Ultra High Purity Electroplated Aluminum Coatings for Critical Components in Dry Etch and Process Chamber Environments

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I. Executive Summary

A thick, uniform coating of high purity aluminum, applied to the surfaces of processing equipment components fabricated from aluminum alloys, greatly enhances their surface properties. Alloying elements and impurities are eliminated; the inherent risk of contamination from exposed surfaces is reduced. Subsequent anodization is greatly enhanced by eliminating the root cause of contaminants and defects in the surface oxide layer.

The semiconductor fabrication equipment industry (SemiFab) uses alloys of aluminum to construct integrated circuit fabrication equipment. Aluminum alloys are inherently preferred because aluminum is a low hazard contaminant to silicon wafer circuitry. Aluminum is also preferred because it lends itself to the formation of an aluminum oxide surface layer through the process of anodizing. The resultant surface oxide layer is tough, durable, inert and can be created with lower impurities and alloying elements than are present in the underlying aluminum alloy substrate.

The SemiFab industry must continuously increase its capabilities to meet ever increasing demands, primarily for smaller circuitry features. Smaller features are more sensitive to contamination; therefore processing equipment must operate more cleanly. Aluminum alloys used to fabricate equipment components must be processed to prevent contamination from alloying elements or impurities, either by surface cleaning or conversion to aluminum oxide (anodization). Anodizing processes have evolved to meet these increased requirements, moving from traditional sulfuric acid anodizing solutions to next generation solutions based upon oxalic acid.

The SemiFab industry is struggling to develop processes to prepare the surfaces of aluminum alloy components with the purity, inertness and durability required to fabricate the next generation of smaller integrated circuits with high yields and low operating costs. Purity and inertness affect the yield of good circuits obtained from each wafer. Durability affects the frequency of downtime for cleaning and maintenance cycles. Yield and downtime are primary determinants of SemiFab equipment value.

Test results are presented that demonstrate the superiority of anodized high purity aluminum in all critical performance areas. The purity of the electroplated aluminum is shown to be >99.99%. The resultant oxide surface has fewer contaminants (for example: Zn @ 5 ppm vs 180 ppm). Corrosion resistance is superior (HCl bubble test @ 140 hours vs 13 hours). The dielectric strength is measured as high as 2500 V per 0.001”.

A thick, uniform surface coating of high purity aluminum enhances the value provided by SemiFab tools and allows the effective migration to reduced integrated circuit feature sizes.

II. Introduction: The Problem of Contamination in Semiconductor Processing

Challenged with the tenets of Moore's law, the semiconductor industry has kept pace via innovative materials and coatings solutions. Semiconductor geometries and features are migrating to 22 nm and roadmaps are already established for further reductions to 14 nm and lower sizes.

With lower feature spacing, SemiFab processing equipment is increasingly less tolerant of even minute levels of contamination. The inherent purity of the substrate or surface coating plays a more important role since contamination is undesirable at any level.

The performance of aluminum components for use in SemiFab equipment is highly dependent on the cleaning, finishing and protection of the chamber surfaces and interior components. It is critical that these surfaces withstand the highly corrosive reactants and high energy plasma fields seen during etching and metal deposition operations.

Aluminum process fabrication components are frequently made from Al 6061. The components are protected from corrosion and plasma attack with a variety of oxide coatings. Alumina is the most common. Conversion of the aluminum surface to alumina can be most readily achieved through anodization, a well-known finishing technique for Al alloys. The type of anodization is determined by the amount of exposure of the component in question.

SemiFab equipment OEM's are most concerned with critical components that experience more direct and severe exposure. The corrosive reactor environment exposes the substrate through plasma arcing, corrosion, abrasion and erosion, leading to diffusion of undesirable contaminants into the process. Critical components require a more protective, controlled and tightly specified anodized layer. Each manufacturer has set limits for anodize layer hardness, corrosion resistance, di-electric strength, erosion and abrasion resistance.

Until recently, conventional Al anodization with sulfuric acid electrolytes has provided sufficient protection. With the desire for lower contamination, conventional anodization has reached its practical limits for mechanical properties and resistance to chemical erosion. Conventional oxide coatings can no longer mitigate contamination and failures due to the aggressive process reactions.

Next generation critical components will require higher levels of protection. The finishing industry has responded by improving on the sulfuric anodizing process, through tighter controls, and introducing new anodizing electrolytes. Electrolytes of oxalic acid or in combination with sulfuric can yield a modest improvement to the resultant anodize properties. These new and multi-acid electrolytes are widely called "high performance" anodizes.

High performance alumina layers are still limited by the alloying elements, structure and contaminants of the substrate material. The inherent inclusions, alloying elements and trace contaminants of aluminum alloys are converted into voids, defects and trace contaminants even with high performance multi-acid electrolyte anodization.

Higher purity and more durable reactor materials and coatings are a necessity for the next generations of semiconductor device fabrication.

III. A High Purity Aluminum Solution

High purity electroplated aluminum coatings have been used to protect ferrous and non-ferrous hardware and reactor components since the late nineties. The plating is 99.99% pure Al with high levels of corrosion resistance, high service temperature, excellent adhesion and no porosity. Screws, straps and flanges are coated with 0.0003 to 0.001" of pure electroplated aluminum. These aluminized components show extended service life and improved corrosion resistance. Current applications include semiconductor and solar equipment for etching and deposition.

Aluminum components that are more directly exposed to corrosive process reactants and plasmas are protected with ceramic coatings. These typically require high corrosion resistance, di-electric strength, admittance, abrasion resistance and adhesion. Protective oxides, such as alumina or yttria, are converted from the bulk surface and/or preferentially deposited on the critical surfaces of these components. Sulfuric acid anodization converts the Al alloy surface into alumina. The resultant anodize layers are well understood and commonly specified as a protective finish. Current specifications have been developed around the properties of sulfuric electrolyte anodizes, with higher protection governed by higher anodize thickness.

However, reactor components have increasingly lower tolerance for contamination while concurrently requiring higher service temperature, corrosion and di-electric properties. These new specifications challenge the performance limits of sulfuric anodization. High performance anodize layers have been developed from alternative electrolytes or by combining multiple electrolytes (for example oxalic or oxalic with sulfuric acids). While these can yield a modest improvement in corrosion resistance and di-electric strength, they are limited by substrate properties.

Reactor components are manufactured from 6000 series or 5000 aluminum alloy substrates. These materials have alloying elements, voids, inclusions and trace elements that affect the resultant anodize. Any substrate flaws are converted into voids, defects and contaminants in the resultant anodize layer. Voids and defects reduce the structural uniformity of the anodize layer. Contaminants limit the life of the component in actual service by lowering its performance (corrosion resistance and di-electric strength). In the worst case, contaminants from the substrate or in the anodize layer, may diffuse out into the process and contaminate the deposition or etch environment.

Higher purity aluminum substrates are available and represent another option for reactor components. These materials offer an improved substrate for fabrication of reactor components. However, they are typically custom poured and have exceedingly long lead times and exorbitant costs. Manufacturing techniques for these materials are not common in the supply chain, making them logistically challenging to integrate.

A better solution to this materials conundrum is to convert the surface of a reactor component with a pure, anodizeable aluminum coating. Most aluminizing techniques, such as vacuum deposition processes, do not yield anodizeable thick, dense or pure aluminized layers, and do not support uniform coating of complex geometries.

However, aluminum electroplating can uniformly deposit ultra-high purity, dense, and thick aluminum layers. AlumiPlate Inc. offers its 99.99% pure AlumiPlate® Al coating for plating semiconductor and solar fab components. With an innovative plating technique using the AlumiPlate Bond Layer™, electroplated Al can be deposited directly onto the aluminum alloy and can be fully anodized. The anodizing process may penetrate through to the substrate without undercutting or loss of adhesion.

Without the voids, inclusions, contaminants and alloying elements of Al alloys, high purity electroplated aluminum provides an ideal surface for anodization. The resultant anodize layer has the potential to significantly improve the performance envelope of current and next generation anodize electrolytes.

A variety of tests on the most critical properties were used to characterize and contrast anodize electroplated Al to anodized Al 6061. Better test performance is indicative of improved field protection for critical components.

IV. Experimental

High purity aluminum has the potential to unlock the full performance of standard and next-generation anodization. A series of tests were performed to investigate the properties of anodized electroplated aluminum. The tests were conducted using industry accepted methods and criteria, to contrast anodized Al 6061 with anodized electroplated Al.

Specifications for anodized layers to be used on critical components cover a wide variety of functional and engineering properties. Critical tests were selected to allow for a comparative analysis of anodized electroplated Al and anodized Al 6061 using standard and high performance anodize electrolytes.

To be more representative of actual components, sample specimens were prepared with different surface roughness ("smooth" and "rough"). Smooth specimens had a surface roughness of 8-12 Ra, in line with the roughness in seal and gasket areas of critical components. Rough specimens had a roughness of 180 - 230 Ra, to simulate the roughened areas of critical components that are preferentially coated with yttria or other cophase oxide coatings after anodization.

While high purity AlumiPlate® aluminum has been extensively tested by individual OEM's, the proprietary results have not been published before. This project was undertaken to make the data publicly available. A summary of the tests and methods performed is shown in Table 1.

Table 1: *Summary of tests performed and methods used.*

Test	**Method**
Coating Structure	ASTM B-244, ASTM B-487
Chemical Composition	GDMS ASTM F-1593
Corrosion Resistance	AlumiPlate HCl Corrosion Test SWI 8.2.4.12
Breakdown voltage	ASTM D-149-09 Method A, Short Time Test
Abrasion Resistance	FED STD 141 Method 6192.1, MIL-A-8625F, ASTM D-4060-10
Microhardness	ASTM E384, ASTM C-1327
Crazing	SEM 1000X min
Color	ASTM D-2244 CIELAB Scale
Adhesion Test	AlumiPlate Pull Test SWI 8.2.4.11

Two separate lots of sample specimens were plated in accordance with MIL-DTL-83488D Type I with 0.003" of high purity AlumiPlate® Aluminum using the AlumiPlate Bond Layer™.

Both plated and unplated Al 6061 specimens were then anodized with sulfuric, mixed acid and oxalic acid as follows:

a. Oxalic anodization per MIL-A-8625, TYPE III, CLASS 1, 0.0009" - 0.0012" with hot de-ionized water seal (94 - 98 °C & pH 5.5-6.5) for 1 hour per 0.001" of anodization thickness.
b. Mixed acid anodization per MIL-A-8625, TYPE III, CLASS 1, 0.0018" - 0.0024" with hot de-ionized water seal (96 - 99 °C & pH 5.6-6.2) for 1 hour per 0.001" of anodization thickness.
c. Sulfuric acid anodization per MIL-A-8625, TYPE III, CLASS 1, 0.0026" TO .0035" with hot de-ionized water seal (94 - 98 °C & pH 5.5-6.5) for 1 hour per 0.001" of anodization thickness.

Certified anodization services were secured from a well-known qualified vendor to the semiconductor industry.

Statistical analysis was performed by a statistician using analysis of variances (ANOVA) modeling and Minitab Version 16.2.4. Where possible, a desirable industry target for each test is referenced in the analysis.

V. Analysis

Purity

Purity is an essential property for anodic coatings. Anodic coatings with less contaminants and trace elements are expected to provide better protection to critical components in actual use. The lowest possible levels of contamination are desirable.

The purity of the anodize layer is impacted by both the substrate and the anodization electrolyte. Impurities, inclusions and alloy phases in the substrate are passed to the resultant anodize. Additionally, the type and concentration of the acid electrolyte can change the coating composition as well. For example, anodic coatings of sulfuric acid will have more sulfur than anodic coatings of oxalic acid.

High performance anodizes are purer with a higher percentage of the desirable Al and O species. However, the anodize layer is still affected by the underlying substrate. A pure Al substrate or surface should result in a higher purity anodize layer.

Glow Discharge Mass Spectroscopy (GDMS) was performed on various specimens by an accredited laboratory. GDMS testing is a mature and versatile technique widely used across many industries for direct determination of trace elemental composition on a variety of different materials and coatings.

GDMS testing confirms that electroplated aluminum is 99.99%, considered an ultra-high purity level. Contaminant and trace element levels are very low. Fe is the highest contaminant at a level of 35 ppm. These levels are associated with steel components that are also in the Al plating equipment. The Fe levels could be reduced further by limiting the Al plating line to critical Al components.

Trace element composition is shown in Figure 2.

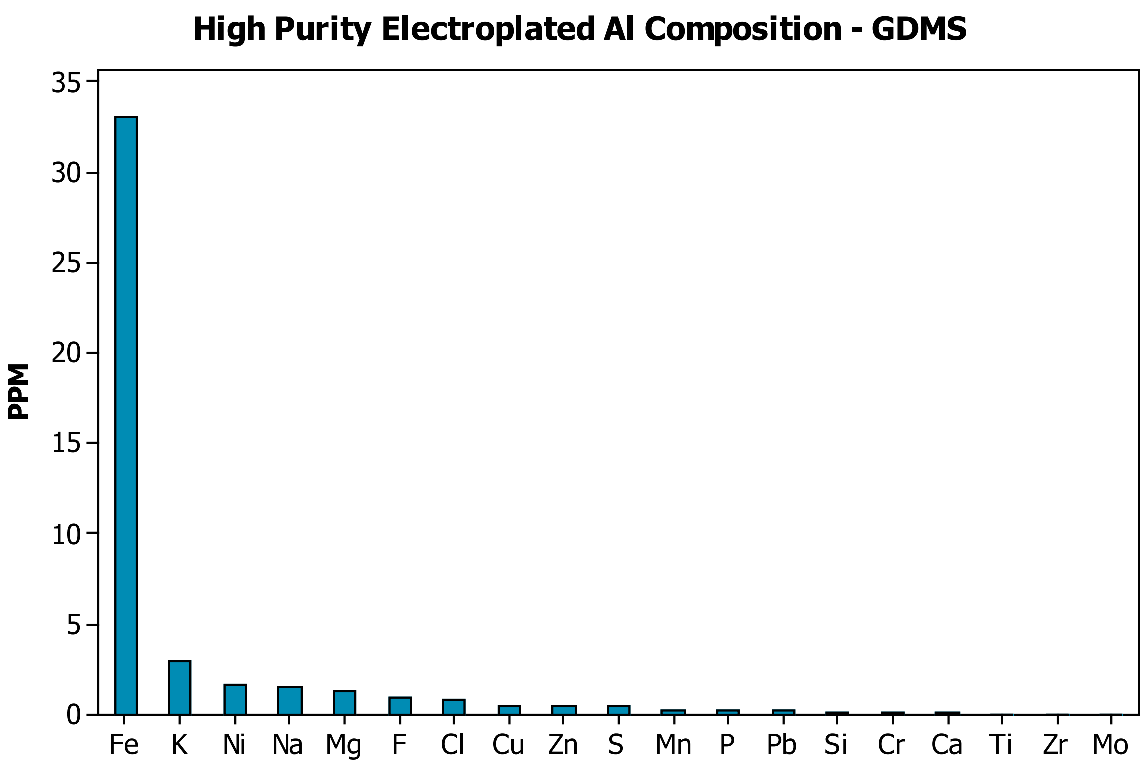

Figure 2: *GDMS elemental composition of high purity AlumiPlate Al. The plating is 99.99% pure.*

For reference purposes, the allowable composition levels Al 6061 are contrasted with the composition of AlumiPlate Al. Because of the much higher magnitude of alloys and contaminants in Al 6061, the differences are difficult to highlight through a bar graph and are better shown in tabular form (Table 2).

Table 2: *Summary of metal element composition for Al 6061 and high purity electroplated Al.*

Content (PPM)		
Element	Al 6061	Electroplated Al
Mg	85000	1.3
Si	68000	0.16
Mn	32000	0.27
Cu	22000	0.5
Zn	7000	0.5
Fe	7000	33
Cr	6000	0.14
Ti	5000	0.02
Others	15000	8.99

It follows that the much higher purity electroplated Al should yield a higher purity anodize layer than Al 6061. GDMS testing was completed on oxalic anodized specimens. Anodized electroplated Al showed significantly lower levels of metals than anodized Al 6061, as shown in Figure 3. Subsequent testing to follow in the next sections proves that the anodize electroplated Al layer has better corrosion resistance, due to its higher purity.

Additionally, diffusion of contaminants from the substrate and from the anodize itself should be limited for anodized electroplated Al. This is a key consideration for limiting contamination of the reactor environment as the critical component is attacked by process gases and plasmas.

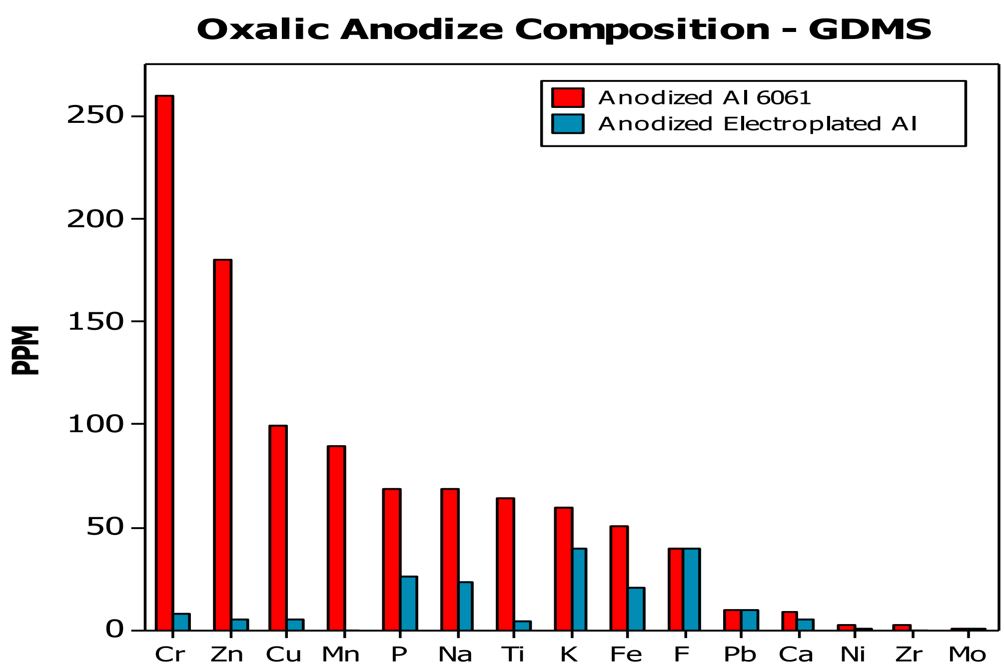

Figure 3: *GDMS analysis of metal contaminants in oxalic anodized Al 6061 and oxalic anodized electroplated Al. Note that the anodized electroplated Al shows significantly lower trace metal content.*

Structure

Cross sections of anodized Al 6061 and anodized electroplated Al were metallographically prepared and photographed by an accredited laboratory. The cross sections showed that the Al plating and resultant anodize were consistent and uniform in thickness, with only minor variations from the targeted deposition thickness.

No blistering, spalling, bubbling, nor separation indicative of poor adhesion were noted.

Differences in the structure of anodized Al 6061 vs. anodized electroplated Al were quite evident. Anodized electroplated Al showed a very sound and optimum anodize structure, free of voids or other defects. However, anodized Al 6061 contained voids and defects in its structure. These defects are most likely converted from voids, alloys and other contaminants in the Al 6061 substrate upon anodization.

Unlike Al 6061, high purity Al is free of voids, alloys and inclusions and thereby yields a superior anodize structure. This superior structure leads to the differences in corrosion, di-electric and mechanical performance between anodized Al 6061 and anodized electroplated Al. The improved structure should result in a better performing coating for protection of critical components.

Structure differences were noted for sulfuric, mixed acid and oxalic anodizations. Micrographs of the oxalic anodized coatings are shown in Figure 5 below. These are representative for mixed acid and sulfuric acid.

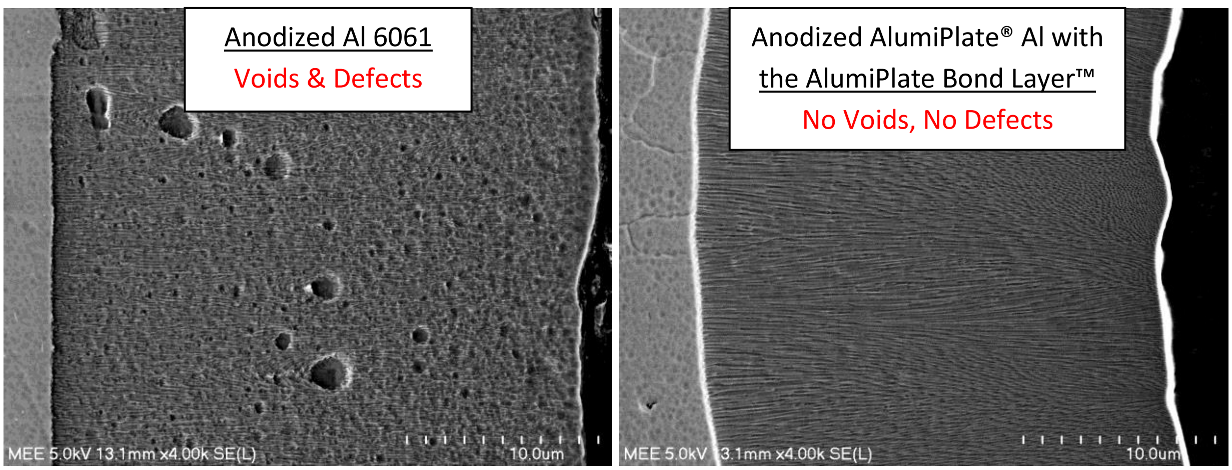

Figure 1: *Micrographs of anodized Al 6061 (left) and anodized electroplated Al (right). Note the converted voids and defects, a result of inclusions and impurities in the Al 6061 substrate. In contrast, anodized structure of electroplated Al is optimum, resulting from the conversion of pure layer free of voids and contaminants.*

Corrosion Resistance

Corrosion resistance testing was done using an HCl bubble test. This accelerated corrosion test is most useful for comparative analysis of different coatings. It is useful in predicting the relative resistance of coatings exposed to reactor acids and gases. The test was done per an AlumiPlate Work Instruction and can be described as follows:

A 1" long optically clear cast acrylic tube is epoxied to the face of an anodized aluminum plated coupon. Five weight percent HCl is introduced into the tube at a minimum depth of 0.6 inches. Using a microscope at 20X magnification, the sample solution is inspected once an hour for the formation of hydrogen bubbles. Failure is when a stream of continuous bubbles appears (one or more bubbles every 2 seconds) from one or more discreet points on the surface. The bubbles reveal the locations where the HCl has penetrated the anodize and is consuming the base aluminum.

The desirable industry target for HCl testing is a minimum of 4 hours until continuous bubbles appear. Some high performance anodizations reportedly reach 6 to 8 hours of exposure.

Statistical analysis determined that the surface roughness was a significant factor, with low surface roughness improving the corrosion resistance.

Overall, anodized electroplated aluminum shows significantly higher corrosion resistance than anodized Al 6061. The corrosion resistance is superior across the 3 types of anodization, with sulfuric anodize averaging 60 hours of exposure and both high performance anodizes reaching over 140 hours of exposure (see Figure 4). As expected, anodized Al 6061 averages 5 to 13 hours of exposure, with the mixed and oxalic outperforming sulfuric acid anodize.

According to HCl bubble testing, anodized electroplated Al offers a substantial improvement in corrosion resistance for critical components.

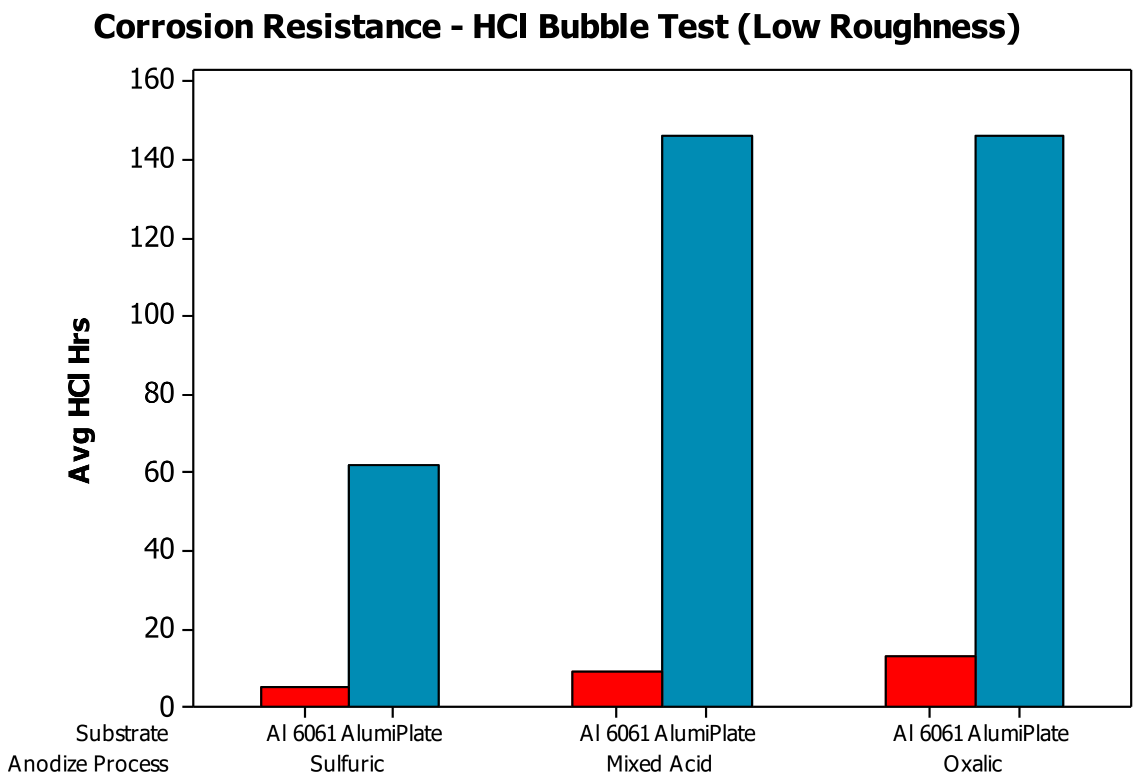

Figure 4: *Corrosion resistance measured by the HCl bubble test. Electroplated Al withstood in excess of 100 hours when anodized with mixed and oxalic acid electrolytes. It also outperforms sulfuric anodized Al 6061.*

Breakdown Voltage

Coatings for critical components must withstand the high voltages used to ionize reactor gases into plasmas. Coatings with high di-electric strength are required. The desirable industry target is 1,000 volts per 0.001" of anodize thickness, normally reported as "volts per mil". Where higher di-electric strength is needed, thicker anodize coatings are specified. High performance anodizes cannot be grown as thick as sulfuric acid anodization because of the self-limiting nature of the oxalic and mixed acid electrolytes. However, they are known to have higher di-electric strength.

These high performance anodizes are preferred for high tolerance features where the thick and more variable sulfuric anodize layer makes the fabrication process more difficult and expensive.

Di-electric strength was measured by an accredited laboratory using ASTM D-149-09.

For anodized electroplated aluminum, statistical analysis showed that specimens with low roughness had higher di-electric strength. Roughness effects might be related to a more variable anodize layer or a layer more prone to cracking when anodized over a rough substrate. To avoid confounding from extraneous factors, data from specimens with excessive cracking were excluded (greater than 0.001" inch crazing width).

The results for sulfuric and mixed acid anodizations were unexpected. Past OEM testing had shown higher di-electric strength for anodized electroplated Al on traditional and high performance anodizations. Even though excessively crazed samples were excluded, it is likely that crazing may have affected the results, with a resultant lower di-electric strength on samples with higher crazing density.

The best performing specimen was electroplated Al anodized with oxalic acid, averaging over 1,600 volts per mil. It is important to note that oxalic anodized electroplated Al has the potential to reach very high di-electric strengths. Some specimens had breakdown voltages higher than 2,500 volts per mil. The results are summarized below.

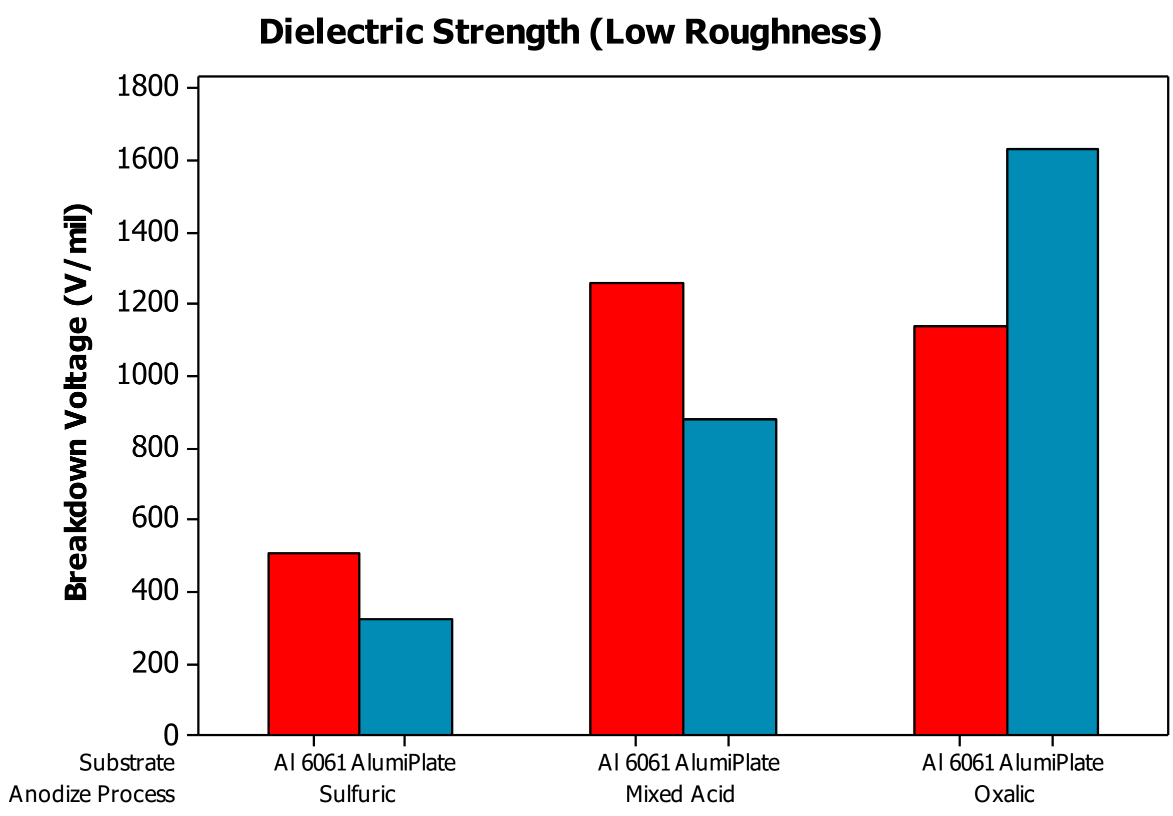

Figure 5: *Breakdown voltage of anodized electroplated Al and anodized Al 6061. With an oxalic anodization, Al electroplated samples averaged more than 1,600 V/mil. Individual values as high as 2,500 V/mil were observed.*

Abrasion

Abrasion is an important mechanical property for critical components. Abrasion measures the coating's ability to withstand rubbing, scraping and erosion. Taber abrasion testing per MIL-A-8625F Section 4.5.5 was performed by a NADCAP accredited laboratory. The desirable industry target is a maximum mass loss of 1.5 mg per 1,000 cycles.

The roughness of the specimen is a statistically significant factor, with lower roughness improving abrasion resistance.

Analysis of the results show that anodized electroplated Al performs consistently with acceptable abrasion resistance for all types of anodization. However, this is not the case for anodized Al 6061. Oxalic acid anodized Al 6061 shows very low abrasion resistance with mass loss 3X higher than desired.

The implications are important for the service life of critical components. When plated with high purity aluminum, anodized components can be expected to react consistently and predictably regardless of the type of anodization used.

The results are summarized in Figure 6 below.

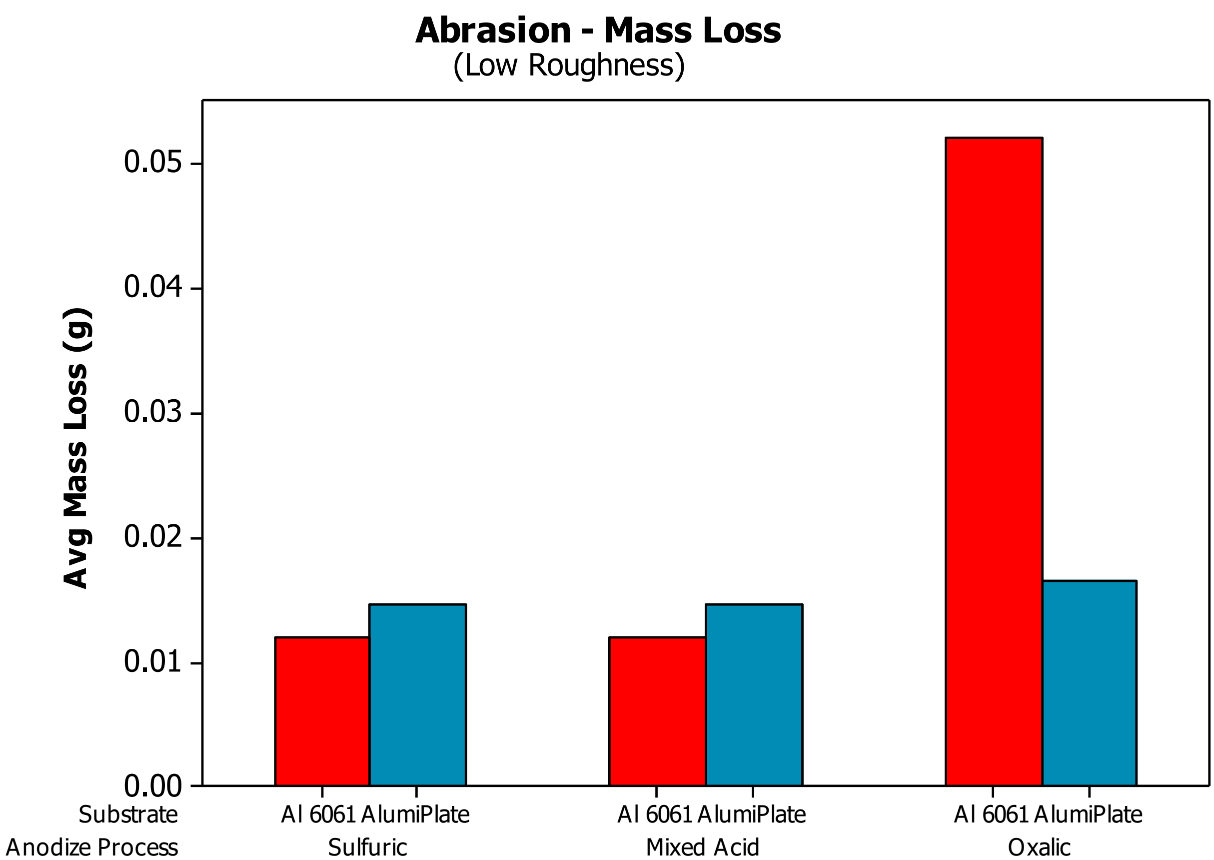

Figure 6: *Taber abrasion resistance of anodized electroplated Al and anodized Al 6061. The performance was similar except with oxalic acid, in which case electroplated Al showed better abrasion resistance.*

Microhardness

Microhardness is an important mechanical property for coatings. It measures the resistance of the coating to indentation. High microhardness is preferred to withstand scratches and handling during the assembly process. Microhardness is locally measured and assumed to be representative of the entire coating.

Microhardness was measured by a NADCAP accredited laboratory using ASTM E-384. Microhardness numbers were reported using the Vickers scale (VHN) and 100 g of load. Test anomalies such as insufficient coating penetration, tip region displacement and excessively large tip cracks were invalidated and not used for the statistical analysis. Duplicate samples were included to cover these expected test anomalies and ensure a sufficient volume of data points. The desirable industry target ranges from minimums of 350 to 400 VHN at 100 g of load.

In this test, roughness was not a statistically significant factor. Specimens with low and high surface roughness showed similar microhardness.

Anodized electroplated Al has the same or slightly higher microhardness vs. anodized Al 6061. More importantly, anodized electroplated Al showed high average microhardness with sulfuric and mixed acid ranging from 500 to 600 HVN. Oxalic acid was lower but close to the desirable industry value of 350 HVN. Refer to Figure 7 for a summary of the results.

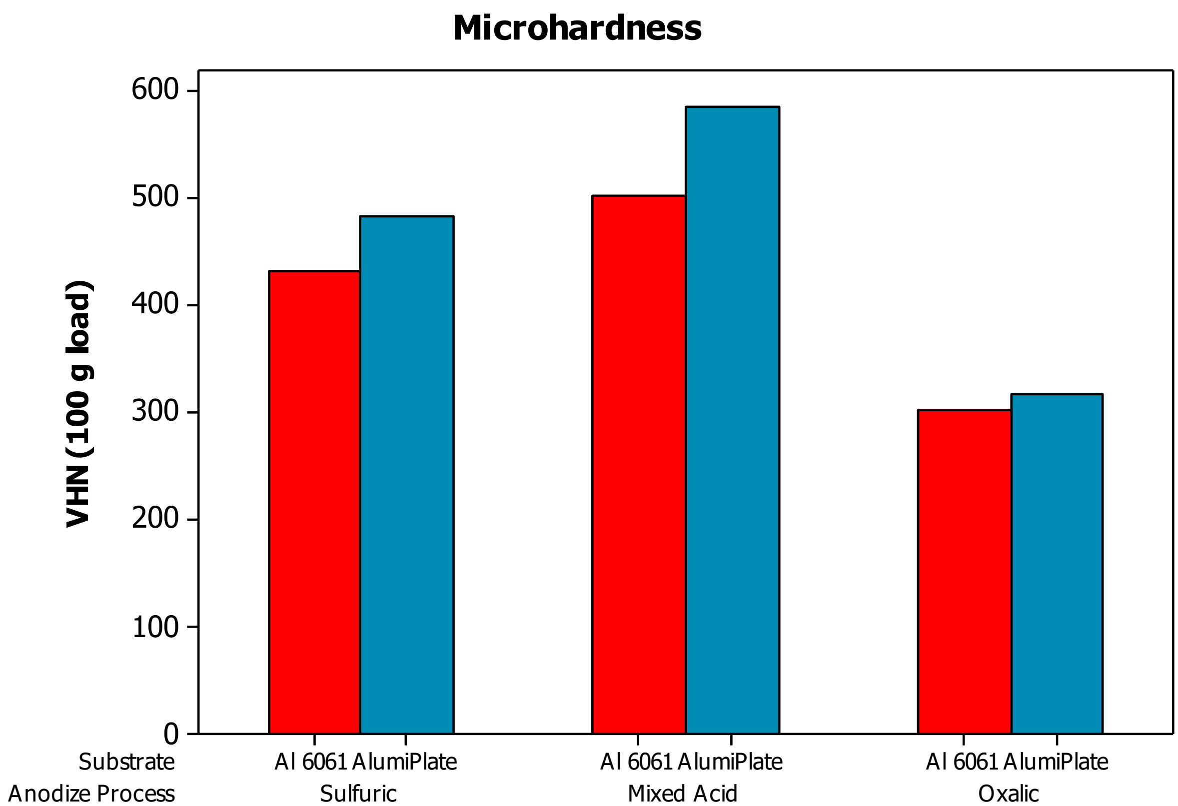

Figure 7: *Hardness of anodized and electroplated Al. Both populations were similar, with slight improvements in average hardness for anodized electroplated Al.*

Color

Color is an important consideration for anodic coatings. Uniformity of color is indicative of consistent workmanship and can provide quantitative acceptance criteria for the finished product.

ASTM D-2244 was used to measure the color differences and tolerances from the desirable industry targets. These targets are specified using the three coordinates of the CIELAB color space, which represent the lightness of the color. The targets are broken down into L*, a* and b* coordinates. Generally, the color of anodic coatings is required to be uniform and continuous, with each OEM specifying a specific color space. A common target color space is defined as CIELAB values of L* between 50 and 70, a* between -2 and 2, and b* between 5 and 20.

Analysis of the data showed that roughness did not statistically impact the color of the anodize. For the three anodize conditions, color was consistent and continuous on all samples. Anodized electroplated Al has higher L* values than anodized Al 6061. L* measures the black to white color axis. The difference is visually evident as an overall lighter color for anodized electroplated Al. The results are summarized in Table 3.

It can be concluded that, for this specific color space, components anodized with electroplated Al should be consistent in color across the 3 anodize electrolytes.

Table 3: *Color co-ordinates for anodized electroplated Al and anodized Al 6061. The anodized electroplated Al was consistent and continuous for all anodize electrolytes.*

Color Parameters		L*	a*	b*
	Common Target	50 to 70	-2 to 2	5 to 20
Al 6061	Sulfuric Acid	25.34	0.47	0.89
	Mixed Acid	26.925	-0.15	0.44
	Oxalic Acid	65.76	0.8	11.035
AlumiPlate Al	Sulfuric Acid	47.64	1.07	13.738
	Mixed Acid	62.68	0.287	18.492
	Oxalic Acid	76.185	-0.625	15.717

Crazing

Crazing in anodic coatings is a result of thermal or mechanical stresses during the anodization process or in service. According to the literature, the variables are numerous and include sealing, electrolyte temperature, current density, chemistry of the anodize electrolyte, anodize thickness, service temperature, thermal cycling, and even relative humidity. Because of the quantity of relevant factors, it is widely recognized that crazing cannot be eradicated. Since excessive crazing is deleterious to the performance of the anodic coating, the industry seeks to minimize it where possible. The desirable industry target range is approximately 0.0001" for maximum allowable crazing or crack width.

No conclusions could be drawn from the results since many of the relevant variables were not studied. However, roughness was statistically significant, with higher roughness associated with more cracking. For anodized electroplated Al, some crazing was noted with sulfuric and mixed acid electrolytes. No crazing was noted for electroplated Al anodized with oxalic acid. These haphazard results may also be related to workmanship issues during the anodization process.

For both anodized 6061 Al and anodized electroplated Al, the maximum crack widths observed were well below the desirable range. A summary of the findings is shown in Figure 8.

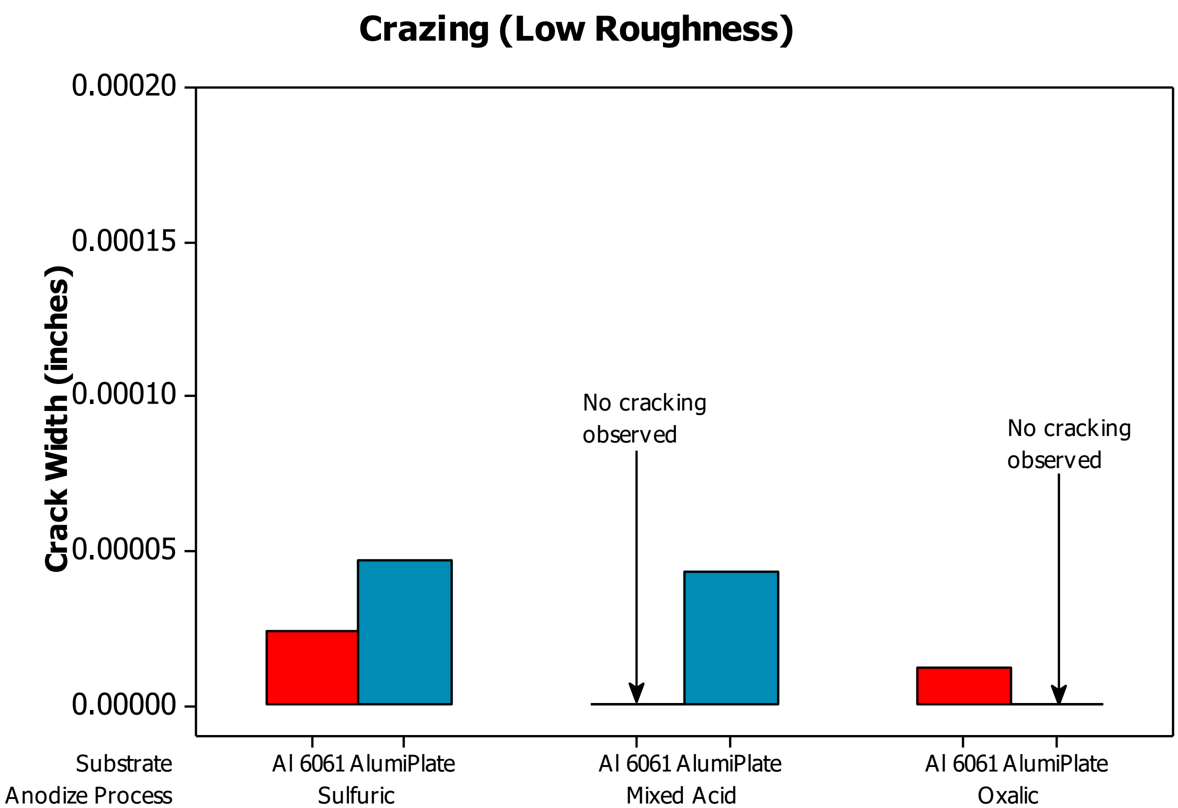

Figure 8: *Crack width diameters for anodized Al 6061 and anodized electroplated Al. For the samples showing cracking, the crack size was well below the desirable 0.0001" target.*

Adhesion

Adhesion is a critical property for any coating. Poor adhesion could lead to separation or delamination, exposing the substrate to the environment and voiding the coating's protective effects. There are no specifications for adhesion of anodic coatings because they are chemically grown from the substrate using anodizing techniques.

However, the desirable industry adhesion strength is 2.9 ksi (20 MPa) for yttria coatings used in critical components. For completeness of this characterization project, adhesion strength of oxalic anodized electroplated Al layer to the substrate was investigated.

The testing was performed per an AlumiPlate Work Instruction and can be summarized as follows: Using fixtures designed at AlumiPlate, the 10 mm diameter face of an aluminum dolly is glued to the aluminum plated coupons with an epoxy adhesive. After the epoxy has cured, a DeFelsko PosiTest AT-A Pull-off Adhesion Tester is attached to the dolly. Using hydraulic pressure, the Adhesion Tester pulls the dolly perpendicularly from the coupon. It measures the force on the dolly required to either separate the plated aluminum from the substrate or break the epoxy bond between the dolly and the plated aluminum.

The test method has higher than desired variability. Work is on-going to reduce the variability of the adhesion values. A boxplot graph summarizing the results is shown in Figure 9.

The results show that surface roughness of the substrate prior to plating is statistically significant, with low roughness yielding higher adhesion. However, the adhesion strength of the anodized Al plating was high for both conditions. Samples with a surface roughness of 210-250 Ra had adhesion strength of 6.41 ksi (44.2 MPa). Samples with a surface roughness of 6-9 Ra had adhesion strength of 8.56 ksi (59.0 MPa). Both are significantly higher than the comparable requirement for yttria.

The implication is that anodized electroplated Al will adhere satisfactorily to both smooth (seal surfaces) and rough areas (to be yttria coated) of critical components. Furthermore, the high adhesion should allow for facile integration of the Al plating into the fabrication cycle by providing a layer that can withstand downstream operations.

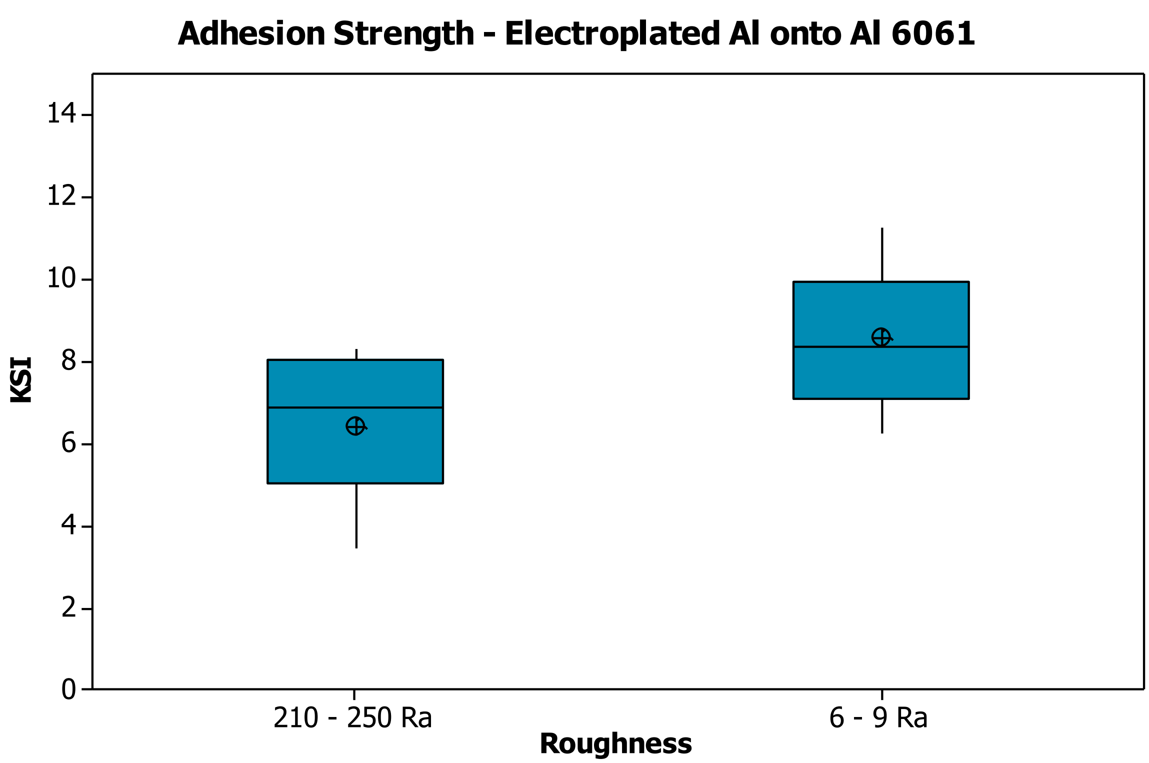

Figure 9: *Adhesion strength of oxalic anodized electroplated Al over smooth and rough substrates. The adhesion is high in both cases. Adhesion strength is statistically higher for smooth roughness conditions.*

VII. Conclusion

Ultra high purity electroplated aluminum coatings deliver superior performance for the semiconductor fabrication industry.

The purity of the coating is > 99.99% pure aluminum.

The metal content of the resultant oxide surface is greatly reduced. Zinc content is 5 ppm vs. 180 ppm in 6061.

Corrosion resistance is much better with any of three anodize acids tested, as high as 140 hours vs. 5-13 hours on 6061 anodized with either mixed or oxalic acids.

Di-electric testing shows promising results. High purity aluminum underperformed 6061 when anodized with sulfuric or mixed acids, but outperformed when anodized with oxalic acid. The low breakdown voltage may be associated with crazing observed on sulfuric and mixed acid anodized Al plated specimens. Values as high as 2500 V per 0.001" were measured for anodized high purity aluminum, which demonstrate the potential for a significantly increased performance level.

Abrasion resistance was equivalent to 6061 anodized with either sulfuric or mixed acids and was much better when anodized with oxalic acid.

Microhardness is equivalent when any of the three anodize processes are used.

Color uniformity is good with either substrate but high purity aluminum is generally lighter.

Analysis of crazing cracks did not demonstrate any consistent difference between high purity aluminum and 6061. The data suggests that crazing is a poorly controlled phenomenon with erratic results regardless of the substrate employed. Crazing may also be related to workmanship issues during the anodization process.

Superior, consistent surface attributes can be obtained independent of the substrate material used with an electroplated coating of high purity aluminum.

VII. Acknowledgements

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