New Methods

For

Non-Contact Anodize

Measurement

Joseph Price
Chief Technology Officer
Sensory Analytics, LLC
www.sensoryanalytics.com

Table of Contents

Introduction

Anodized aluminum and titanium are rapidly becoming the materials of choice for many new products within aerospace, automotive, medical, and other industrial markets including engine and brake systems, surface finishes and building structure components. These increasing uses for aluminum and other alloys have pushed demand to all-time highs - along with their material costs. The subsequent investment of labor, engineering and manufacturing costs into the production and processing of high technology products from these materials have increased the need to control scrap, rework and ancillary costs. These higher value and profile product applications have generated more rigorous quality control along with increased outlays of labor and increased scrap costs associated with testing. New, non-destructive methods of quantifying surface finishes that do not introduce additional scrap and help minimize labor hours and reduce manufacturing costs are needed by manufacturers to maintain a competitive edge.

1.0 Background : The need for non-contact measurement capability

Traditional coating thickness measurements still generally employ contact based techniques or various destructive means for coating and finish thickness validation. Although these traditional measurement means may appear to be economical at initial purchase, they still involve labor hours for both measurement and interpretation of results and can not provide assurance against downstream costs for scrap material.

This presentation will serve to illustrate new non-contact measurement capabilities that are not only more precise and accurate in their real-time results than traditional contact methodologies, but are a cost-effective means to generate a near term return on investment.

1.1. Current State of the Art:

The measurement of anodize layers has been frequently challenged by presentation of the measuring device to the sample. Surface structure or irregularity routinely causes variation in any measurement device but is especially punctuated in contact measurement techniques. Contact based techniques are also challenged when employed near edge or curved or abstract shaped specimens require measurement.

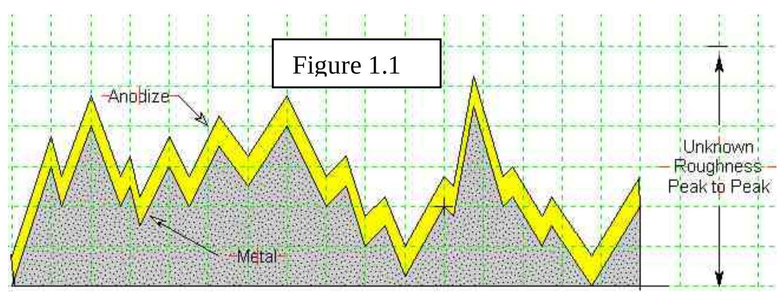


Figure 1.1

Figure 1.1 illustrates how an oxide layer may form in perfect conditions. Because of the surface irregularity of this specimen, the accuracy of contact based measurement is restricted.

The new *SpecMetrix* measurement system design shown at this conference for the first time focuses on providing solutions to chronic measurement problems that have burdened the industry for decades—including the inability to actually measure thin layers without their damage or destruction, which may be caused by simply contacting them or exposing them to air-born contamination. It is my premise that a contact free measurement will help manufacturers and the metal finishers that support them to solve some longstanding problems related to measuring anodized layers.

2.0 The case for non-contact measurement capability

A new method for non-contact coating measurement has been needed to help eliminate increasing costs of scrap and labor, and to help make manufacturers and processors more 'lean' in their methods and processes. The increasing demand and greater volume of components will require a more accurate, capable, and timely measurement technique. However, the method must also prove precise and accurate enough to replace current techniques. Some key considerations of new measurement systems must include:

Cost reduction: Primarily, non-contact measurement techniques are a vehicle for reducing cost and ultimately producing a better product.

Better quality: Larger sample sets (more data) may be collected and analyzed since the metrology methodology does not directly impact operations.

Productivity: Methods can improve start-up efficiency, provide real-time actual measurement results and eliminate scrap and rework

Scalability: Help advance QA toward process automation and 100 % inspection of piece goods, while reducing overall time to manufacture and test pieces.

2.1. Cost Issues

Scrap materials: Scrap reduction is especially important when witness samples are used to verify quality as well as with early 100% measurement validation.

Labor impact: New methods can reduce the cost of rework, material handling and information management. A great myth in metrology is that hand-held instruments offer a cheap solution. In many cases, they can be more expensive because they require additional labor to operate/collect data.

Dwell time Reduced time "waiting" to validate the process is now possible with new non-contact methodologies. Port measurement data directly for reporting in regulated industries provide automatic reports.

Capability: Expanded market share may be possible through additional capabilities.

Capacity: New methods will help make better use of equipment and increase efficiency and lean operations.

3.0 **Validation**:

Validation for the non-contact methodologies can be accomplished through the use of several different techniques. Although such validations can be initially costly, all measuring systems require this procedure to establish confidence.

Microscopy in different forms seems to remain the most common choice of validating anodized coating thickness. There are also other techniques such as “weigh-strip-weigh” and a ball drop technique, which are still employed to validate anodized layers. Each technique still requires a sacrificial sample which adds some additional cost and a temporary manufacturing inefficiency.

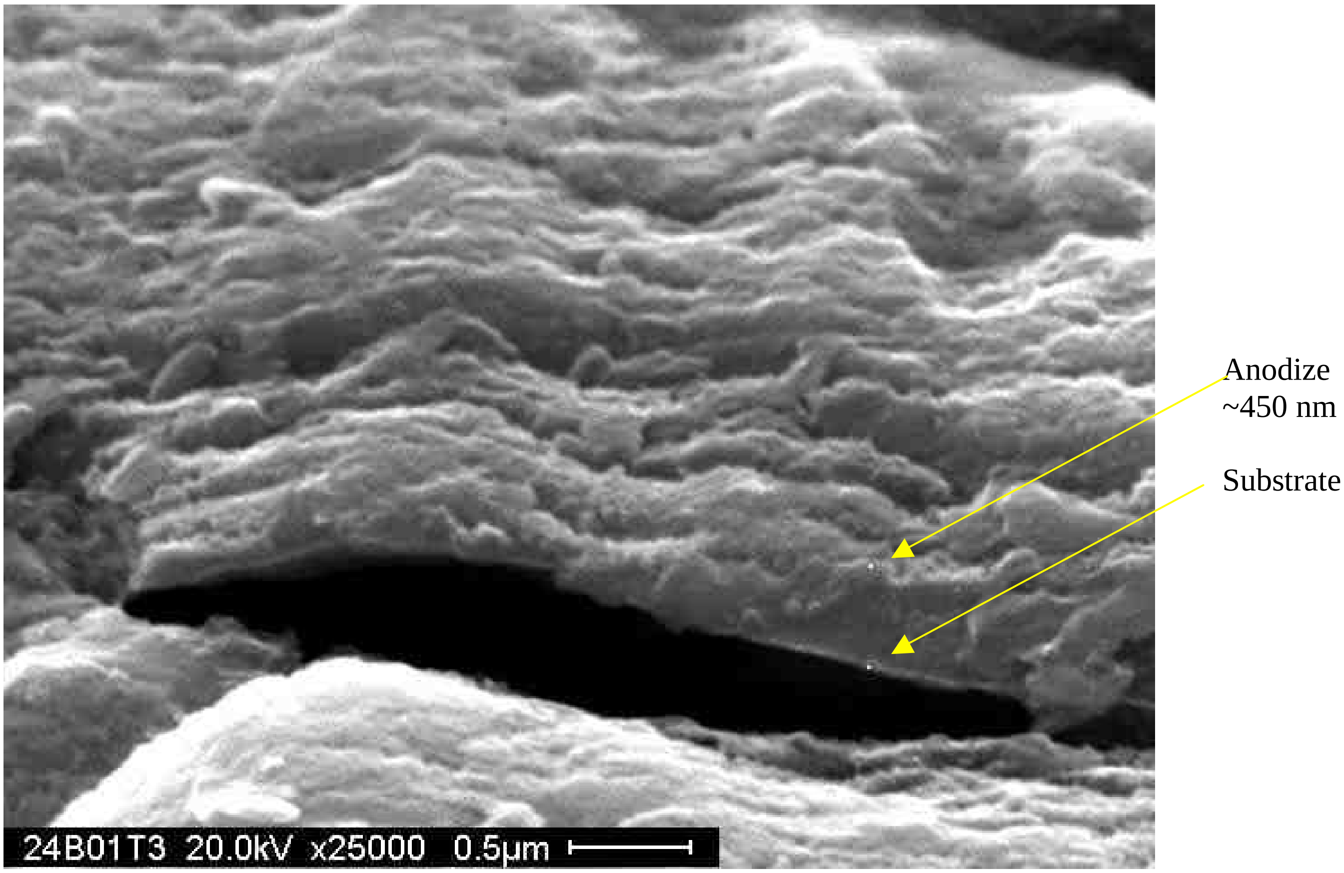
Herein, we intend to introduce techniques that approach precision and accuracy needed to be worthy of a new standardized measurement technique for anodization layers without adding any inefficiencies into the manufacturing stream. This technique has been validated independently by numerous1 parties.

Even at best, each technique introduces individual type of subjectivity and does not sufficiently provide statistically significant data without large costly production runs and related burdens associated with sampling. Gross errors associated with manual measurement compound the usual cost risks as anodized aluminum becomes widely used in regulated industries such as health and medical devices.

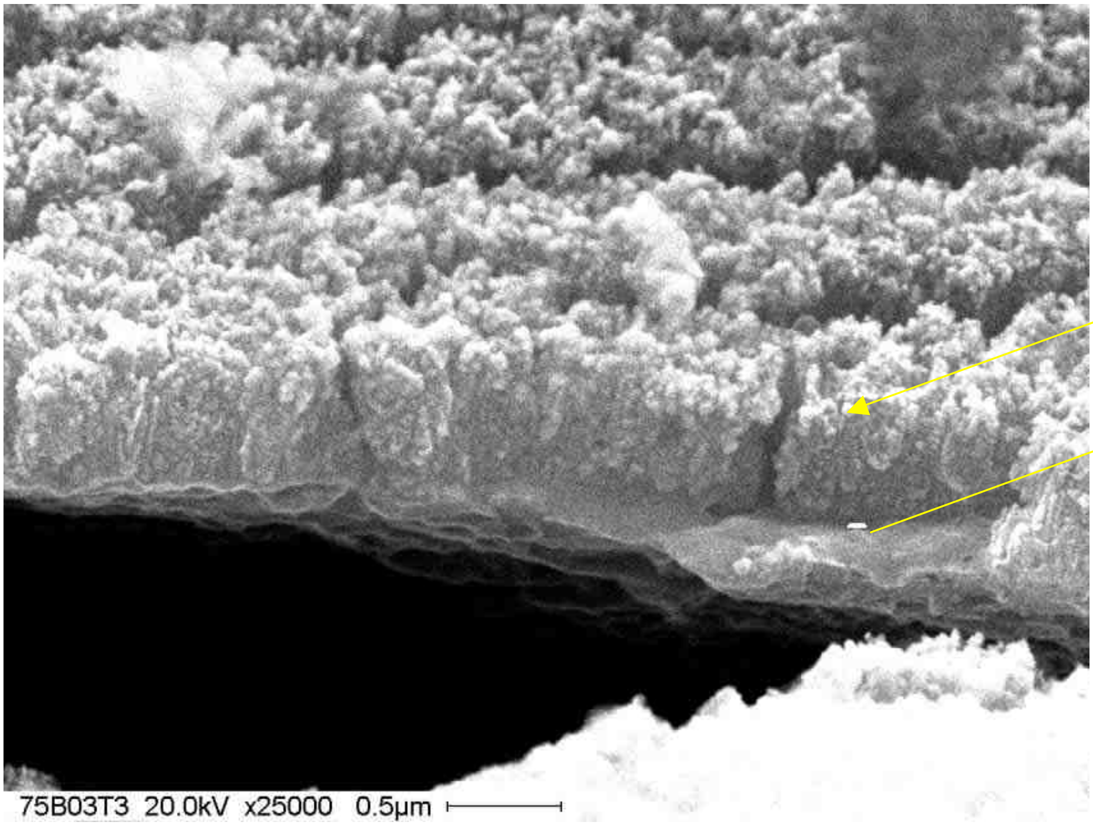
However, in today’s economy, large production runs may fade to outsourced runs, where manufacturing costs are scrutinized or outsourced all together. Because of this inefficiency, we are discussing means of providing such data with minimal impact so that the user can provide a better product at less cost. This is an universal benefit that can be now enjoyed by manufacturers and processors in all markets.

3.1. <u>Scanning Electron Microscopy</u>: As a Validation Technique

Below are two SEM profiles for 2025 and 7075 alloys that show how an optical measurement and visual measurements might be used. The limitations of each type of measurement should be considered.



The SEM image above is for a 2024 alloy containing what appears to be about a 450 nm layer of PAA. The SEM requires a highly skilled operator and a substantial investment in time for sample preparation. Successful interpretation of results requires considerable experience.



This SEM image above is for a 7075 allow containing about a 500 nm layer. This photo serves to validate our non-contact coating thickness results but further illustrates how delicate the samples are and easy the sample is to become damaged simply in preparation.

3.2. Contact Measurement Techniques

Weigh-Strip-Weigh is generally accepted as a means of validating coat weight within coil coating and other applications and is used to communicate the amount of anodization applied to a surface instead of thickness. Weigh-Strip-Weigh is beneficial in that it is not subjected to surface reflectivity or sample presentation; however, it is very costly in terms of scrap and machine or process dwell time, and may be subject to human error.

Microscopy in general employs significant time and effort required to section a part and prepare the surface for measurement. At best it is subject to the skill of operators that must prepare the samples as well as some interpretation of results. Photographic records have helped this technique for validation but it is not a practical solution for everyday use on the shop floor.

Displacement techniques such as eddy current and capacitance measurement depend on isolation from the base metal. Moreover, the thickness of the layer forces the probe away from the conductive aluminum border. There are inherent problems in using displacement techniques due simply to the surface conditions, which are generally solved by increasing the measurement area of interest (AOI). This can improve the variation from point to point but without a range of and deviation in measurements the information may become over damped and ineffective near edges. These techniques do not accommodate measurement in holes, edges or abstract shaped parts so one is left to guess work.

Coat weight measurement can rely on the measurement of weight addition or subtraction. The contribution of the substrate influences accuracy of the measurement. Coat weight is measured by simply weighing a sample before and after coating, in most cases, destructively by cutting a witness sample weighing, stripping off the coating, and reweighing it. Precision depends on the ratio of substrate to coating. Accuracy depends generally on a balance.

Illustrated is a very complex aircraft structure which has been machined and coated. It is sacrificed for quality validation using sectioning and photo-micrograph techniques. The goal has been to reduce the need for such labor and material intensive testing techniques.

4.0 <u>Optical Thickness:</u>

1. Interferometry is a mature science that is more popular in the semiconductor industry.
2. Its mainstream application has been used traditionally for much thinner layers than anodization.
3. Typical layer structures are very smooth and the environment is a pristine, clean and with minimal contamination.
4. A new approach was required to successfully address issues of anodized layers.

Key challenges in developing a new non-contact measuring system have included:

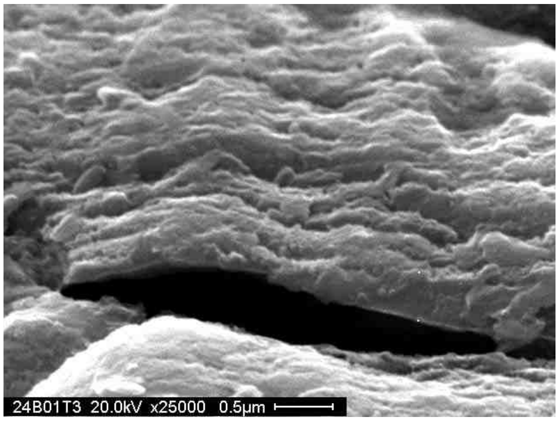
1. Finding non-contact techniques that were suitable for anodizing and related processing;
2. Finding a robust method that could be implemented in multiple aspects of surface finishing;
3. To make the system user friendly and require minimal operator training;
4. Compartmentalize quality measurements into a single integrated platform;
5. Build a system suitable for shop floor, QC lab, field testing, in-line and automated (robotic) integration.

Capabilities of these systems needed to include:

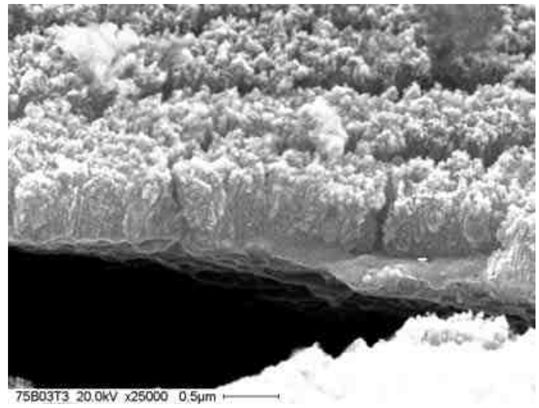
1. Ability to measure film thickness with minimal influence of surface reflectance, intensity fluctuations and distance from the sample;
2. Integrated into one self contained, transportable assembly: (patent pending);
3. Simplified operator interface suitable for shop floor operation (patent pending);
4. Integrated thickness, gloss, and color designed into one ruggedized system;
5. Built into a scalable platform suitable for automation (robotic, fixed and in-line) installation.

Why do multiple industries need new NDT measurement methodologies?

1. Aluminum is a fast-growing substrate of choice within the aerospace, automotive, transportation, semiconductor, consumer products and building component markets;
2. Market expansion into more regulated industries will drive increased quality assurance demands;
3. Profit margins are at risk if new technology is not available to assure 100% part compliance without adding substantial labor costs;
4. The use of automation in new processes will reduce the risks of outsourcing.



SEM micrographs of 2024 alloy



SEM micrographs of 7075 alloy

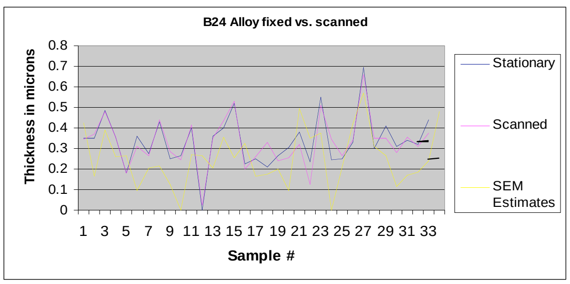


Chart 1:

Illustrated is comparison of approximately 33 different positions that were used to validate the optical thickness measurements performed on the 2024 alloy with SEM estimates. The data indicates that some trending occurs even though the estimates prove the difficulty in measurements near the same spot.

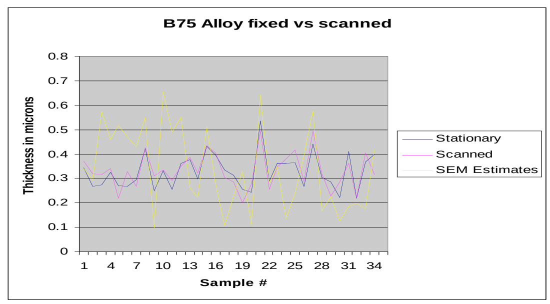


Chart 2:

Illustrated here is a comparison of approx. 33 measurement positions which were used to validate optical thickness measurements performed on 7075 alloy with SEM estimates. The data also indicates some trending.

An average of all data sets are compared which shows a slight bias in measurement results

4.1. Subjectivity

Charts 1 and 2 illustrated above in Section 3.0 demonstrate some of the subjectivity that occurs in almost any validation process simply because it is virtually impossible to measure in the same position with each sample. However, with smoothing and averaging, the agreement can be reasonable for most non-contact measurement applications.

Which technique is correct?

- SEM results are subjected to slight changes in the viewing angle. SEM data are used extensively for small areas.

- Optical results are subjected to correction in the refractive index of the layer as well as sample roughness.

- Other contact techniques are subjected to sample roughness and variations in contact angle with the substrate.

What is good enough?

Can the process be controlled more accurately than the measuring devices?

5.0 Non Contact Advantages

- Non-destructive means of securing 100% piece part measurement
- Can provide accurate and objective results
- Provides real-time results
- Scaleable for process automation and control
- Minimizes labor impact

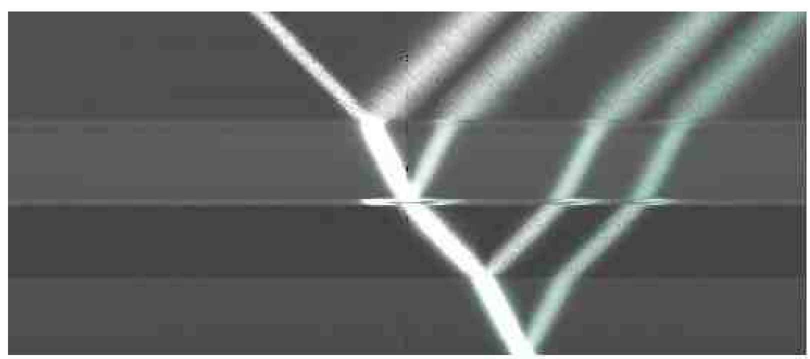
5.1. Non-Contact Techniques Using the Interference Principal:

Interference is an optical phenomenon that almost everyone is familiar with that dates back to days after the great flood - as seen in the form of a rainbow. Other examples are soap bubbles and oil slicks where we see a color difference due to the presence of a layer. This occurs simply due to a slight difference in the refractive index between air and the layer. The greater the difference the more pronounced a layer may appear to the eye.

A visual interference occurs in thin layers. Limited by three internal detectors, the human eye can only resolve very thin layers ranging up to a few hundred nanometers which appear as different colors. However, visual judgment is only safe for gross inspection of clear layers and is virtually useless in black or color anodized specimens. Nearly 8% of all males in the USA are color deficient in some degree and human imperfection allows for skewed data. Therefore, visual colorimetry should not be used as a thickness assessment.

For the anodizer, it may be difficult or impossible to reliably see interference even though some manufacturing processes still call for a visual test for the presence of an anodized layer. Even if a layer is visibly present, ambient lighting and the observation angle will always influence what is observed. Color deficiency further complicates visual judgments.

Interference model



Illustrated to the right is a model showing how light might enter and exit a coated anodized layer. This example explores a two layer system where a semitransparent bond coating is applied to protect the anodization layer.

The majority of light is directly reflected from the surface yet portions of the light energy are returned from the border of each layer to the surface. Interference occurs as the light path through each layer changes due to the thickness and change in refractive index.

The change in optical path difference between the surface and light returned from within the layer causes constructive and destructive interference which produces very accurate results because only the anodized layer itself influences the measurement. Therefore, the technique is independent of the substrate weight or thickness.

Measurement results can be compiled and processed in a few milliseconds as opposed to minutes, which makes interferometry highly suited for both lab and in-process measurement.

Illustrated to the right is our non contact optical thickness measurement system, the SpecMetrix™, which provides high speed measurement capability for both anodization and bond coating layers without destroying the sample. Time to acquire measurement is about 500 ms.

Considerations:

1. The SEM image contains an area of about 5 microns. A small bar indicator is used to scale anodize thickness.
2. The viewing angle can distort the observed thickness; which is still a photograph that must be interpreted so it is still subjective to some degree.
3. The amount of preparation required in advance to measurement introduced a practical limitation in the amount of data one could analyze.
4. What’s good enough?

5.2. <u>Why Non Contact Measurement?</u>

Many processing challenges and problems can be solved more simply using non-contact methodology. This discussion is to illustrate techniques that provide not only a non-contact measurement, but accurate and repeatable methods that can be utilized in a QC lab, floor of a job shop, an in-line control system for coil coating, and an in-tank measurement for precision end point controls that solves many of the chronic problems that have plagued material processors for years.

It should be noted that a visual interference occurs in thin layers. Limited by three internal detectors, the human eye can only resolve very thin layers ranging up to a few hundred nanometers which appear as different colors. However, visual judgment is only safe for gross inspection of clear layers and is virtually useless in black or color anodized specimens. As indicated earlier, nearly 8% of all males in the USA are color deficient in some degree and human imperfection allows for skewed data. Therefore, visual colorimetry should not be used as a thickness assessment.

For an anodizer, it may be difficult or impossible to reliably see interference even though some manufacturing processes still call for a visual test for the presence of an anodized layer. Even if a layer is visibly present, ambient lighting, and observation angle will always influence what is observed. Color deficiency further complicates visual judgments.

It is based upon the requirements of forward looking manufacturers and material processors that the Sensory Analytics team has developed the first means for non-contact thin film and anodized coating measurements in real-time: The *SpecMetrix* portable coating measurement system. This system is available for viewing and sample test use following this session at the Sensory Analytics booth in the Exhibit Hall.

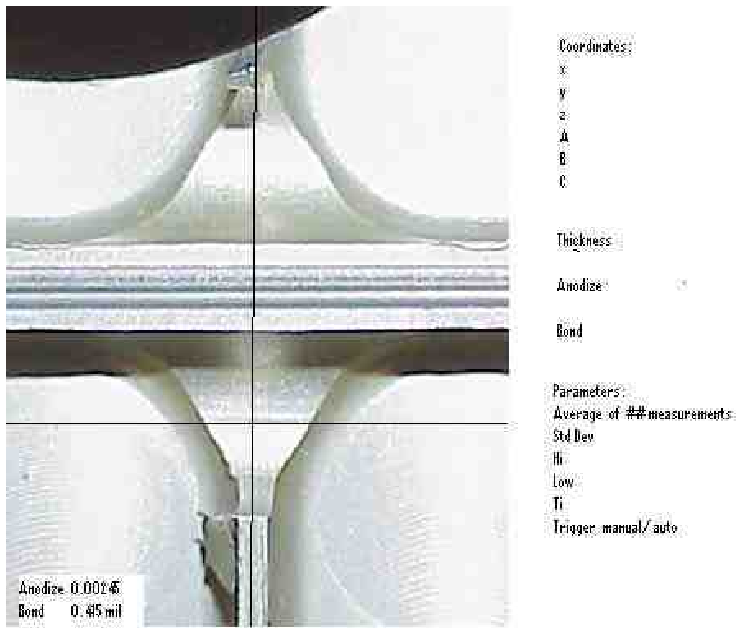
*SpecMetrix* Results:

1. In order to achieve reliable interference, the instrument required about a 1 mm 2 area of interest (AOI).
2. The integration caused some (smoothing) of the results which may not fully present a void or spike in the coating.
3. In order to measure absolute thickness, one must know the refractive index of the coating. Our research indicates that the refractive index varies from theoretical due in part to the nebulous border between the aluminum and oxide layer.

5.2.1. Non-Contact Measurement Examples

Grid-Lock Aluminum Aerospace Structure

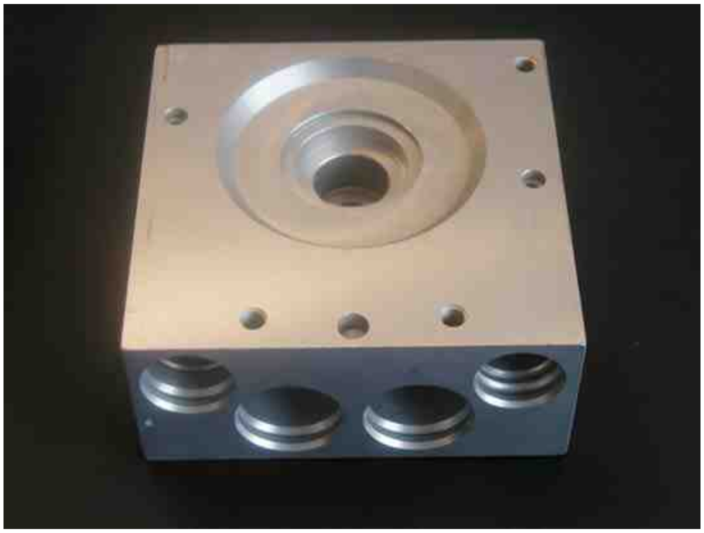
Illustrated you will see how a focused beam of light allows measurement of the anodization layer inside a small channel.



Grid-Lock Aluminum Aerospace Structure with Powder Coated Bond Primer

To the left is a part similar to the previous with a bond primer applied. A hand operated probe is used to measure in corners. In this case, the system allows a two layer analysis so that the anodized layer and bond primer layer can be measured independently at the exact same spot without destroying the sample.

Multi-Port Anti-Lock Brake Modulator



The *SpecMetrix*™ non-contact measurement mode provides inspection capability inside small ports that would normally require destructive testing. The optical system can be focused to reach down in each inspection point to guarantee coating thickness measurement. The system is easily integrated into a manufacturing stream to provide 100% inspection of all parts.

Automotive Break Caliper

Critical inspection points can be easily measured by simply focusing the optical system to acquire thickness data without contacting the surface or creating scrap components by traditional destructive testing.

Integration of the system into a robotic test cell allows every component to be fully tested and automatically documented.

Integration Methods

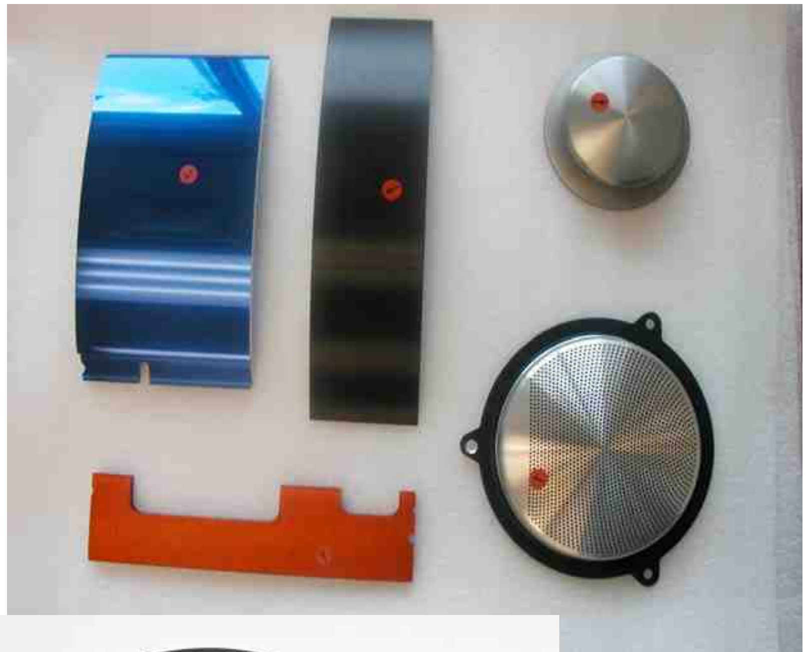
Automated thickness measurement is easily accomplished by substituting a robotic test cell for fixed point test stand. Here the system engineer configures critical inspection points for routing quality inspection.

Fiber optics used in the *SpecMetrix* ® system simplifies integration because it totally isolates the measuring system from the positioning system thus allowing "shoe-in" approach to current automated inspection systems. It further provides intrinsic safety so it can be used in C1D1 environments, vacuum deposition, as well as in the anodization bath itself.

In addition to robotic integration, active automation efforts are currently underway with leading providers of coordinate measuring machines and fixed arm metrology instruments.

Various Audio System Components

For the surrounding samples, our system can be used to measure both clear and color anodized components. The contact free operation simplifies tooling for automated inspection and provides an advanced quality control system with minimal labor impact for distinguished high quality tolerances. The automated reporting capability provides a near turn-key quality program by design.



<u>Various Medical Devices, Screws, and Clamps</u>

SpecMetrix ™ contains an automated measurement procedure to address the rigors of regulated measurements in these medical devices which require high precision measurement of anodization thickness and color. *SpecMetrix*™ reporting capability produces lot and product history recording capability with minimal labor impact.

6.0 For additional information

For additional information on *SpecMetrix* systems or to explore the technologies that are helping to shape a next generation of surface finishing metrology, please visit the Sensory Analytics booth at the show or contact the author at:

Joseph K. Price
Chief Technology Officer
Sensory Analytics, LLC
Joseph.price@sensoryanalytics.com
Tel: 336.315.6090
Fax: 336.315.6030

www.sensoryanalytics.com

References

Price, Joseph K. Anodizing System With a Coating Thickness Monitor and an Anodized Product. US 6674533 B2. Washington, D.C.: United States Patent and Trademark Office, 2004. 6,674,533

