DOE/NV/25946--1814

# ANODIZING OF HIGH ELECTRICALLY STRESSED COMPONENTS *

**Paul Flores**ξ
*Los Alamos Operations*
*National Security Technologies, LLC*
*Los Alamos, New Mexico, USA*

**David J. Henderson, Douglas E. Good, Keith Hogge,**
**Charles V. Mitton, Isidro Molina, Chris Naffziger**
*National Security Technologies, LLC*
*Las Vegas, Nevada, USA*

**Steve R. Codova, Eugene C. Ormond**
*Sandia National Laboratories*
*Albuquerque, New Mexico, USA*

*Abstract*

Anodizing creates an aluminum oxide coating that penetrates into the surface as well as builds above the surface of aluminum creating a very hard ceramic-type coating with good dielectric properties. Over time and use, the electrical carrying components (or spools in this case) experience electrical breakdown, yielding undesirable x-ray dosages or failure. The spool is located in the high vacuum region of a rod pinch diode section of an x-ray producing machine. Machine operators have recorded decreases in x-ray dosages over numerous shots using the reusable spool component, and re-anodizing the interior surface of the spool does not provide the expected improvement. A machine operation subject matter expert coated the anodized surface with diffusion pump oil to eliminate electrical breakdown as a temporary fix. It is known that an anodized surface is very porous, and it is because of this porosity that the surface may trap air that becomes a catalyst for electrical breakdown. In this paper we present a solution of mitigating electrical breakdown by oiling. We will also present results of surface anodizing improvements achieved by surface finish preparation and surface sealing. We conclude that oiling the anodized surface and using anodized hot dip sealing processes will have similar results.

***Keywords—Anodizing, sealing, dielectric strength, surface quality***

## I. INTRODUCTION

Anodized components in an electrically highly stressed environment eventually failed via electrical breakdown and were remanufactured to military anodizing specifications, which resulted in sooner-than-expected failure. Unfortunately, the original anodized components were stripped of the anodized coating so the pedigree of the original component was lost. In addition, original fabrication drawings existed, but while drafting new fabrication drawings, crucial details to the original anodizing process may have been changed or omitted. Oiling the surface seemed to mitigate the electrical breakdown of the components, therefore we suspected that there may be insufficient anodizing finish. In an effort to try to understand or recreate the original anodized surface, it is possible to imagine that the original components may have had a different finish from the typical military specification that would have increased the surface dielectric strength.

Anodizing is a process whereby aluminum and its alloys receive a conversion coating of *porous* aluminum oxide ($AL_2O_3$) on the surface. This aluminum oxide coating is a ceramic also commonly known as alumina. The coating is applied by passing DC current through the aluminum part (anode) in a chromic or sulfuric acid electrolyte bath. Military specifications for anodizing are given in MIL-A-8625 where sulfuric acid anodize and hard coat anodize are identified as Type II and Type III processes, respectively. Some reasons and benefits of anodizing are to [1]:

- Increase corrosion resistance, abrasion resistance, paint adhesion, emissivity
- Improve decorative appearance, adhesive bonding, lubricity
- Provide unique and decorative colors, electric insulation
- Detect surface flaws

---

* This manuscript has been authored by National Security Technologies, LLC, under Contract No. DE-AC52-06NA25946 with the U.S. Department of Energy. The United States Government retains and the publisher, by accepting the article for publication, acknowledges that the United States Government retains a non-exclusive, paid-up, irrevocable, world-wide license to publish or reproduce the published form of this manuscript, or allow others to do so, for United States Government purposes.

ξ email: florespa@nv.doe.gov

For the purpose of discussion, the electric insulating (or dielectric strength) is one benefit of the anodizing coating that is of most interest for use of high electrically stressed components in pulsed-power devices.

## II. COMPONENT FAILURE

The electrical component (rod pinch diode outer blank spool section), as depicted in Figure 1, is a high-vacuum anodized component of a dual axis radiographic source machine.

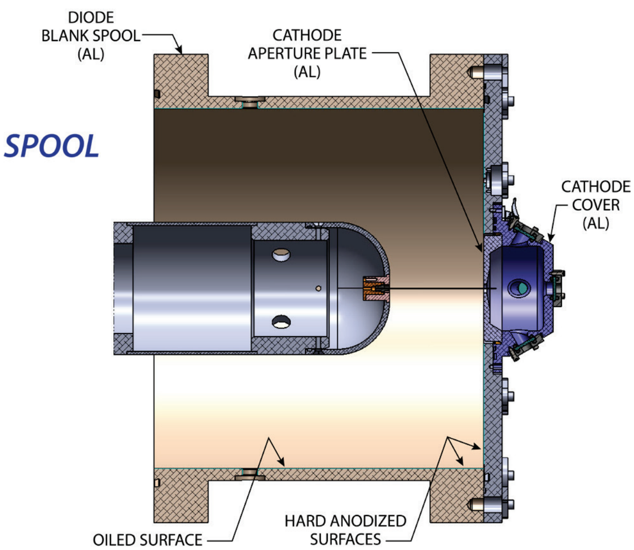


**Figure 1.** Diode outer blank spool.

The spool began to break down after roughly 1300 shots, resulting in unacceptable doses. Similarly, the identical component in the second axis broke down within a week of the first axis failure. Curiously, the second axis came into service within a few weeks after the first axis. The internal anodized surface of the spools were cleaned and visually inspected for damage, arc traces, or any other visible blemishes. Visible inspection of the spools revealed pitting on the surface due to the splatter of small particulates embedding into the anodized surface and impact of the cathode aperture plate coming off the cathode cover. Post-shot cleaning of the anodized surface typically requires cleaning or wiping spatter debris produced from the rod pinch discharge. This debris consists of tungsten, aluminum 6061, and other trace elements. The embedded particulates are typically removed during the cleaning of the spool and may leave contamination or conductive media in the remaining pit. Depending on the size of the pit, the indention into the anodized coating decreases the insulating thickness of the coating and could also be suspect to create electric breakdown. To reestablish pristine uniform coating thickness and dielectric strength, new diode spools were fabricated.

## III. NEW COMPONENT FAILURE

The new rod pinch diode outer blank spools were fabricated and installed on the radiographic source machine. Surprisingly, after approximately 20 shots the spools electrically broke down and failed. The spools were removed, and the anodized thicknesses were measured and reported to be below the target thickness of 0.002″. Unfortunately, anodized aluminum cannot be re-anodized to build thickness of hard coat over hard coat; additional coating, even as thin as 0.0005″ cannot be added to the surface [2]. Also, the lead machine operating subject matter expert was concerned about the component appearance (color and texture). This new spool did not look like the old spool.

In order to salvage the existing diode outer blank spools, new fabrication prints were created by NSTec to loosen the internal diameter tolerance to allow the machinist to strip the existing alumina oxide surface and prepare the internal surface for anodizing. NSTec opted for the military specification Type III for hard anodizing. The original spools were then stripped of the anodized coating to expose the bare aluminum required for anodization. This is the point where we lose pedigree of the original anodized surface. In addition, the internal surface quality of the spools was improved from a 32 RMS finish to a 16 RMS finish. This higher quality surface finish was recommended by the anodizing finisher to achieve an improved anodized surface finish. The new fabrication drawing also specified the use of a silicon carbide wet sanding operation to achieve a 16 RMS finish or better. We need to verify with the anodize vendors if any traces of silicon carbide that may become embedded into the aluminum can be etched out. We will anodize the two spools after we learn more about the anodizing process. More about the surface finish will be discussed in the anodizing study section.

## IV. OILING TO PREVENT ELECTRICAL BREAKDOWN

After they were anodized, the surface of the new spools appeared different than the original spools surface finish. The original anodized surface color appeared to be a darker brown/grey, whereas the new spools' anodized color was much lighter. Also, the surface of the original spools had a sheen on the surface finish, but the new spools looked dull. With the failure of these spools and the fact that operations of the radiographic source machines needed to continue, the machine operators applied diffusion pump oil to the diode internal surface for added protection. The problem with oils having higher dielectric strength is that many are not very compatible in high-vacuum environments. Diffusion pump oil is silicon-based oil, and it is used especially for vacuum components. Table 1 shows the dielectric strength for various materials at room temperature and normal atmospheric pressure. Silicon oils listed in the table are shown to have slightly higher dielectric strength than alumina, which is an added plus for the insulating surface.

The values given in Table 1 for dielectric strength of materials depend on sample thickness, the electrode shape, the rate of the applied voltage increase, the shape of the voltage vs. time curve, and the medium surrounding the sample. For electrical breakdown in a gas environment, the dielectric strength strongly depends on the electrode geometry, surface conditions and the gas pressure (or vacuum). Because our medium is a vacuum, any trapped air on the surface becomes the weak link for electrical breakdown [3].

**Table 1**. Dielectric Strength of Insulating Materials.

| **Substance** | **Dielectric Strength (MV/m)** | **Dielectric Strength (V/mil)** |
|---|---|---|
| Helium (relative to nitrogen) | 0.15 | 3.8 |
| Air (flat electrodes) | 3 | 76.2 |
| Alumina | 13.4 | 340.4 |
| Silicone oil, mineral oil | 15 | 381.0 |
| Transformer oil | 110.7 | 2811.8 |
| Distilled water | 65 to 70 | 1651 to 1778 |
| High vacuum (field emission limited) | 20 to 40 | 508 to 1016 |
| PTFE (Teflon, Extruded ) | 19.7 | 500.4 |
| Mica | 118 | 2997.2 |

In a strong electric field environment the air or gas free electrons acquire kinetic energy and collide with other atoms in the medium and ionize (impact ionization). Electrons are released by this impact ionization and become accelerated by the electric field and further collide with atoms. This chain reaction creates an electron avalanche effect that then becomes a plasma. The plasma containing these charged particles is electrically conductive, and the result is electrical breakdown or surface flashover [3].

Care was taken with the spool (Figure 1) to maintain a clean surface and create an even, uniform coverage or layer of oil. Diffusion pump oil, weighed in grams, was applied in carefully controlled amounts in final spool surface preparation for each shot rebuild. Initially, oiling was done to create an extra insulating barrier to the electric field created by the pulsed-power. As a result, the oil may have seeped into the pores of the anodized aluminum surface. By observation, oiling seems to be just another way of sealing a non-sealed anodized aluminum porous surface. To seal, or not to seal, that is the question. This idea will be discussed later.

## V. ANODIZING STUDY

The authors of this paper are not anodizing subject matter experts, but have pieced together information from web searches, forum discussions posted by users, and from telephone discussions with industry experts.

Aluminum anodizing is accomplished in five carefully controlled stages: (1) chemically cleaning to remove grease and surface dirt, (2) pre-treatment surface etching or brightening, (3) anodizing, (4) coloring, and (5) sealing.

In anodizing, there are three major types of processes.

- Type I (chromic acid anodizing) uses a chromic acid for the active agent
- Type II (sulfuric acid anodizing) uses sulfuric acid as the active agent.
- Type III (hard anodic coating) also uses a sulfuric acid alone or with additives as the active agent.

The differences in the three major types of processes are the thickness of the anodized coating produced and the ability to dye or color the surface. The differences between Type II and Type III processes are the operating temperature, the use of additional agents in the bath, and the voltage and current densities at which the anodizing is accomplished.

The principal military specification, which covers six processes including Type I through Type III, is MIL-A-8625, Anodic Coatings for Aluminum and Aluminum Alloys. The classes for color applications are Class 1 for non-dyed surface (typical of Type III anodic coatings) and Class 2 for dyed surfaces where the color is usually specified and the surfaces are sealed to lock-in the color.

Type III anodizing, the focus of this paper, produces a higher dielectric strength due to a thicker aluminum oxide coating as well as having less porosity than Type II coatings at similar thickness. The dielectric strength of hard anodic coatings is given to withstand up to 800 volts (DC) per 0.001″ (1 mil) thickness of coating [2]. Hard anodic coatings penetrate into the material approximately equal to the growth of the coating above the surface for a more uniform coating ranging from 0.0005″ to 0.0045″ depending on the alloy. Table 2 shows different thicknesses of Type III hard anodize for given alloys, including color variations.

**Table 2**. Type III Anodize Thickness Guide [4].

| **Alloy** | **Major Constituent** | **Maximum Thickness** (generally accepted) | **Color Overtones** (may vary) |
|---|---|---|---|
| 1100 | 99.5% pure Al | 0.003″ | Grey/Green |
| 2024 | Copper | 0.0015″ | Bronze |
| 3003 | Manganese | 0.002″ | Grey |
| 4032 | Silicon | 0.0012″ | Grey |
| 5052 | Magnesium | 0.0035″ | Grey/Brown |
| **6061** | **Magnesium/Silicon** | **0.003″** | **Dark Grey** |
| 6063 | Magnesium /Silicon | 0.004″ | Green |
| 6105 | Magnesium /Silicon | 0.0035″ | Grey/Green |
| 7075 | Zinc | 0.004″ | Bronze |

### *A. Growth and Color*

The growth of the anodic coating is in the form of a hexagonal cellular structure; within the each cell is a pore. Figure 2 shows such a structure. The rod pinch diode outer blank spool is made of 6061-T6 aluminum alloy and is capable of receiving maximum anodized thickness of 0.003″. As the aluminum oxide grows in thickness the pore diameter increases in size. Typically, over extending the maximum thickness or limits of dissolution will cause the anodic coating to easily chip and become lighter or milky in color [5]. Because the aluminum oxide is grown only from aluminum, the presence of non-aluminum alloying elements will affect the color of the anodic coating. Aluminum 6061-T6 alloy has composition limits as follows [1]:

| | |
|---|---|
| 0.40 to 0.8 Si | 0.7 Fe max |
| 0.15 to 0.40 Cu | 0.15 Mn max |
| 0.8 to 1.2 Mg | 0.04 to 0.35 Cr |
| 0.25 Zn max | 0.15 Ti max |
| 0.15 max others (total) | Balance is Al |

Because of the larger limit ranges primarily in copper, silicon, and manganese in the alloy, the colors could vary from part to part when the aluminum alloy material is from different supply sources. Also affecting color is pore size. Smaller pores in hard anodic coatings (resulting from lower temperatures, higher current densities and lower sulfuric acid concentrations in the anodizing process) contribute to a darker coating color [5].

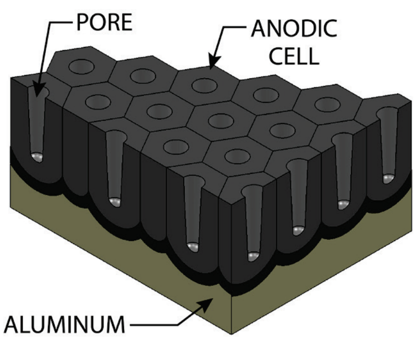


**Figure 2.** Anodic oxide coating with no sealing.

### *B. Growth Phenomenon and Surface Quality*

Aluminum oxide coating grows perpendicular to the surface of the part, and because there will be less coverage on the sharp corners the coating can be thinner in these areas. As with pitting, these inherent thin spots or voids become a localized site for low dielectric strength [6]. Unlike the obvious sharp corners on components, machining surface finishes will also have peaks and valleys (sharp points) when viewed under a microscope. With the growth of the coating in mind, the increased surface quality prior to anodizing may improve or create a more uniform coating thickness.

To further improve the machining surface quality, etching or surface brightening such as electro polishing may be employed to smooth out the peaks and valleys of the aluminum surface (on a microscopic level). Scratches or rough surfaces are accentuated during the anodizing process. After anodizing the surface finish will roughen by slight or moderate amounts depending on the surface quality and the coating thickness. With an average coating thickness of 0.002″, a pre-anodized surface finish of 2 RMS (mirror like) will roughen to approximately 4 RMS [7]. This is not an increase of double but an increase of 2 RMS in roughness.

The original spool's surface finish, specified as 32 RMS, was later changed to 16 RMS on the fabrication print. Recommendations of experts further suggested preparing the spool surface to a mirror 2 RMS finish if possible due to spool diameter size. This has been done, and we will anodize the spools after this study.

### *C. Porosity*

Porosity may play a vital part in dielectric strength. As shown in Figure 2, the anodic coating has pores as it comes out of the acid bath. The thicker the coating, the bigger the pores are at the surface. A typical anodized cell diameter is in the range of 50 to 300 nm, and pore diameter is about $^1/_3$ to $^1/_2$ the cell diameter. The density varies from 10 to more than 100 $\mu m^2$ [5].

With porosity, the anodized coating will absorb and hold dyes and colors well. With hard anodized 6061-T6 aluminum, the anodic coating color is a dark grey to black, which gives no reason to dye the part. Sealing is in most applications only needed to lock-in the color. But, if anodic coating can hold dyes, then it stands to reason that the open pores can retain air, contaminants, moisture, or any other conductive media that can penetrate into the pores and become trapped. These trapped contaminants eventually release under high vacuum, which is not good. Electrical breakdown studies in vacuum have shown that outgassing from improperly conditioned surfaces in a vacuum may induce electrical breakdown [8].

Diffusion pump oils with low vapor pressure and low viscosity have been applied to the internal surface of the spools having unsealed hard anodic coating and have shown to mitigate electrical breakdown. So, if oiling helps reduce electrical breakdown frequency by filling the pores, then why not seal the pores initially?

### *D. Sealing*

Sealing is the last step in the anodizing process and can be done by several different methods, though all methods do the same thing: close the porous aluminum oxide layer after the anodizing step. Non-sealed anodic coating will feel sticky and will absorb dirt, grease, oil, and stains.

The simplest sealing process takes place in boiling deionized water, which begins a precipitation of hydrated aluminum oxide known as pseudoboehmite. The pseudoboehmite fills the pores, and during the last stage of the sealing process it crystallizes to form a clear and hard boehmite (*2 AlOOH)*; see steps shown in Figure 3 [9]. Boehmite color is white to pale greyish brown, yellowish or reddish when impure, and colorless in thin sections. The hardness of boehmite is 3.0 to 3.5 on the

Mohs scale, whereas diamond is 10; 8 on the Mohs scale is approximately 60 to 65 Rockwell C [10]. The hard anodized coating for most aluminum alloys are considered "file hard" and are between 65 and 70 Rockwell C [2].

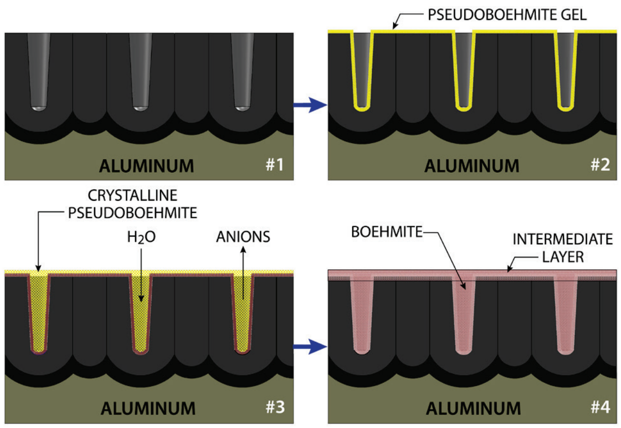


**Figure 3.** Hot deionized sealing steps.

In our spool example, maintaining the hard, wear- and abrasion-resistance is important in mitigating the sticking effects of the splattered particulates onto the surface. The source of the splatter is the result of the rod pinch diode physics vaporizing the tungsten anode explosively. Sealing may help further mitigate the splatter sticking to the spool surface. As seen in our unsealed hard anodized spool, the splatter sticks to the surface like Velcro.

### *E. Tradeoff*

The tradeoff between the hardness of the aluminum oxide vs. the dielectric strength is weighed based on the application of the component. For the diode outer blank spool component, the dielectric strength takes precedence over abrasion resistance. Further testing of sealing is needed on the diode spool in order to verify or compare the dielectric strength and electrical breakdown to that of an unsealed surface.

## VI. ANODIZED TEST COUPONS

To test the breakdown voltage of hard anodized 6061-T6, sample coupons were fabricated with a machined surface finish of 16 RMS and sent for hard anodizing. The coupons were tested using the HIPOT test for breakdown voltage and the results are shown in Table 3. To be clear, the dielectric strength values given in Tables 3 and 1 are not the same voltage comparison. The choice of the nature and value of the test voltage, AC or DC, is determined via the standards which apply to product being tested.

The AAI cleaning is a proprietary cleaning process of Applied Anodizing, Inc. (AAI). The process removes unwanted alloy elements from the surface of the part and exposes the aluminum. In both cases, the hot deionized

**Table 3.** Dielectric Strength of 6061-T6 Type III Anodized Coupons.

| | **Anodizing Process** | **Average Coating Thickness (mil)** | **HIPOT Test** | |
|---|---|---|---|---|
| | | | **Max. Breakdown Voltage (DC)** | **Volts/mil** |
| 1 | AAI Cleaning + No Seal | 2.3 | 1300 | 565 |
| 2 | Electropolish + AAI Cleaning + No Seal | 2.2 | 1200 | 545 |
| 3 | AAI Cleaning + BDI Seal | 2.2 | 1750 | 729 |
| 4 | Electropolish + AAI Cleaning + BDI Seal | 2.2 | 1750 | 795 |

water sealing showed an improvement in the dielectric strength. In our test coupons, the electro polishing surface preparations show improvement between the sealed coupons but not the others. After anodizing, the colors of the coupons were all black with slight differences in sheen between the electro polished and other coupons. Also, there was no significant difference in appearance between the sealed and non-sealed coupons.

## VII. THE RIGHT ANODIZING PROCESS

The anodizing finish notes on the fabrication prints were not defined in enough detail to accomplish the intent of the coating, which is to provide the highest dielectric strength for the surface. The intent here is not to decide the perfect anodizing note for the pulsed-power community. If five exact parts were sent to five different anodizing shops, chances are the five different finished parts will vary in color overtones, dielectric strength and thickness variations.

There are many factors in anodizing that affect the thickness, porosity, and color of the hard anodized coating. To achieve good and consistent results, a high level of quality control in each stage of the anodizing process must be taken. It is important to begin with a good surface finish followed by quality control of the anodizing baths. Strictly controlling the bath conditions, such as the processing time, temperature, pH level, agitation, location of cathode, and current density, are all important to obtaining quality anodic coatings. Table 4 shows how changing the sulfuric acid concentration, bath temperature, and current density affect parameters of the anodic cell growth.

Different fabrication processes resulting in softening or hardening the surface could affect anodic coating growth. AAI studied the effects of temperature as the temper of the aluminum material could be affected by machining operations. With today's high-end metal cutting machines and better tooling, fabricators are capable of machining or removing large amounts of material, so the surface temperature increase. The increased temperature may cause topical annealing, which allows the grain structures in the alloy to grow larger and softer resulting in an increase in growth of the anodic coating. Alternately, fabrication operations such as tube drawing through a die may work-harden the material, creating smaller grain structure in the material, and the anodic cell growth could then decrease.

**Table 4**. Effect of Changing Anodizing Process Parameters [5].

| Parameter | As you *Increase*/ *Decrease* Concentration | As you *Increase*/ *Decrease* Temperature | As you *Increase*/ *Decrease* Current Density |
|---|---|---|---|
| Dissolution | Increases/ Decreases | Increases/ Decreases | Decreases/ Increases |
| Pore Size | Increases/ Decreases | Increases/ Decreases | Decreases/ Increases |
| Hardness | Decreases/ Increases | Decreases/ Increases | Increases/ Decreases |
| Thickness | Builds Slower/ Builds Faster | Builds Slower/ Builds Faster | Builds Faster/ Builds Slower |
| Clarity of Coating | Clearer/ Less Clear | Clearer/ Less Clear | Less Clear/ Clearer |

MIL-A-8625F provides anodizing acquisition requirements for fabrication documents to specify. Remember, you only get what you ask for.

## VIII. OTHER PROCESSES IN INDUSTRY

Plasma electrolytic oxidation (PEO), also known as micro arc oxidation (MAO), is an electrochemical surface treatment process for generating oxide coatings on metals. Coatings produced by the MAO process are harder, less porous, and smoother than Type III anodic coatings. The pores range from 10 to 100 nanometers. The ceramic MAO finished surfaces have dielectric properties that anodized surfaces do not. Another advantage of MAO is that it coats the interior of long cylindrical shapes. Uniformity is difficult with anodizing cylinders if not properly racked with the cathode placement crucial during the anodizing process. MAO finishes work with new applications which have failed with anodized surfaces in the past due to their diversity [11].

## IX. CONCLUSIONS

Hard anodizing is a suitable coating for electrical carrying components located in the high-vacuum rod pinch diode section of a radiographic source machine, provided the surface is adequately prepared and the coating is sealed. If sealing is uncertain, then oiling with vacuum-compatible oil will help seal the anodized pores and reduce or minimize outgassing from the pores. Although it has been concluded that sealing is advantageous for improving the dielectric strength of the surface, in-situ testing is needed to validate this claim. Also, alternate processes such as PEO or MAO should be considered and tested.

## X. ACKNOWLEDGMENTS

We greatly appreciate the expertise of Applied Anodizing, Inc. in helping with the anodized coupons and providing feedback and test results. We also thank KAEHR Plating for taking time to talk about anodizing and make recommendations for improvements to our components. We also appreciate PGI machine shop for lending us the book "Aluminum How To: The Chromating–Anodizing–Hardcoating Handbook."

## XI. REFERENCES

[1] M. F. Stevenson, ASM Handbook, Volume 5, Surface Engineering, Anodizing. PA: ASTM International, pp. 483-493, 1994.

[2] MATWEB, "Aluminum anodizing process and design guide," www.matweb.com/reference/anodize

[3] L. I. Berger, "Dielectric strength of insulating materials," http://library.sciencemadness.org/library/index.html.

[4] "Anodizing reference guide," Aluminum Anodizers Council, www.anodizing.org.

[5] R. H. Probert, Aluminum How To: The Chromating–Anodizing–Hardcoating Handbook. 2005.

[6] "Effects of sharp edges, and corners on anodizing," Alpha Metal Finishing Company, http://alphametal.com/faq-s#effects.

[7] "Hard anodizing of aluminum alloys and its effecs on Bal™ Seal performance," Ball Seal Engineering, Technical Report TR-17 Rev. C, 03-22-01.

[8] Y. Saito, "Vacuum systems of high-energy accelerators: electrical breakdown in vacuum," KEK-High Energy Accelerators Research Organization, 2003.

[9] "Why the sealing is so important," Anodizing World, http://aluminumsurface.blogspot.com/.

[10] "Boehmite," http://en.wikipedia.org/wiki/Boehmite.

[11] "Anodizing versus ceramic coating," http://www.thomasnet.com/articles/custom-manufacturing-fabricating/.

[12] MIL-A-8625F Military Specification, Anodic Coatings for Aluminum and Aluminum Alloys, 1993.