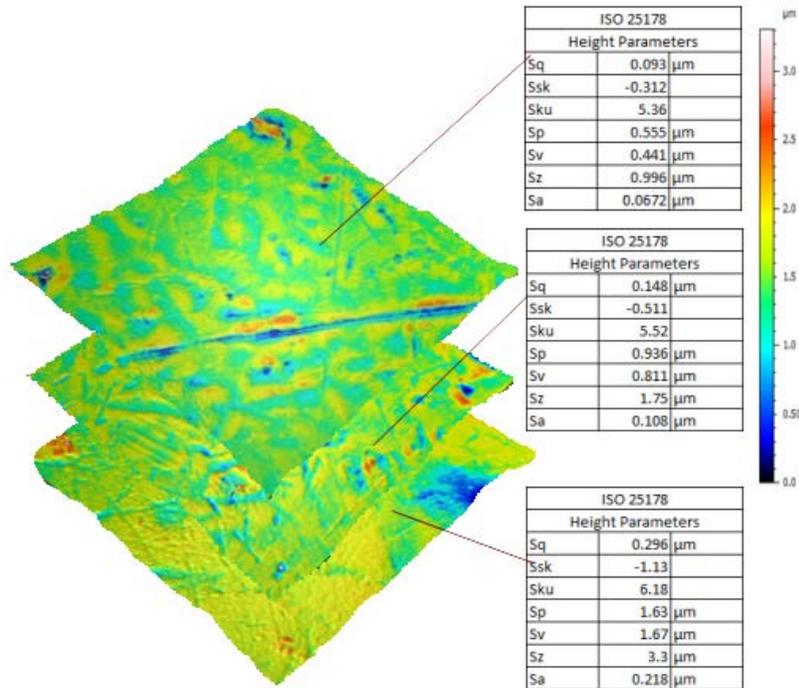


Figure 4: Surface Texture (with 3D ISO parameters)

The ability to obtaining 3D surface maps from surfaces tends to stretch the limits of possible experiments. Any object of inquiry may be placed under a confocal microscope for unique case studies. Figures 6 and 7 show surface profiles taken from banknotes.

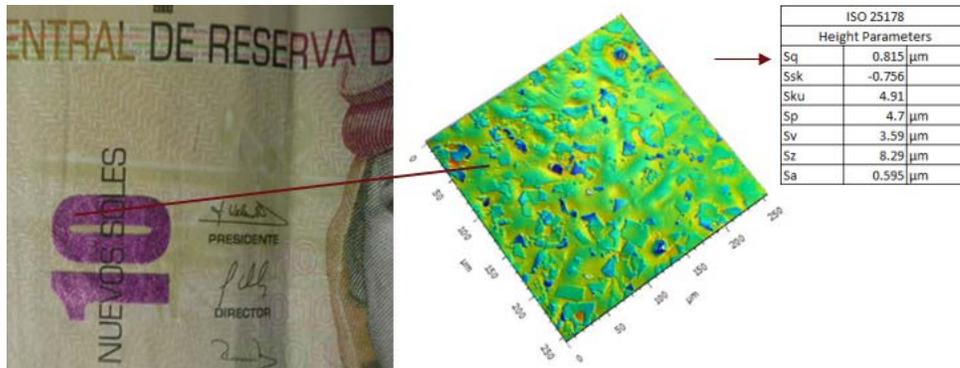


Figure 5: Peruvian Currency 3D surface Profile

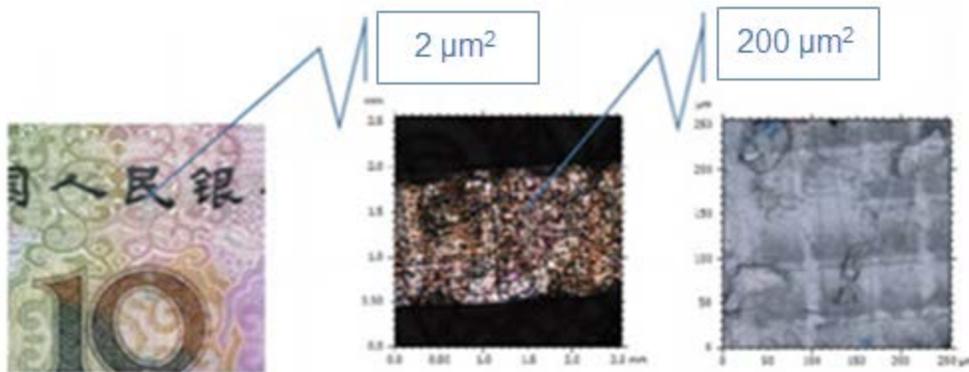


Figure 6: Magnification on security thread on banknote

### 2.3.2. Scale-Based Analysis

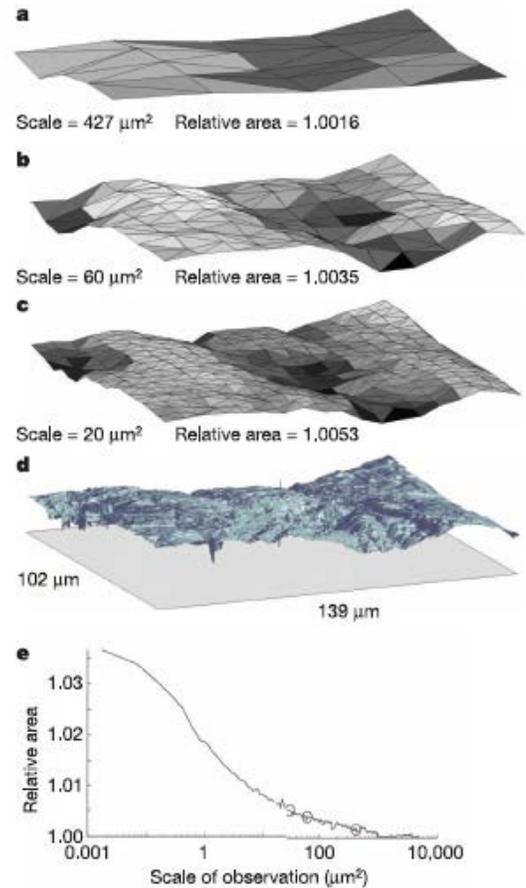
Despite the availability of several roughness analysis methods, there still remains no single fixed way to quantify surface texture. One aspect that hasn't been addressed in several Surface Metrology techniques is the roughness value as a function of time. Benoit Mandelbrot (2006) discovered that surface dimensions have fractal properties, and are therefore, dependent on the scale of measurement. WPI's surface metrology laboratory primarily focuses research on area-scale fractal analysis, which yields surface roughness results as a function of varying scales of measurement (Brown et al., 2003).

Two metal foil samples are used to demonstrate the process of making a scale-based analysis. The microscope used for taking measurements is the Olympus LEXT laser scanning microscope. The measurements taken with the microscope are extracted in the form of surface topography maps.

The extraction of surface profiles is carried out using the Mountains Map software. This software allows a variety of operations to analyze the surface roughness. 2D roughness profiles can also be extracted at any chosen line of measurement on the surface. Outliers that distort statistical data can be removed using an outlier removal operation in the software. There is, however, a separate outlier filter developed by Le Goic et al. (2012), which performs the task more reliably and in a shorter operational time. The filtered

surface is then returned to Mountains Map for a *thresholding* operation—image segmentation to set upper and lower limits. This final operation is especially important to ensure that the areas where outliers have previously been removed doesn't affect the final results. The surface, after filtering and threshold operations, will be ready for further statistical analysis.

In WPI's Surface Metrology laboratory, area-scale and length scale analyses can be carried out using the statistical analysis software, Sfrax. The software can perform detailed peak-to-peak



**Scale-sensitive fractal analysis.** a–e, Relative area is calculated by dividing the area of a surface, calculated using triangles of a given scale in a virtual tiling algorithm (a, b, c), by the projected area of the surface (d). Relative area can then be plotted against scale in a log–log plot (e).  $Asfc^{30}$  is a scale-sensitive measure of roughness and is the slope of the steepest part of the curve fitted to the plot of relative area over scale, multiplied by  $-1,000$ .

Figure 7: Relative Area illustration (Brown et al., 2003)

and filling analyses on measured surfaces and profiles. The relative area of a surface is calculated in terms of virtual triangles (see Figure 8). Upon laying down larger sized triangles on the surface, the individual triangle areas are then added to obtain area of the whole surface. In order to account for smaller features on the surface, the size of the triangles are consequently decreased, whereupon their collective areas are evaluated. Therefore, by decreasing the size of the virtual triangles, we can obtain the area of the surface at varying scales. This type of analysis is known as a scale-based fractal analysis.

Another aspect of scale-based fractal analysis is area complexity, which is the first derivative of the relative area. Calculating complexity is important with decreasing scales of measurements, to avoid the larger features' data points interfering with that of the small scale features. This aspect makes complexity a more reliable measurement parameter to conduct scale-based fractal analysis. This software is provided by our laboratory's partner company, Surfract.

The F-test is a statistical algorithm that can be used to clearly identify where the data points being showing differences. A horizontal line in F-test results typically shows where there data points begin to diverge. From this information, it can be inferred that, at points above a certain designated scale, the surfaces cannot be told apart. But at scales lower than that scale, the surfaces can be told apart. A user interface allows the user to set a certain confidence level in which the F-test can distinguish the surfaces. Figure 9 shows F-test results for two metal foil samples and the appearance of the surfaces before and after the decisive scale ( $100\mu\text{m}^2$  in this case).

The relative Area graphs (figure 9) show that the data points on the metal foil samples' surfaces begin to diverge at the scale of  $100\mu\text{m}^2$ .

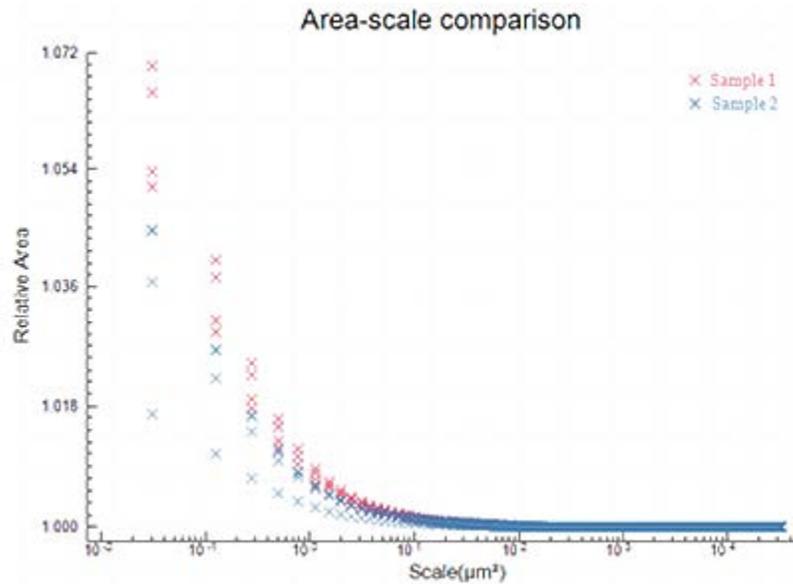
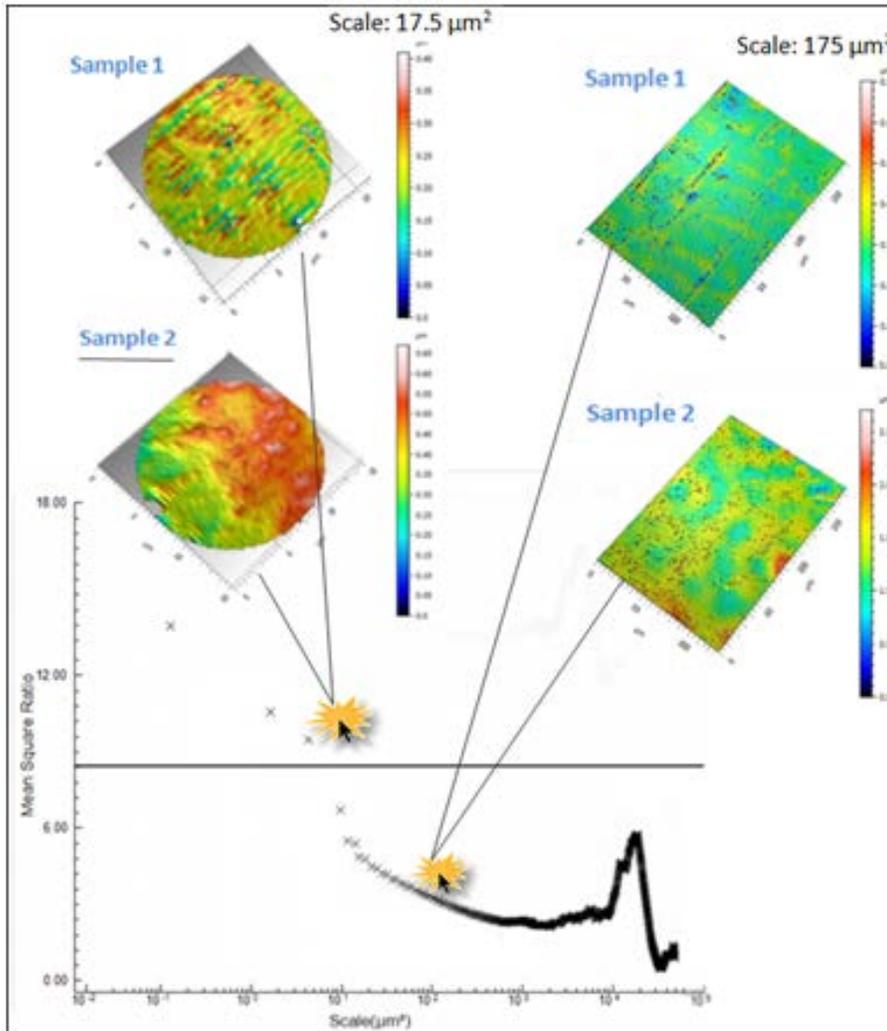


Figure 8: Relative-Area vs Scale graph (Metal foil sample 1 vs 2)

The information we can get from this F-test graph is that, at points below this cut-off scale, there would be clear differences in the surfaces. In order to demonstrate this tendency, the surface maps of two metal-foil samples were extracted. By taking measurements at a larger scale (175 μm<sup>2</sup>) and a smaller scale afterwards (17.5 μm<sup>2</sup>), we can observe the differences visually with between the respective 3D images. The horizontal line represents this cut-off. In Samples 1 and 2 at 175 μm<sup>2</sup> (i.e. under the line), the differences between them are not clear. At a scale of 17.5 μm<sup>2</sup>, however, the differences are much more noticeable with a texture directionality in Sample 1 and the more free-form type texture of Sample 2.



**F-test:** This statistical analysis was carried out by taking four measurements on the metal-foil samples. Extracted from Sfrax, points above the horizontal line indicate scales at which the sample surfaces can be told apart. The x-axis represents the scale of measurement and the y-axis is the mean square. 3D images are shown for the samples below and above the horizontal line of discrimination.

Figure 9: F-test Illustration

## 2.4. Computer Simulation

If actual measurement of the surface is somehow unfeasible, then the surface can be generated using a computer. There are several properties that are affected by the surface

characteristics of an entity. However, it is not always possible to measure the surface, especially if the focus is on the chemical and elemental level. Although making computer models for physical entities is not a new practice, limited work has been done in modeling surface roughness. There are, however, some NSF funded projects that deal with computer simulated Surface Metrology. Industrial demand for computer simulated Surface Metrology is so-far limited which restricts the practice to research applications only.

The major issue with this type of analysis is the fact that the subject is completely isolated from real world micro-elements, which would be difficult to account for during simulation. For macro-scale studies, the factors involved, such as wind, can more reasonably be accounted for during simulations. However, there are several micro-scale elements that do not have standardized simulation techniques making the error factor more significant. Nevertheless, this technique offers a new approach to studying surface metrology.

If the abovementioned elements are reproduced at the micro and macro-scales, then it may be possible to design surface roughness characteristics for the most optimal levels of a desired property. For instance, if simulations show that a certain texture type maximizes adhesion, then quality professionals can produce a product guided by the information obtained from the simulations. So far, however, most of these correlations are studied experimentally.

### **3. Methods**

There were two aspects of the methods used to complete this project: the research phase and the analysis phase. The research phase involved finding NSF funded projects related to Surface Metrology, whereas the analysis phase involved summarizing and critiquing the publications based on their use of Surface Metrology.

### 3.1. Research Phase

This section was the more important and, consequently, the more time-consuming aspect of this project. The first task was to identify the general use of Surface Metrology in available scientific publications. This helped establish a basis by which our target publications (NSF funded projects that involve Surface Metrology) could be compiled before further analysis. The objective of this phase was to find the best keyword combinations, the most relevant scientific databases, and researchers who have conducted several studies on the Surface Metrology.

The next step was to narrow down the search to NSF funded projects. This additional parameter further constrained the search and made it difficult to obtain a broad selection of publications. Overall, around 13 of the most relevant research publications were eventually compiled, 5 of which were selected for further analysis.

The breakdown of the research phase is as follows:

- Keywords used: Surface, Average, Roughness, Texture, Stylus, Confocal, Topography, Characteristics, Fractal, Areal-scale, Relative Area, Interferometry, Phase, Shifting, White, Light, and Characterization.
- Databases Interrogated: Mainly the *Web of Science*, *Research Gate*, and *Google Scholar*. These online databases offer a comprehensive selection of search parameters. The *Web of Science* website was found to be the most optimal for our study. In addition to its rigorous keyword matching, the website also has a search refining option, which, when selected, returns projects that were funded by a specific organization. In our case, the searches were narrowed down using the "NSF" option.

Each of these research projects used Surface Metrology to achieve a certain overarching objective, which primarily included the optimization of surface finish and the pursuit of correlations with certain functions. The measurement techniques and the type of surface analysis conducted in each research publication will be described in the analysis phase, along with the researchers' summaries.

### **3.2. Analysis Phase**

In this phase, the research publications were summarized. This was followed by identifying the use of Surface Metrology in these publications and its relation to the research objectives. Once an overall breakdown is complete—that is, when research goals and the use surface metrology have been identified—we then continue to analyze the effectiveness of the techniques chosen by the researchers. The general proposition that this project seeks to investigate, as introduced earlier, is that the NSF does not fund advanced Surface Metrology techniques. For instance, some researchers have characterized an anisotropic (uneven) surface using only a one-dimensional roughness parameter, which can vastly misrepresent the surface. This project highlights such instances and, by using lab-resources alongside available literature, build a critique on NSF funded publications.

The methods used in this project can be summarized in the following flow chart:

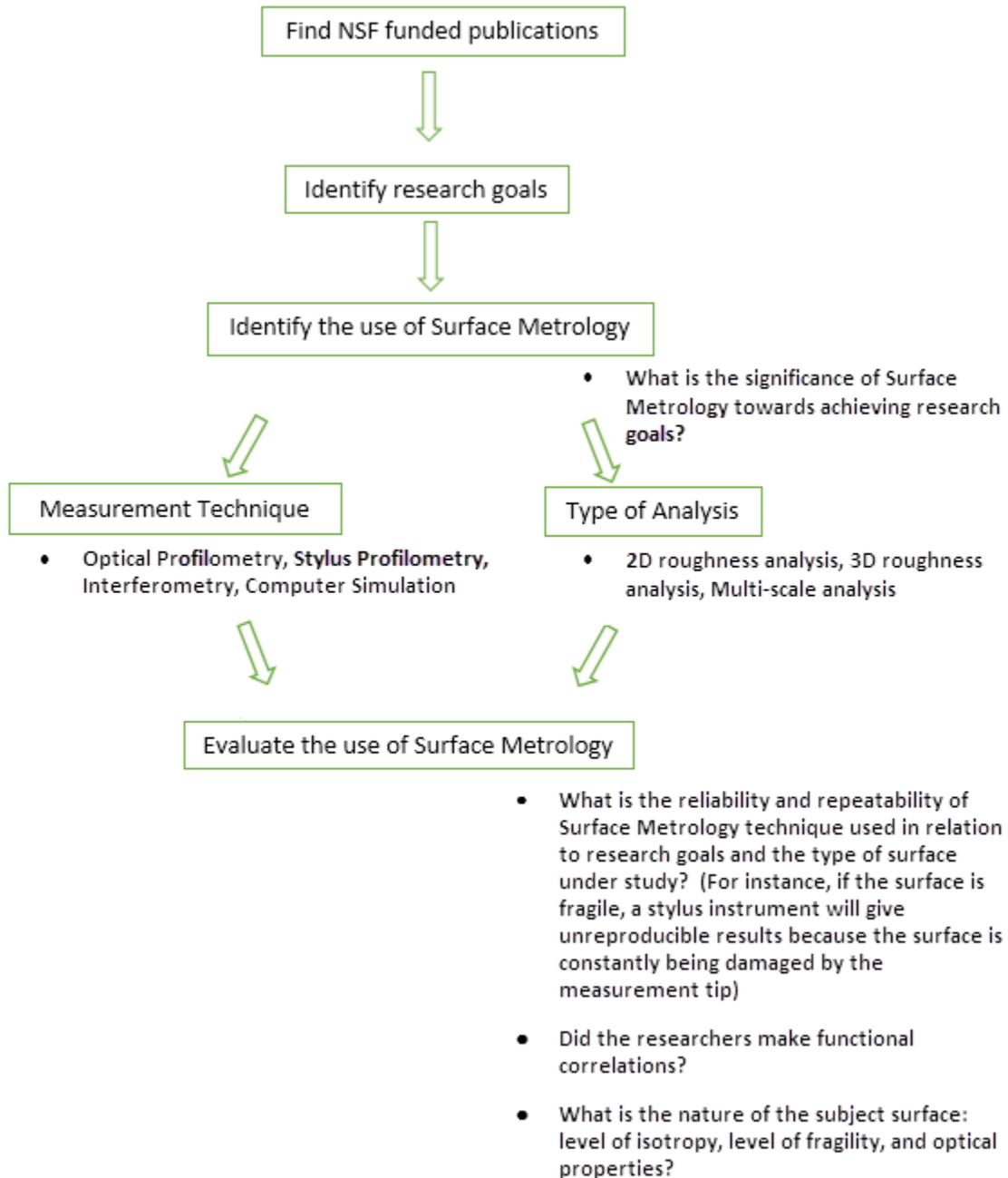


Figure 10: Project Methods flow-chart

## 4. Results

## 4.1. NSF Project 1

### 4.1.1. Research Summary

Title: <i>Formation and Characterization of Tribofilms</i>
NSF number: <i>0535578</i>
Researcher(s): <i>Kar et al. (2008)</i>

This research focuses on the effects of Industrial lubricants on tribofilms. Tribofilms are thin films generated on metal surfaces by during frictional sliding (Luo, 2013). These films are most often formed during manufacturing processes such as cutting and turning, acting as a new surface layer, and thereby, reducing friction and protecting the metal surface below it. The research conducted by Kar et al. (2008) quantified the effect of industrial lubricants on tribofilms. The four lubricants used were castor oil (vegetable-based oil), polyethylene glycol, margarine and mineral oil (the most widely used industrial lubricant). Medium carbon steel disks were used as sample metal surfaces.

The goal was to use Iron power as a binding element, which, when added to the lubricant, undergoes tribo-chemical reactions. Iron Power was used as an alternative to the more commonly used Zinc Dithiophosphate, which was found to be harmful to the environment. After the tribofilm was formed, the researchers then characterized the texture of the new surface in order to study its correlation with friction.

The average surface roughness (Ra) for the four lubricants used was 0.02  $\mu\text{m}$ . The graph below depicts the roughness values of the carbon steel disks formed by the different tribofilms. Each tribofilm is a result of a different lubricant.

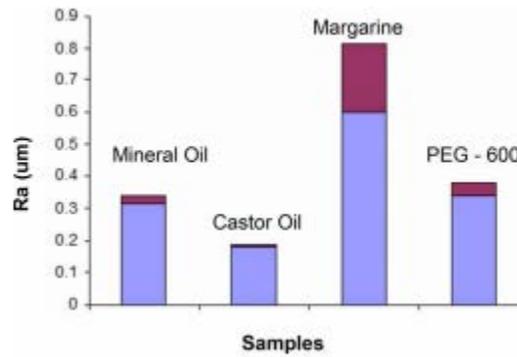


Figure 11: Roughness Values of tribofilms: Red depicts the ranges of error (Kar et al., 2008)

#### 4.1.2. Use of Surface Metrology

Name of measurement Instrument: <i>Qualitest TR200 Surface Profilometer</i>
Type of Instrument: <i>Stylus Profilometer</i>
Type of Analysis: <i>2D Roughness Analysis</i>

In this research project, Surface Metrology was used to quantify the effect of different lubricants in the formation of tribofilms on the surface. The Ra (average roughness) values were used to quantify the characteristics of the tribofilms formed. There are a few problems with the analysis method used. For instance, the significantly high roughness value and the error range of the margarine-formed tribofilm was attributed to the formation of nanoparticles and other loose particles from debris. This experiment made the use of a stylus profilometer to quantify surface roughness. However, the analysis method used, a two-dimensional parameter, could go no further than simply accounting for the various peaks and valleys. It could not account for non-linear patterns in observation. This tendency makes for an unreliable repeatability. In order to investigate

the high error ranges and the high roughness values of margarine, a scanning electron microscope (SEM) had to be used. The Figure below shows the SEM images.

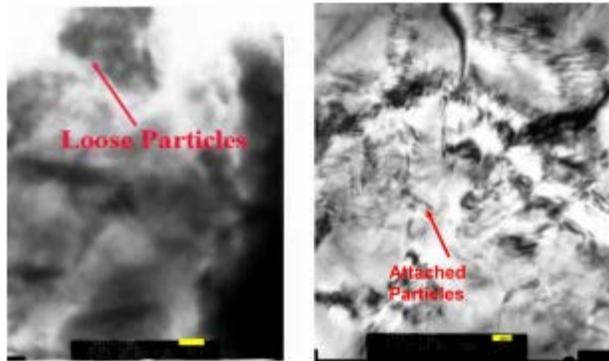


Figure 12: SEM images of Tribofil with mineral oil (left) and castor oil (right) (Kar et al., 2008)

Roughness characterization methods such as optical profilometry could have easily identified such features on surfaces prior to any analysis, giving a more comprehensive experimental result.

The other problems arises from the measurement instrument used: stylus profilometry. One of the major limitations emerges from the size of the stylus tip (Conroy and Armstrong, 2012). In order to acquire more accurate measurements, the tip diameter needs to be reduced. However, upon the reduction of the tip diameter, the risk of damaging the surface increases. The other drawback of stylus profilometry, which is connected to the tip diameter, arises from the error ranges associated with missed data points. Figure 14 shows how the tip of a stylus instrument can affect data accuracy.

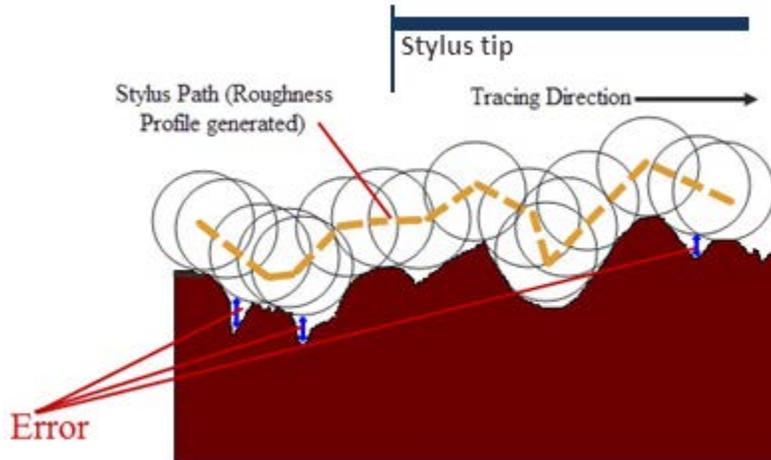


Figure 13: Missing data points in stylus profilometry

## 4.2. NSF Project 2

### 4.2.1. Research Summary

Title: <i>Surface Finish Analysis of Surgical Tools Created by Direct Metal Laser Sintering and Subtractive Manufacturing</i>
NSF number: <i>EEC-1001065</i>
Researcher(s): <i>Ragland et al. (2012)</i>

The research goals were focused on obtaining the most optimal surface finish on surgical tools. In the medical industry, a low surface roughness is desired for surgical instruments. The researchers compared surface roughness values of surgical tools produced by additive and subtractive manufacturing. The surgical instruments studied include pliers, tensile bars, condyles and cylinders. The surface roughness results for several subtractive manufacturing processes were compared side by side with that of direct metal laser sintering (DMLS), which is considered as an

additive manufacturing process. The research results are tabulated below, showing the surface roughness ranges for each manufacturing process.

Table 2: Ra Range of Surgical Tools and Manufacturing Process Used

Tool	Method	Ra Range
Circip Pliers	Milled	0.2187-1.1850
Pliers	Milled	0.2062-0.4607
Cylinder Co-Cr	Forged	0.1224-0.9533
Cylinder Co-Cr	Forged + Turned	0.0429-0.6905
Cylinder Co-Cr	Forged + Wire Cut	0.7798-1.0222
Condyle	Casted + Machined	0.1543-1.1232
Cylinder Ti-64	Machined	0.0824-0.1947
Cylinder Ti-64	Extruded	0.0756-0.4503
Tibial Jig	Boring + Turning	0.0614-0.1833
Tensile Bar	DMLS + Machined	0.2751-1.2954
Tensile Bar	DMLS as sintered	0.7840-0.9576

#### 4.2.2. Use of Surface Metrology

Name of measurement Instrument: <i>Form TalySurf Intra50 (FTS 50)</i>
Type of Instrument: <i>Stylus Profilometer</i>
Type of Analysis: <i>2D Roughness Analysis</i>

Although the range of measurement scale for this research was in the micrometer ranges, 2D roughness analysis would still have several accuracy issues. The ranges of Ra values were used here to distinguish one surface from another. A lower Ra value meant, for this particular research, a better quality surface. In order to examine the effectiveness of Ra, we can conceptually compare two surfaces side by side.

As demands for precision engineering increase, quality assurance professionals need to find more accurate methods of quantifying surface texture. Average roughness is currently the most common roughness parameter in research and industry. The limitation of this method, despite the fact that it only provides 2D profiles, also arises from its lack of reliability. Average roughness does not distinguish between two surfaces that have similar peak to valley deviations. Figure 15 depicts this phenomenon. Although the surfaces shown below have noticeably different surface textures, they will, nonetheless, have similar average roughness values.

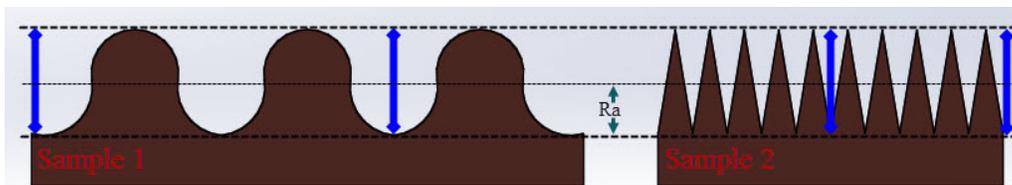


Figure 14: Ra value limitation in distinguishing two surfaces

The other limitation of this method is concerning anisotropic surfaces. The average roughness is extracted along a straight line. If the two points making up this line change locations, then the roughness profile will be distorted. Currently, there are some developments in stylus profilometry where, with some instruments, 3D profiles can be extracted. The P17 stylus profiler from KLA Tencor and the Dektak 150 surface profiler from Veeco Instruments are among the few examples. There are also new branches of surface metrology, such as atomic force microscopy, that use advanced contact methods to extract surface profiles.

### 4.3. NSF Project 3

#### 4.3.1. Research Summary

Title: <i>An Experimental and Numerical Study of Effect of Textured Surface by Arc Discharge on Strength of Adhesively Bonded Joints</i>
NSF number: IIP-1034652
Researcher(s): Asgharifar et al., 2012

Using a surface treatment method, this research correlates adhesive bond strength with the surface roughness of a substrate. Aluminum 6111 alloy was used as the subject substrate and the Terokal 5089k was used as an adhesive. The researchers also carried out simulations using the finite element analysis (FEA) software ANSYS to study the effect of surface texture on adhesive bond strength.

Adhesives are dependent on the surface property which they are bonded with. By altering the surface's texture, adhesive properties can be greatly enhanced. The relationship between adhesion and surface roughness is that of a direct proportionality. In this research, the arc discharge method was used to achieve a higher surface roughness, making it more optimal for adhesion.

The arc discharge method, using dielectric breakdown of a gas medium, creates a constant plasma current. Lightning, for instance, is a naturally occurring electric arc. This mechanism can be used for surface treatment by creating high temperature spots (craters) on the surface, thereby increasing the surface roughness (see figure 16).

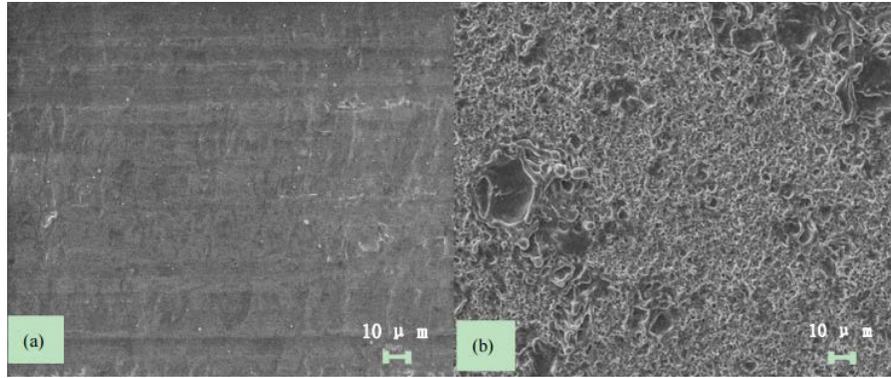


Figure 15: Arc Discharge a) Surface before treatment b) surface after treatment (Asgharifar et al., 2012)

As per the experiments, it was shown that treatment increased the bond strength. Surface treatment with low arc current and velocity resulted in a 19% increase in bond strength. The arc-current used was 5 amperes and the velocity was 20mm/s. When these parameters were increased to 20 A and 70 mm/s, the improvement from the untreated surface increase to 22.3%. In addition, non-treated surfaces were found to undergo adhesive failure with applied loading, while the failure for treated surfaces was closer to a cohesive failure, meaning the failure occurred within the adhesive.

#### 4.3.2. Use of Surface Metrology

Name of measurement Instrument: <i>ST 400 Optical Profilometer</i>
Type of Instrument: <i>Optical Profilometry</i>
Type of Analysis: <i>3D Roughness Analysis</i>

In order to characterize surface roughness, the ST400 optical profiler was used. Ra values were extracted from three random locations before being entered into ANSYS for FEA. The numerical analyses were validated by shear stress tests to see if test the bond strength between adhesive and substrate as a function of surface roughness. As mentioned above, adhesive strength was increased when surface roughness was increased. The arithmetical mean height of the surface (Sa) values of treated and untreated surfaces were 0.6 $\mu\text{m}$  and 1.57 $\mu\text{m}$ , respectively.

Brown et al. (2001) have shown that correlations with adhesion are dependent on the scale of measurement. While Sa does make up for some of the shortcomings of Ra, the measurements for both roughness analysis parameters occur at a single scale of measurement. However, the properties of a surface and consequently, any related functional correlations, tend to vary with changing scales. The figures below show how the change in scale affects the correlation coefficient ( $R^2$ ) for adhesion.

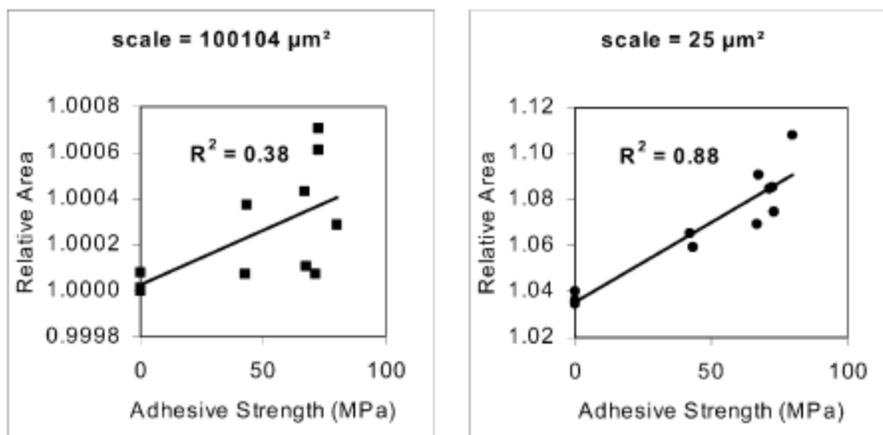


Figure 16: Correlation of Relative Area with Adhesive Strength (Brown et al., 2001)

The principle is the same in both cases. The Adhesive strength increases with higher surface roughness values. However, the phenomenon can be observed in a clearer and more substantive manner if the scale of measurement were factored in as a controlling parameter. Industry standard parameters, so far, do not integrate a varying scale of measurement when characterizing surfaces.

Confocal microscopes are applied in a wide range of fields from microbiology to manufacturing quality inspection. As with all optical profilometry techniques, the disadvantages arise from the use of light and with it, the possibility of irregular diffractions. Surfaces with irregular optical properties can result in distortion of data. This is especially characteristic in the case of alloys, where each material has a distinct optical property. The other limitation arises from the reduction of the field of view as larger magnifying objectives are used (see Figure 18).

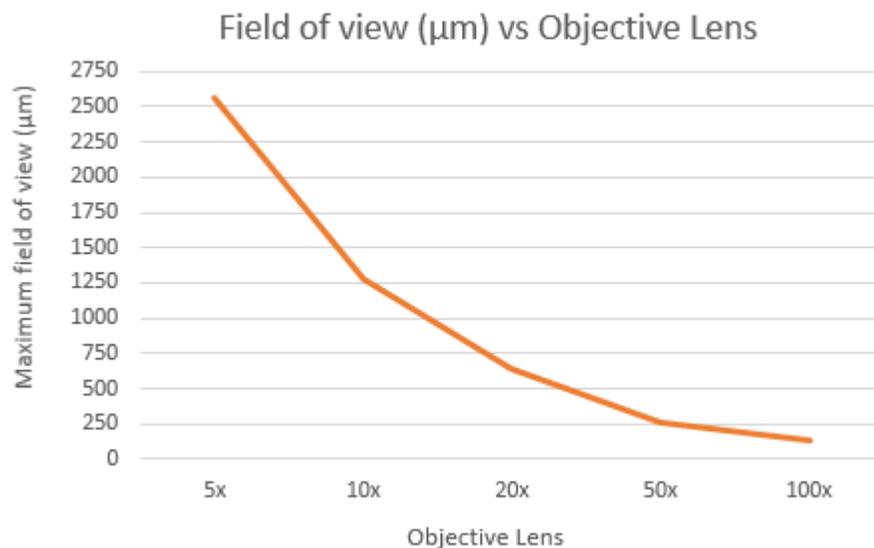


Figure 17: OLS4000 microscope: Field of View vs Objective Lens

While these limitations exist, 3D parameters still provide a more comprehensive information about surface roughness. Since the mechanism does not involve contact, confocal microscopy is more repeatable than stylus profilometry. In the case of the latter, surface damage

may occur as a result of the stylus tip dragging across the surface. Each time the profile is measured, it is possible to get errors as the line of measurement on the surface becomes more and more flattened.

#### 4.4. NSF Project 4

##### 4.4.1. Research Summary

Title: <i>Influence of acid slurries on surface quality of LBO crystal in fixed abrasive CMP</i>
NSF number: 51132005
Researcher(s): <i>Li et al. (2015)</i>

This research project studied the effect of acids on surface quality and material removal rate in fixed abrasive chemical mechanical polishing of a material ( $\text{LiB}_3\text{O}_5$  crystal). Four different types of acids were used in their experiments. The goal of the research was to identify which acid best enhances surface finish (that is, lowers the surface roughness during polishing). The researchers also correlated surface roughness with the PH value of the acid used.

The material chosen for the research project ( $\text{LiB}_3\text{O}_5$  crystal) is a crystal used in high-energy laser systems and it is desired to have significantly smooth surface. Chemical mechanical polishing (CMP) was the method chosen by the researchers to reduce the roughness of the crystal. The process requires chemical additives, such as, acids.

Their research showed that surface quality greatly influenced the material removal rate. Surface quality was in turn influenced by the type of acid used. The researchers drew correlations were drawn between material removal rate, surface roughness and PH value of the acids. Citric acid

was chosen as the most suitable chemical additive for polishing the  $\text{LiB}_3\text{O}_5$  crystal. The  $S_a$  value of the crystal polished with the aid of citric acid was 0.52 nm and the material removal rate was 435 nm/min.

#### 4.4.2. Use of Surface Metrology

Name of measurement Instrument: <i>CSPM4000 Atomic Force Microscope</i>
Type of Instrument: <i>Atomic Force Microscopy (Advanced Stylus Profilometry)</i>
Type of Analysis: <i>3D Roughness Analysis</i>

Using an Atomic Force Microscope, the researchers extracted surface roughness  $S_a$  results from the crystal, polished with the aid of four different types of acids. Their results are shown in the figure below, with citric acid corresponding with the lowest  $S_a$  value.

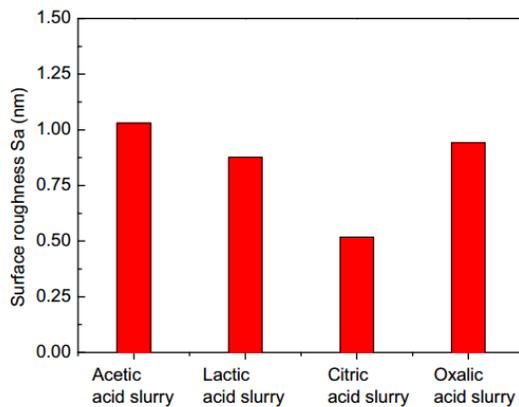


Figure 18: Surface roughness obtained with the aid of four different acids (Li et al., 2015)

With this information, the researchers were able to draw correlations between surface roughness, PH value and the material removal rate. The surface roughness demonstrates a peculiar characteristic, where it originally decreases with increasing PH and then, at a certain PH value, increases significantly.

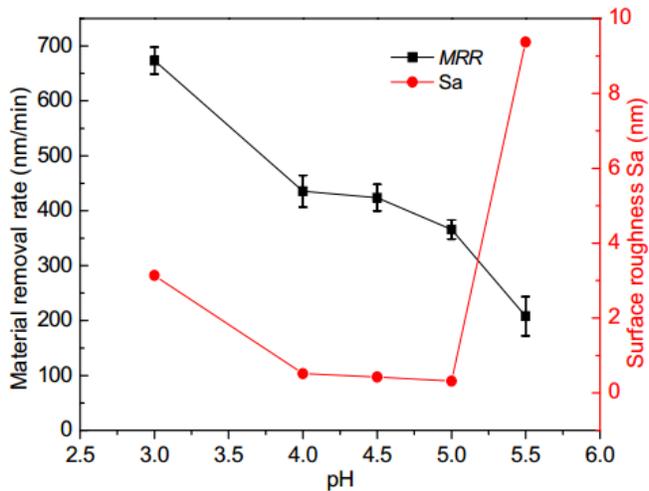


Figure 19: Surface roughness (Sa), Material Removal Rate and PH value (Li et al., 2015)

The Surface Metrology technique used in this research is effective in the respect that it employed a 3D roughness parameter. The measurement technique still falls under the stylus profilometry type because contact is still needed to obtain the surfaces' information. AFM makes use of a small tip extending from a cantilever beam to trace the profile of the surface.

The major disadvantage of AFMs comes the area and height that can be accounted for in a single measurement. The maximum area that can be measured is 100 microns (Conroy and Armstrong, 2005). The average height does not exceed 20  $\mu\text{m}$ . The other disadvantage is also the time taken (2-5 minutes), which is much smaller than that of optical profilometry.

#### 4.5. NSF Project 5

##### 4.5.1. Research Summary

Title: <i>A quantitative study of the effect of surface texture on plasticity induced surface roughness and dislocation density of crystalline materials</i>
NSF number: DMI0084992
Researcher(s): <i>Zamiri et al., 2008</i>

The researchers here focused on the plastic deformation properties of superconducting niobium. However, since the study was conducted on a granular basis, it was impossible, they concluded, to take actual physical measurements. Therefore the granular arrangements of the crystals were recreated using a computer simulation. The functions correlated to surface roughness were dislocation density, electron work function and photon emission.

Dislocation is a measure of irregularity within a crystalline structure, and dislocation density is the amount of such irregularities per unit volume. Electron work function (EWF) is a property of solid materials that describes the energy threshold to remove an electron from the surface. EWF, according to the observations by Vic et al., can give insights into the various roughness properties (friction, adhesion, and oxidation) of materials. Photon emission, which describes the thermal properties of a surface, is also another property that was correlated to surface roughness. All of these properties were assumed to have some time of sensitivity to the surface roughness.

Correlations between surface roughness and plastic deformation were obtained by simulating uniaxial tension and observing the changes in orientations of the grains. The [001] orientation (shown under section 4.5.2.), being the arrangement that produces the roughest surface, was found to have the highest dislocation density and EWF.

#### **4.5.2. Use of Surface Metrology**

Name of measurement Instrument: <i>Computer Simulation</i>
Type of Instrument: <i>Computer</i>
Type of Analysis: <i>Micro-scale modeling</i>

The computational method used was micro-scale modeling. The figures below show the simulated surfaces with differences in grain orientation in each crystal. The grain orientation is the descriptive factor of the surface roughness.

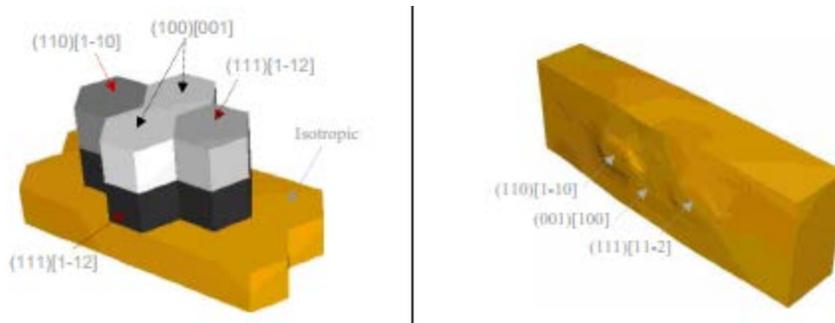


Figure 20: Surface frame showing individual grains (left) and full surface with a combination of certain orientations (right) (Zamiri et al., 2008)

Figure 21 shows the internal frame of the simulated model, where the pairs of three numbers are the surface roughness properties as described the orientation distribution function. Each grain has got different orientations but the geometry was assumed to be the same. The surface can therefore be represented with these internal features, and the changes in the orientations will demonstrate a change in surface roughness due to a processes such as dislocation and plastic deformation. Figure 21 also shows (on the right-hand side) the full surface labeled with the grains which are its underlying building blocks.

While this research study was particularly unique, it opens the door for new ways of studying surface roughness properties using virtual analysis.

## **5. Discussion**

It was found, after a rigorous search, that the majority of NSF funded research projects do not use advanced Surface Metrology techniques. Stylus profilometry, despite having several shortcomings, is the most widely used Surface Metrology technique.

The scale of measurement is one factor that was not accounted for in these projects. A study conducted by Berglund et al. (2010) showed that the scale of observation plays an important role in finding correlations between the surface topography and friction. Similarly, Brown and Siegmann (2001) have showed that the scale of observation is an important factor when finding correlations between surface topography and adhesion. Vulliez et al. (2014) have also demonstrated that the fatigue behavior of materials, related to a certain manufacturing technique, can be predicted by narrowing down the scale of observation (to where fatigue limit correlates more strongly with curvature). Multi-scale analysis, introduced in the section 2 of this paper, offers a comprehensive means of defining the surface with respect to scale and, thereby, help find reliable functional correlations. However, the Surface Metrology techniques used in NSF funded projects have not accounted for scale of measurement.

The use of conventional Surface Metrology techniques, as is the case with NSF funded research, limits the reliability and repeatability of the final results. Several factors proven to influence the result of observation are overlooked by such techniques. For instance, another parameter that would enhance functional correlations is narrowing down the range of calculations (the bandwidth). Berglund et al. (2010) have shown that even 2D roughness parameters correlate

well with a certain function if the bandwidth was limited. Using a filter, the wavelength can be narrowed down (as in tuning a radio) and several parameters tend to correlate significantly well with a function of interest.

## **6. Conclusions**

- After analyzing NSF funded research projects, we found that advanced surface metrology techniques have not been funded by the NSF. Stylus profilometry, aside from being the most widely used technique in industry, was found to be the most commonly used technique in most funded research projects.
- Surface Metrology techniques used in research projects do not account for scale of measurement, which is an important factor when defining surface texture and establishing correlations with functions.
- In addition, research and industry standard Surface Metrology techniques do not limit the range of calculations (the bandwidth) when seeking to find functional correlations.

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## Appendix A

### 2D Stylus Parameters

Table 3: 2D R-parameters log

	ISO 4287	ASME B46.1
<b>Height Parameters</b>		
<b>Ra</b> <u>Average Roughness</u>	<p>The arithmetic mean of the absolute values of the deviations of the profile within the limits of the base length.</p> $R_a = \frac{1}{l_{mr}} \int_{l_{mr}}  r(x)  dx$	<p>The arithmetic average of the absolute values of the profile height deviations recorded within the evaluation length (L) and measured from the mean line. Analytically, Ra is given by:</p> $Ra = (1/L) \int_0^L  Z(x)  dx$ <p>For digital instruments, an approximation of the Ra value may be obtained by adding the individual <math>Z_i</math> values without regard to sign and dividing the sum by the number of data points N.</p> $Ra = ( Z_1  +  Z_2  +  Z_3  \dots  Z_N ) / N$
<b>Rq</b> <u>Mean Square Slope</u>	<p>The root mean square deviations of profile, within the base in Length.</p> $R_q = \sqrt{\frac{1}{l_{mr}} \int_{l_{mr}} r(x)^2 dx}$	<p>The root mean square roughness average of the profile height deviations are taken within the evaluation length and measured from the mean line. Analytically, it is given by:</p> $Rq = \left[ (1/L) \int_0^L Z(x)^2 dx \right]^{1/2}$ <p>The digital approximation is as follows:</p> $Rq = [(Z_1^2 + Z_2^2 + Z_3^2 + \dots Z_N^2)/N]^{1/2}$
<b>Rp</b> <u>Peak Height</u>	<p>The maximum height of the roughness profile within a given evaluation length.</p>	<p>The distance between the highest point of the profile and the mean line within the evaluation length.</p>

	$R_p = \frac{1}{n_r} \sum_{i=1}^{n_r} \left  \max_{x \in R_i} r(x) \right  \text{ where } R_i = \{x : (i-1) l_r \leq x < i l_r\}$	
<b>Rpm</b> <u>Rp mean value</u>		The average of the successive values of Rp, calculated over the evaluation length.
<b>Rv</b> <u>Maximum valley depth</u>	$R_v = \frac{1}{n_r} \sum_{i=1}^{n_r} \left  \min_{x \in R_i} r(x) \right  \text{ where } R_i = \{x : (i-1) l_r \leq x < i l_r\}$	The distance between the lowest point of the profile and the mean line within the evaluation length.
<b>Rzi</b> <u>Single Roughness Depth</u>		The vertical distance between the highest and lowest points of the profile within the evaluation length.  $R_t = R_p + R_v$
<b>Rz</b> <u>Mean Roughness Depth</u>	Average of five Rzi values. $R_z = \frac{1}{n_r} \sum_{i=1}^{n_r} \left( \max_{x \in R_i} r(x) - \min_{x \in R_i} r(x) \right) \text{ where } R_i = \{x : (i-1) l_r \leq x < i l_r\}$	The average of the successive values of Rt calculated over the sampling length.
<b>Rmax</b> <u>Maximum Roughness depth</u>	Maximum peak-to-valley profile height. $R_{\max} = \max_{i=1, \dots, n_r} \left( \max_{x \in R_i} r(x) - \min_{x \in R_i} r(x) \right) \text{ where } R_i = \{x : (i-1) l_r \leq x < i l_r\}$	The largest of the successive values of Rzi, calculated over the evaluation length.
<b>Spatial Parameters</b>		
<b>Rsm</b> <u>Mean Width of profile elements</u>	Rsm is the mean value of the roughness profile width within the sampling length and requires the definition of height discrimination matching the function of the surface.	The mean value of the spacing between profile irregularities within the evaluation length.  $RSm = (1/n) \sum_{i=1}^n Sm_i$ (similar equation to ISO 4287)
<b>Rsk</b> <u>Skewness of the Roughness Profile</u>	It is a measure of Skewness ( <i>asymmetry</i> ) of the amplitude over the evaluation length. n is the number of points within a sampling length, and Yi is the height value at point i.  $R_{sk} = \frac{1}{R_q^3} \frac{1}{l_{mr}} \int_{l_{mr}} r(x)^3 dx$	A measure of the asymmetry of the profile about the mean line calculated over the evaluation length. In analytic form:  $Rsk = \frac{1}{R_q^3} \frac{1}{L} \int_0^L Z^3(x) dx$  For a digitized profile:  $Rku = \frac{1}{R_q^4} \frac{1}{N} \sum_{j=1}^N Z_j^4$

<p><b>Rku</b></p> <p><u>Kurtosis of the Roughness Profile</u></p>	<p>The Kurtosis describes the sharpness (peakedness) of the height distribution.</p> $R_{ku} = \frac{1}{Rq^4} \frac{1}{l_{mr}} \int_{l_{mr}} r(x)^4 dx$	<p>A measure of the peakedness of the profile about the mean line calculated over the evaluation length. In analytic:</p> $Rku = \frac{1}{Rq^4} \frac{1}{L} \int_0^L Z^4(x) dx$ <p>For a digitized profile:</p> $Rku = \frac{1}{Rq^4} \frac{1}{N} \sum_{j=1}^N Z_j^4$
<p><b>Rt</b></p>	$R_t = \max_{x \in R} r(x) - \min_{x \in R} r(x) \text{ where } R = \{x : 0 \leq x < l_{mr}\}$	
<p><b>Hybrid Parameters</b></p>		
<p><b>Rdq</b></p> <p><u>Root Mean Square Gradient of the Ordinate Values</u></p>	<p>The Rdq describes the mean of the absolute values of the gradient of the ordinate values</p> $R_{dq} = \sqrt{\frac{1}{l_{mr}} \int_{l_{mr}} \left( \frac{\partial r(x)}{\partial x} \right)^2 dx}$	
<p><b>Rda</b></p> <p><u>Maximum Gradient of the Scale-limited Profile</u></p>	<p>The Rda describes the maximum of the absolute values of the the gradient of the ordinate values</p> $R_{dt} = \max_{x \in R} \left  \frac{\partial r(x)}{\partial x} \right  \text{ where } R = \{x : 0 \leq x < l_{mr}\}$	
<p><b>Rdl</b></p> <p><u>Developed Interfacial Length of the Scale-limited profile</u></p>	<p>The Rdl describes the arc length of the ordinate values</p> $R_{dl} = \frac{1}{l_{mr}} \int_{l_{mr}} \sqrt{1 + \left( \frac{\partial r(x)}{\partial x} \right)^2} dx$	
<p><b>Rdr</b></p> <p><u>Developed interfacial length ratio of the Scale-limited profile</u></p>	<p>The Rdr describes the ratio of the increment of the developed interfacial length of the ordinate values</p> $R_{dr} = \frac{1}{l_{mr}} \int_{l_{mr}} \left( \sqrt{1 + \left( \frac{\partial r(x)}{\partial x} \right)^2} - 1 \right) dx$	

### 3D (S-Parameters)

Table 4: 3D S parameters log

	ISO 25178	ASME B46.1
<b>Height Parameters</b>		
<p><b>Sa</b></p> <p><u>Arithmetical mean height of the surface</u></p>	<p>Arithmetic mean of the absolute of the ordinate values within a definition area (A)</p> $S_a = \frac{1}{A} \iint_A  z(x,y)  \, dx dy$	<p>The arithmetic average of absolute values of the measured height deviations from the mean surface taken within the evaluation area. Sa is given in cartesian coordinates as follows:</p> $S_a = (1/Ae) \int_0^{Ly} \int_0^{Lx}  Z(x,y)  \, dx dy$ <p>For a rectangular array of M x N digitized profile values <math>Z_{jk}</math>, the formula is given by the equation:</p> $S_a = \frac{1}{MN} \sum_{k=1}^M \sum_{j=1}^N  Z_{jk} $
<p><b>Sq</b></p> <p><u>Root mean square height of the surface</u></p>	<p>Root mean square value of the ordinate values within a definition area (A)</p> $S_q = \sqrt{\frac{1}{A} \iint_A z^2(x,y) \, dx dy}$	<p>Root mean square roughness (rms): The root mean square average of the measured height deviations from the mean surface taken within the evaluation area. Analytically, Sq is given by:</p> $S_q = \left( (1/Ae) \int_0^{Ly} \int_0^{Lx} Z^2(x,y) \, dx dy \right)^{1/2}$ <p>The digital approximation is as follows:</p> $S_q = \left[ \frac{1}{MN} \sum_{k=1}^M \sum_{j=1}^N Z_{jk}^2 \right]^{1/2}$

<p><b>Ssk</b> <u>Skewness of height distribution surface</u></p>	<p>Quotient of the mean cube value of the ordinate values and the cube of Sq within a definition area (A)</p> $S_{sk} = \frac{1}{S_q^3} \left[ \frac{1}{A} \iint_A z^3(x,y) dx dy \right]$	<p>A measure of the asymmetry of surface heights about the mean surface. Analytically, Ssk is given by:</p> $S_{sk} = \frac{1}{(S_q)^3 A_e} \int_0^{L_y} \int_0^{L_x} Z^3(x,y) dx dy$ <p>The digitized profiles it may be calculated from the following:</p> $S_{sk} = \frac{1}{(S_q)^3} \frac{1}{MN} \sum_{k=1}^M \sum_{j=1}^N Z_{jk}^3$
<p><b>Sku</b> <u>Kurtosis of height distribution</u></p>	<p>Quotient of the mean quartic value of the ordinate values and the fourth power of Sq within a definition area (A)</p> $S_{ku} = \frac{1}{S_q^4} \left[ \frac{1}{A} \iint_A z^4(x,y) dx dy \right]$	<p>A measure of peakedness of the surface heights about the mean surface. Analytically, Sku may be calculated from the following</p> $S_{ku} = \frac{1}{(S_q)^4 A_e} \int_0^{L_y} \int_0^{L_x} Z^4(x,y) dx dy$ <p>For a digitized profile, it may be calculated from the following:</p> $S_{ku} = \frac{1}{(S_q)^4} \frac{1}{MN} \sum_{k=1}^M \sum_{j=1}^N Z_{jk}^4$
<p><b>Sp</b> <u>Maximum height of peaks</u></p>	<p>Largest peak height value within a definition area</p>	<p>The maximum height in the evaluation area with respect to the mean surface</p> <p><i>Mean surface: the three dimensional reference surface about which the topographic deviations are measured.</i></p>
<p><b>Sv</b> <u>Maximum height of valleys</u></p>	<p>Minus the smallest pit height value within a definition area</p>	<p>The absolute value of the minimum height in the evaluation area with respect to the mean surface</p>
<p><b>Sz</b> <u>Maximum height of surface</u></p>	<p>Sum of the maximum peak height value and the maximum pit height value within a definition area</p>	

<p><b>St</b> <u>Root mean square height of the surface</u></p>		<p>The vertical distance between the maximum height and the maximum depth in the evaluation area</p> <p><math>St = Sp + Sv</math></p>
<p><b>Hybrid Parameters</b></p>		
<p><b>Sdq</b> <u>Root mean square surface slope</u></p>	<p>Sdq is a general measurement of the slopes which comprise the surface and may be used to distinguish surfaces with similar average roughness, <b>Sa</b>.</p> $Sdq = \sqrt{\frac{1}{A} \int_0^{Lx} \int_0^{Ly} \left( \frac{\partial Z(x,y)}{\partial x} \right)^2 + \left( \frac{\partial Z(x,y)}{\partial y} \right)^2 dy dx}$	
<p><b>Sds</b> <u>Summit Density</u></p>		<p>The number of summits per unit area making up the surface. Summits are derived from peaks.</p> $S_{ds} = \frac{\text{Number of local maximums}}{(M-1)(N-1)\delta x \delta y}$
<p><b>Sdr</b> <u>Developed surface Area Ratio</u></p>	<p>Sdr is expressed as the percentage of additional surface area contributed by the texture as compared to an ideal plane the size of the measurement region.</p> $Sdr = \frac{(\text{Texture\_Surface\_Area}) - (\text{Cross\_Sectional\_Area})}{\text{Cross - Sectional - Area}}$	
<p><b>Ssc</b> <u>Mean summit curvature</u></p>	<p>Ssc is the mean summit curvature for the various peak structures.</p> $S_{sc} = \frac{1}{N_{\text{Summit - Area}}} \iint \left( \frac{\partial^2 z(x,y)}{\partial x^2} \right) + \left( \frac{\partial^2 z(x,y)}{\partial y^2} \right) dx dy$	
<p><b>Std</b> <u>Texture direction of surface</u></p>	<p>Std is a measure of the angular direction of the dominant lay comprising a surface. It is defined relative to the Y axis.</p>	

## Spatial Parameters

<b>ACF</b> <u>Auto-correlation Function</u>	The ACF is a measure of how similar the texture is at a given distance from the original location. If the ACF stays near 1.00 for a given amount of shift, then the texture is similar along that direction. If the ACF falls rapidly to zero along a given direction, then the surface is different and thus uncorrelated with the original measurement location.	
<b>Sal</b> <u>Auto-correlation length</u>	Length of fastest decay of ACF in any direction	
<b>Str</b> <u>Texture aspect Ratio</u>	Ratio of the Length of fastest decay of ACF in any direction to the Length of the slowest decay of ACF in any direction	