SIGNIFICANT CRACKING PROPENSITY REDUCTION OF HARD ANODIZED 7075 AND IMPROVED WEAR PERFORMANCE OF ANODIZED 2024 OBTAINED THROUGH CRYOGENIC TREATMENT.

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Abstract.
The benefits of an additional cryogenic treatment in 7075 hard anodized and 2024 anodized coatings were evaluated. Comparative investigations between 7075 standard hard anodized and 7075 hard anodized with an additional cryogenic treatment were conducted. The cryogenic treated coating had significantly lower amount of cracks, the cracks were shorter and with smaller crack facets opening. The state of stress was calculated and proved that the cryogenic processed hard-anodized coating is more reliable.
The wear performance of 2024 standard anodized and 2024 anodized with an additional cryogenic treatment was evaluated using a Taber Model 503 Abraser. The tensile and compressive stresses developed during such test at the contact surface were established using the Hertzian theory of elastic contact. The wear rates were calculated by finding the area losses of the anodized coatings at the OD, center and ID of the wear track. The remaining anodized coating areas were determined from SEM views using a CAD Area Measurement System. The additional anodized 2024 cryogenic treated had an impressive 88% - 633% better wear performance than the standard 2024 anodized coating.

INTRODUCTION.

One of the shortcomings of anodized coatings particularly the thicker ones is the development of fissures.
It was widely accepted by the Anodizing Manufacturers that the performance of anodized coatings is controlled by the following parameters:

1. The dielectric constant of the alumina film;
2. The solubility of the alumina film in the electrolyte;
3. The stability of the electrolytic solution;
4. The operating temperature;
5. The state of stress before and after anodizing.

The basic reaction in all anodizing processes is the conversion of the aluminum surface to alumina while the part is the anode in an electrolytic cell. Alumina like all ceramic materials is sensitive to a state of stress that contains tensile components.
During the experimental work described in this paper the first four parameters listed above were carefully selected and never changed.
For comparative reason the “state of stress before and after anodizing” was kept like always (standard) or diminished through cryogenic treatment.

The following comparative investigations of hard anodized 7075 and anodized 2024 with and without cryogenic treatment were conducted:

- Evaluation of crack initiation and propagation, crack morphology, number of cracks per one cm., length of cracks and opening of crack facets in hard anodized 7075 aluminum alloy.
- Residual stress calculation of the hard anodized 7075 materials.
- Wear performance evaluation of anodized 2024 alloy using a Taber Model 503 Abrader apparatus.

Alloy 7075 and 2024 are complex Al-Zn and Al-Cu materials with a broad range of applications and they are frequently anodized. The chemical composition of the two alloys is shown in Table 1. {1}

Table 1.
Chemical of 7075 and 2024 aluminum alloys.

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
7075	0.40	0.50	1.2 2.0	0.30	2.1 2.9	0.18 0.28	5.1 6.1	0.20	Rem.
2024	0.50	0.50	3.8 4.9	0.30 0.90	1.2 1.8	0.10	0.25	0.15	Rem.

COMPARATIVE CRACKING PROPENSITY INVESTIGATION OF HARD ANODIZED 7075 WITH AND WITHOUT AN ADDITIONAL CRYOGENIC TREATMENT.

Most of the cracks in the anodized layer originate from the top surface and transversally propagate toward the aluminum body. During this comparative investigation two anodized 7075 aluminum samples were used. Both samples were extracted from a 4-inch bar stock, machined with the same 12 UM rugosity and hard anodized in the same batch. One sample had an additional proprietary cryogenic treatment.

Appendix 1 shows the SEM view at X500 magnification of the standard hard-anodized sample. The thickness of the hard anodized coating is 60 UM or 0.0023 inch.

In the 185 UM length of the standard hard anodized sample three different crack morphologies are noticed. One crack completely penetrates the hard anodized coating – see Arrow A, one crack is interrupted and also penetrates through thickness – see Arrow B while two arrested cracks have a length of 20 UM – see Arrow C. Approximately 230 cracks with different length were counted in one cm. length.

It is obvious that a large stress gradient is present in this small field shown in Appendix 1. Arrow Y indicates the residual stress increase of the standard hard anodized coating. The paper will comment on this subject later.

It also appears that the crack formation and propagation is not drastically influenced by the discontinuities of the hard anodized layer. One explanation of this peculiar crack propagation in alumina is that atomic bonds are a rare condition.

Appendix 2 is a SEM view of the interrupted crack at X2000 magnification. The newly formed branch shown with Arrow D continues its propagation toward the aluminum body as well.

Appendix 3 is a SEM view at X500 magnification of the hard anodized and cryogenic treated 7075 aluminum sample. Only one small hardly visible crack is present – see

Arrow E. The length of the crack is 15 UM. The total number of cracks counted in one cm. length is between three and five.
The cryogenic treated hard-anodized 7075 sample has the following advantages over the standard practice:

1. The number of cracks was dramatically diminished, from 230 to an average four;
2. The standard sample has two cracks, which penetrate through the hard anodized coating one continuous while the other is interrupted.
3. The length of the cracks in the cryogenic treated anodized layer is 15 UM which is smaller than the two 20 UM arrested cracks developed in the standard hard anodized sample.
4. The crack opening of the cryogenic processed sample is very small – it is barely noticed while the two cracks of the standard hard anodized coating which penetrate through thickness have a 1 UM crack facets opening.
5. Because the additional cryogenic treatment reduced dramatically the number of cracks, the length of cracks and the opening of the crack facets the reliability of such hard anodized coatings will improve.

In order to diminish the damaging effect of surface cracks the Anodizing Industry developed a large variety of "surface sealings". Nonetheless a sealed anodized surface has a limited wear resistance because usually it is quickly removed under sliding or other possible wear conditions.

COMPARATIVE STRESS CALCULATION IN A STANDARD HARD ANODIZED COATING VERSUS AN ADDITIONALLY CRYOGENIC TREATED HARD ANODIZED COATING.

According to the Griffith criterion crack propagation occurs if the energy released during crack growth is sufficient with the energy required for the crack growth. The elastic energy release rate per crack tip "G" is also called the crack driving force.{2}

$\sigma_f = K_{IC} / (\pi.a)^{1/2}$ [1]

σ_f = failure stress;
K_{IC} = plane strain fracture toughness in mode I opening;
a = crack length.

The alumina range of plane strain fracture toughness in mode I opening is:

K_{IC} = (3.58 – 2.69) MPa(m)$^{1/2}$ or (3.25 – 2.69) KSI(inch)$^{1/2}$

These values were published by the University of Oxford – England and seem the most reliable ones.
The following were the values used for calculating the stress in this application:

- 0.0023 inch the crack length through thickness of the 7075 standard hard anodized coating and 0.00057 inch the crack length of the 7075 additional cryogenic treated hard anodized coating.
- The average alumina plane strain fracture toughness range of 3.13 KSI (inch)$^{1/2}$.
- 400 MPa or 43.5 KSI the tensile strength of alumina.

- σ_a and $\sigma_{w/crayo}$ are the failure stresses of the standard hard anodized coating and respectively the failure stress of the hard anodized coating with cryogenic treatment.

$\sigma_a = 3.13 / (3.14 \text{ x } 0.0023)^{1/2} = 3.13 / (0.007222)^{1/2} = 3.13 / 0.085 = 36.82$ KSI

$\sigma_{w/crayo} = 3.13 / (3.14 \text{ x } 0.00057)^{1/2} = 3.13 / (0.0018)^{1/2} = 3.13 / 0.0425 = 73.65$ KSI

The relationship between the residual stresses at failure with and without cryogenic treatment, the crack length in the hard anodized layers with and without cryogenic treatment and the tensile strength of alumina is shown in Appendix 4.
Area I is considered the area without failure of the anodized layer and it is confined by the maximum residual stress value of 73.65 KSI and $5.7\text{x}10^{-4}$ inch crack length. The cryogenic treated hard anodized coating confined in Area I is reliable and provides increased service performance.
Area II exists within a residual strength range of (73.65 – 43.50) KSI and ($5.7\text{x}10^{-4}$ – $17.25\text{x}10^{-4}$) inch crack length range. In this area failure commences.
Area III defines the catastrophic failure of the hard anodized coating; the residual strength is below the tensile strength of alumina while the cracks penetrate through the thickness of the standard hard anodized coating.
Based on this calculation it is expected that the cryogenic treated hard anodized layer should have a significantly lower wear rate.

ESTABLISHING THE EXPERIMENTAL WEAR TESTING CONDITIONS OF ANODIZED 2024 WITH AND WHITHOUT CRYOGENIC TREATMENT.

Two 2024 aluminum plates with a 0.25-inch diameter hole in the center were anodized together. The plates are 4-inch square and 0.062 inch thick.
One plate was standard anodized while the other plate had an additional cryogenic treatment. The average thickness of the anodized layer was 15 UM or 0.0006 inch.
The following are the reasons for selecting a 15 UM thick anodized layer:

- To comparatively investigate with the Taber Abrader the wear damage using a reasonable amount of cycles.
- Usually 15UM-anodized coatings are crack free therefore the wear tests are accomplished on high integrity anodized coatings. This was indeed accomplished - Appendix 5 shows two SEM crack free views at X1000 magnification of the 2024 anodized plates used in this experimentation.

Both plates were abraded with the same Taber apparatus using 1000 cycles, which proved enough to partially remove the anodized coatings and some of the aluminum body as well.
It is widely accepted that understanding the relationship between wear properties and surface texture helps to establish the optimum manufacturing process for various surface needs. In this work the benefits of the additional cryogenic treatment are evaluated by the areas of wear loss, which are proportional to the amount of wear. This technique is

frequently used to evaluate worn areas of gear teeth, bearing retainers, sliding pads with contoured surfaces, etc.
The wear losses of the anodized layer presented in this paper are quantified through surface losses using comparative X500 magnification SEM areas at the OD, center and ID of the wear track induced in the anodized coating with the Taber apparatus.
The average surface of the anodized layer before starting the Taber wear test at X500 magnification is 3205 UM^2. This is the reference surface.

WHY WAS THE TABER TESTER SELECTED, HOW DOES IT WORK AND WHAT ARE THE TYPE AND SIZES OF STRESSES DEVELOPED ON TOP OF THE 2024 ANODIZED SURFACES.

The Taber Model 503 Abraser is a durable and precision build “two-body” abrasion apparatus used by many Industries including the Airspace Industry and Armed Forces to evaluate the sliding wear performance of anodized coatings. Basically the Taber test is a “running-in wear” process.
Appendix 6 shows two views of the Taber Abraser. The following are the main components:

1. A 70 RPM rotating table on which the anodized aluminum piece is mechanically attached.
2. A pair of non-engaged rubber wheels with a 0.5-inch width. In order to obtain reliable results the wheels are refaced after each test.
3. Vacuum nozzle, which absorbs all the debris.
4. A pair of auxiliary wheels.
5. The anodized aluminum testing plate.

At the contact point of both wheels with the testing plate a normal 1000 g (2.2 lb) compressive force P is acting along the common normal.
The tangential force Q in the tangent plane is sustained by friction according to the equation:{3}

$$Q \leq \mu \cdot P \qquad [2]$$

μ = 0.50 coefficient of friction established when considering an alumina fixed piece and a sliding alumina pin.
Q = 2.2 x 0.50 = 1.1 lb

The Hertzian theory of elastic contact established that the radial stress is tensile at the very edge of the wheel contact with the testing plate while all the other stresses are compressive.{3}

$$\sigma_t = P_0 (1 - 2\nu) / 3 \qquad [3]$$

$$\sigma_c = P_0 (1 + 2\nu) / 2 \qquad [4]$$

σ_t = the tensile strength;
σ_c = the compressive strength;
ν = 0.22 the Poisson’s coefficient of alumina;
P_0 = the maximum contact pressure per wheel.

Theoretically the contact between the wheel and the testing plate is liner however practically it is a small area. In this regard a 0.0005-inch contact width was selected across the 0.5-inch thick non-engaged wheels.

$A = 0.0005 \times 0.5 = 0.00025$ inch^2
$P_0 = 2.2 / 0.00025 = 8800$ lb / inch^2
$\sigma_t = 8800 (1 – 2 \times 0.22) / 3 = 2933 (1 – 0.44) = 1642$ PSI
$\sigma_c = 8800 (1 + 2 \times 0.22) / 2 = 4400 (1 + 0.44) = 6336$ PSI

During the Taber test cyclic straining are loaded in the anodized coating.
At starting point of the Taber test the tensile stress component is 1642 PSI however when the anodized surface is roughened up this value will change.
Because the two non-engaged wheels are rotating in different directions the tensile stresses generate a pattern of crossed arcs over a 30-cm^2 area.
Appendix 7 shows the two different rotating directions of the non-engaged wheels and the expected crossed arcs abrasion marks.

COMMENTS ON THE COMPARATIVE WEAR TESTING RESULTS OF STANDARD AND CRYOGENIC TREATED 2024 ANODIZED COATINGS.

Establishing the views of microscopic examination.
Like earlier stated the goal of this experimentation was to comparatively quantify the damaged area of the standard and additionally cryogenic treated anodized layers near the OD, center and ID of the wear track.
The left column of Appendix 8 are photographic views at the end of the wear test of the two 2024 anodized plates. The right column shows the dissection pattern employed to identify the damage of the anodized coating with and without cryogenic treatment near the OD, center and ID of the wear track. The two missing pieces identified with Arrow 3 were glued on the corresponding piece 1. The two-paired pieces were polished together and viewed from the same direction – see Arrow V.

Wear pattern topography.
Appendix 9 shows the standard and cryogenic processed anodized coatings X2.87 magnification. The two photographic views of the 2024 anodized wear tracks were taken after completing the 1000 cycle Taber abrasion test. The location of both pictures is in the immediate vicinity of the transversally cut shown with Arrow V in Appendix 8.
The wear pattern topography of the 2024 cryogenic treated anodized coating shown in Photo A is significantly smaller than the wear damage of the 2024 standard anodized coating presented in Photo B.
The wear pattern topography of the standard anodized coating shows a rough and vitreous topography, which usually generates high wear rates.
Despite the great material loss difference between the two anodized coatings both samples show more or less crater formation damage.

During the Taber test the standard anodized coating broke through and smeared aluminum on the top of the wear track, which filled up partially or completely some of the wear craters – see Arrow A.
The wear rate on both plates is higher toward the ID of the wear track where tensile stresses are present. However the anodized coating likewise all ceramic materials is sensitive to high contact stress or any contact conditions leading to a state of stress that contains tensile components. These sequence of events generated wear failure conditions across the wear track of the standard anodized coating like seen in Photo B of Appendix 9.

Comparative wear measurements of the standard and cryogenic treated coatings.
SEM microscopic analyses and profile measurements are probably the most used technique to reveal wear damage and change in surface finish.
In this regard Appendix 10, Appendix 11 and Appendix 12 show comparative SEM wear measurements through area losses at the end of the Taber test. The areas of 2024 standard anodized coating and 2024 cryogenic treated anodized coating were measured at X500 magnification using a CAD Area Measurement System.
The results are presented in Table 1 and Appendix 13.

Table 2.

Area losses and remaining areas of the 2024 cryogenic treated and standard anodized coatings of the wear track samples on the OD, center and ID. The remaining anodized areas express the wear resistance after completing 1000 Taber test cycles.

Sample position on the wear track	**Area loss [UM^2]**		**Remaining area [UM^2]**		**Wear resistance improvement of cryogenic treated sample [%]**
	Standard	**Cryo**	**Standard**	**Cryo**	
OD	2715	115	490	3090	633
Center	2380	1501	825	1704	207
ID	2748	2346	457	859	88

The wear improvement of the 2024 anodized coating obtained through cryogenic treatment is within an impressive range of 88% - 633%.
During the 1000 cycle Taber test the two 2024 anodized coatings are without cracks, delamination or other defects. This proves that the anodizing process of both samples was properly accomplished.

Chemical composition check of the 2024 standard and cryogenic treated anodized coatings and aluminum base.
Generally after completing a wear test it is recommended to follow up with a chemical composition check of the tested surfaces. If chemical composition alterations occur most likely contaminations or structural changes were present.
Appendix 14 shows the qualitative chemical compositions found with EDAX of A - the 2024 cryogenic treated anodized coating while B is the 2024 body, C – 2024 standard

anodized coating with D the 2024 aluminum body. The chemical composition was checked in the middle of piece 1 shown with Arrow V in Appendix 7.
The cryogenic 2024 anodized coating has the following differences:

- The amount of oxygen is higher;
- Magnesium reading were detected;
- The sulfur peak is slightly smaller.

At this time the chemical composition differences between the cryogenic and standard 2024 anodized coatings are not discussed.

FUNDAMENTALS OF CRYOGENIC PROCESSING UTILIZED AT A.R.WILFLEY.

For years some of the castings used in the Wilfley centrifugal pumps are cryogenically processed. The field performance of these parts was equivalent or better than the competitors' heat-treated version.
Appendix 15 shows a basket of cryogenically treated wear resistant castings. The cryogenic process was designed to assure superior wear performance by providing identical hardness in each point of the casting and uniform hardness through wall thickness.
During the cryogenic treatment the following favorable microstructure changes occurred:

- Optimize the shape, size and distribution of the primary and eutectic C_6M_7 chromium carbides;
- Precipitate secondary carbides in the matrix;
- Convert the inhomogeneous austenitic matrix into martensite.

The cooling of castings / materials by the refrigerating agent is mainly accomplished through forced convection because thermal conductivity at low temperature is minimum. The heat extraction takes place on the thermal boundary layer. Appendix 16 is a backside view of the Wilfley 10 ft x 10 ft x 5 ft cryogenic unit, which was designed to provide uniform temperature within the processing chamber.

CONCLUSION.

1. The 0.0023-inch thick cryogenic treated 7075 hard-anodized coating has significantly lower cracking propensity than the standard 7075 anodized coating. The number of cracks per one cm. length was dramatically diminished from 230 to four, the length of the remaining cracks reduced to about 15 UM and the crack facet openings minimized.
2. Using the Griffith criterion it was found that the residual strength value in the cryogenic treated 7075 hard-anodized coating is considerably higher than 43.5 KSI the tensile strength of alumina. Under these conditions the 7075 cryogenic treated hard anodized coating is reliable and could be used without sealing.
3. The comparative sliding wear performance of 2024 anodized coating with and without cryogenic treatment was tested with the Taber Model 503 Abraser. The average thickness of the anodized coating was 0.0006 inch. At the end of 1000 cycles the wear performance was evaluated by calculating the remaining areas of the anodized coatings at the OD, center and ID of the wear track. The remaining areas of the anodized coatings were calculated from SEM pictures using a CAD

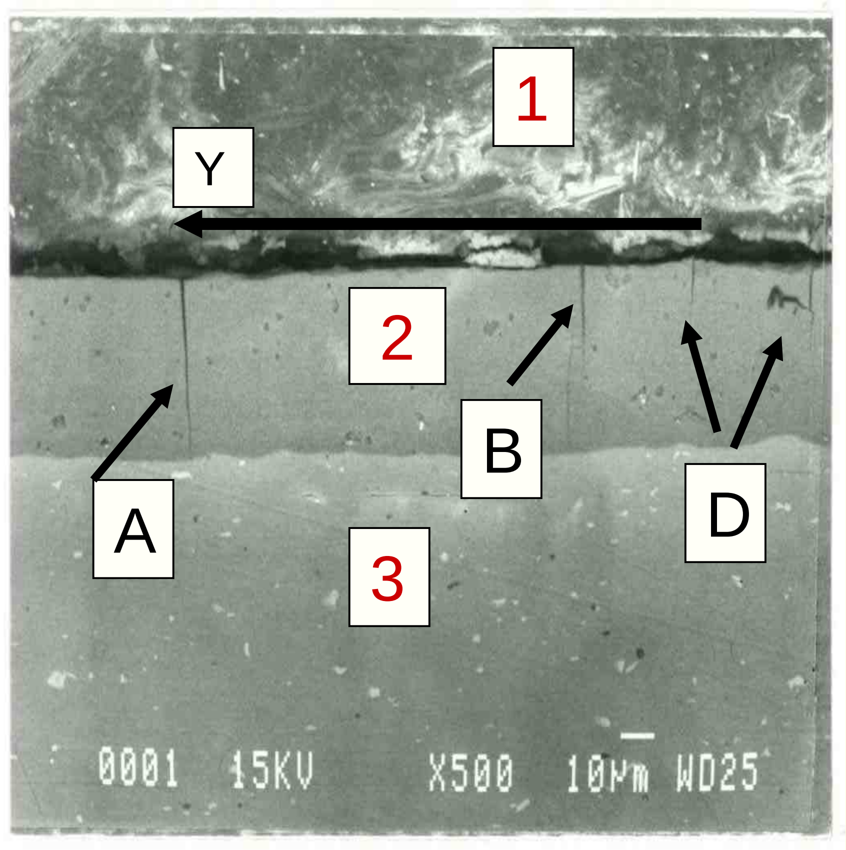
Area Measurement System. Both 2024 anodized coatings are without cracks, delamination or any other defect. This assured repeatable wear testing conditions.

4. Based on the Hertzian theory of elastic contact the tensile and compressive stresses were calculated for which the 2024 hard anodized and cryogenic treated coating had 88% - 633% better wear resistance than the 2024 standard anodized coating.
5. The EDAX chemical composition of the cryogenic anodized coating is different than the standard anodized coating – the oxygen content is higher, magnesium is present while the sulfur content is slightly lower.

Bibliography.

1. ASM Specialty Hand Book "Aluminum and aluminum Alloys" pag.20 and pag.23
2. Broek David "Elementary engineering fracture mechanics" Third revised edition – pag.16 and pag.6.
3. Johnson K.L. "Contact mechanics" Printed in Great Britain at the University Press Cambridge – pag.204 and pag.94.

APPENDIX 1



SEM view of multiple crack morphologies developed in the standard hard anodized coating.

One crack completely penetrates the anodizing coating – see Arrow A, one crack is interrupted and also penetrates through thickness – see arrow B while two cracks have a length of 20 UM- see arrow C.

Arrow Y indicates the residual strength increase along the top surface of the standard anodized coating.

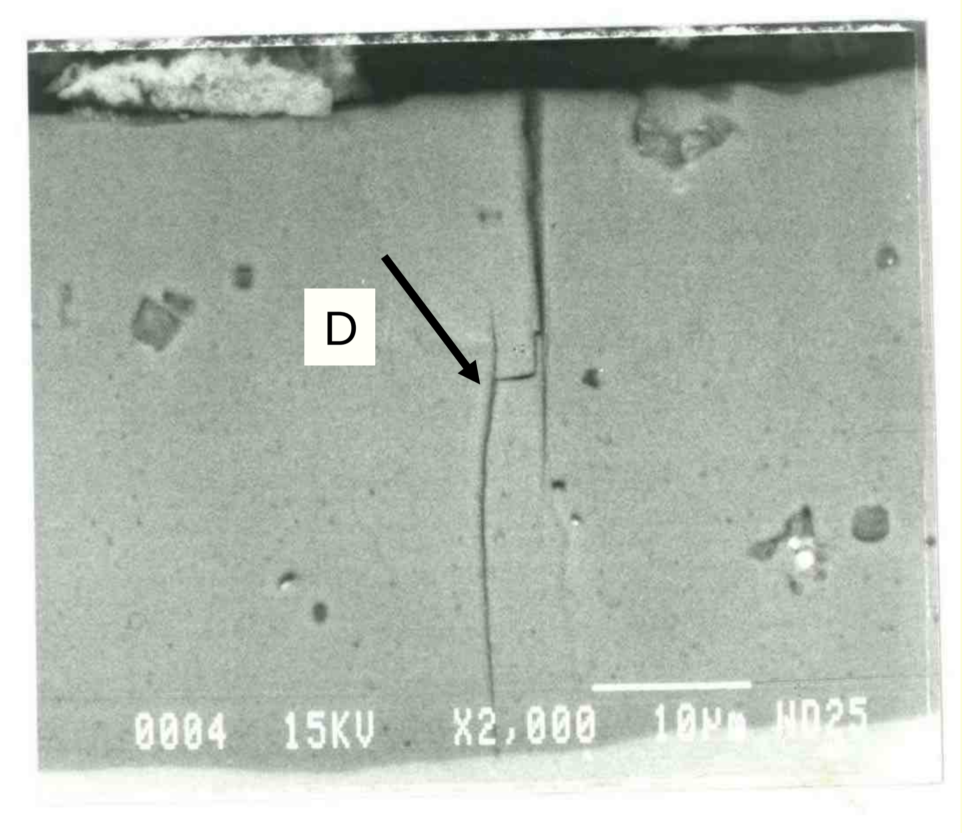
Around 230 cracks were counted in one cm length of the standard anodized layer.

1 – Bakelite mount.

2 – Hard anodized layer.

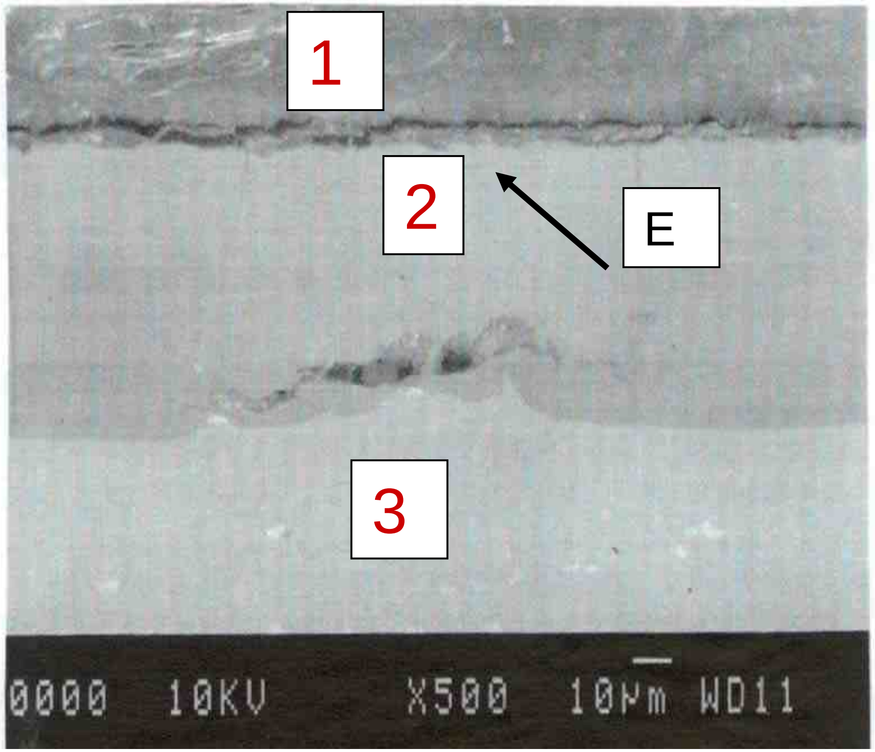
3 – 7075 Aluminum body.

APPENDIX 2



SEM view of the interrupted crack at X2,000 magnification. The newly formed interrupted crack shown with arrow D in the standard anodized coating continues its propagation toward the aluminum body.

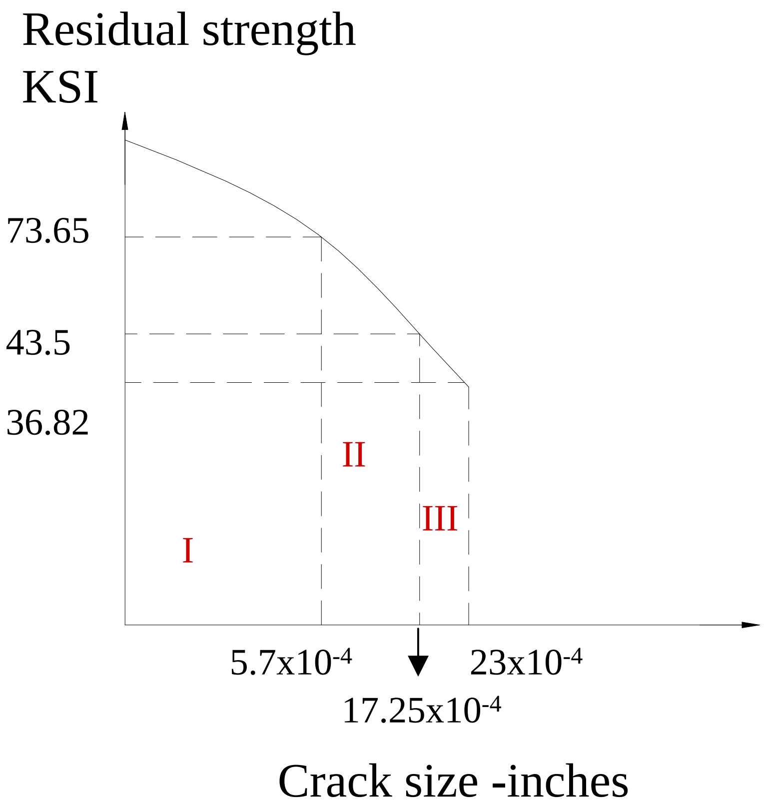
APPENDIX 3



SEM view at X500 magnification of the hard anodized and cryogenic treated 7075 aluminum sample. Only one small hardly visible arrested crack is present – see Arrow E. The length of the crack is 15 UM. The total number of cracks counted in one cm length is between three and five.

1. Bakelite mount
2. Hard anodized layer
3. 7075 aluminum body

APPENDIX 4



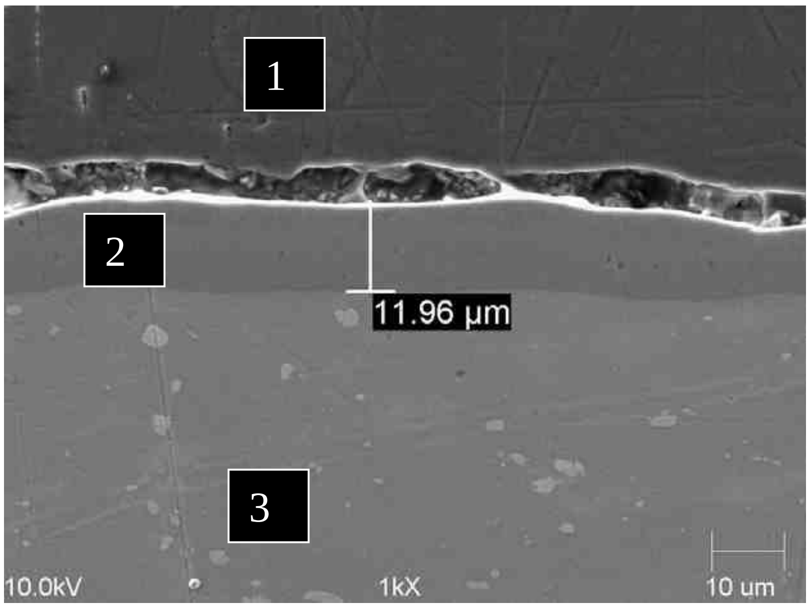
Area I – is the area without failure of the hard anodized layer. This area is confined by the maximum residual stress value of 73.65 KSI for a crack length of 5.7×10^{-4} inch. Such occurrence of very small arrested cracks was basically achieved when the hard anodized coating was cryogenic treated.

Area II – exists within a residual strength range of (73.65-43.50) KSI and a (5.7×10^{-4}-17.25×10^{-4}) inch crack length range. In this area failure commences.

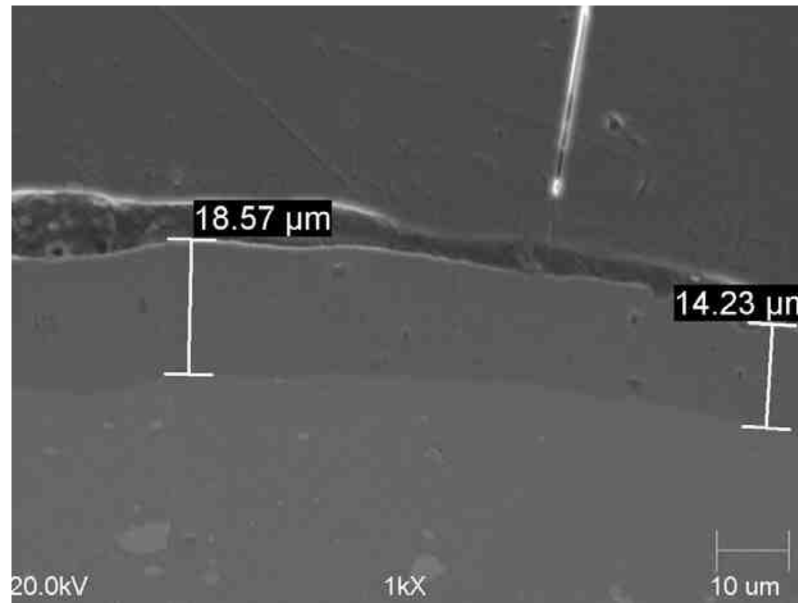
Area III – defines the catastrophic failure of the anodized coating. The residual strength is below the tensile strength of alumina while the cracks penetrate through the entire thickness of the standard anodized coating.

APPENDIX 5

The two transversally dissected SEM views at X1000 magnification of the 2024 anodized samples are without any crack. The Photos were taken before the Taber wear test started. The average thickness of the anodized layer is 15 UM or 0.0006 inch.



A



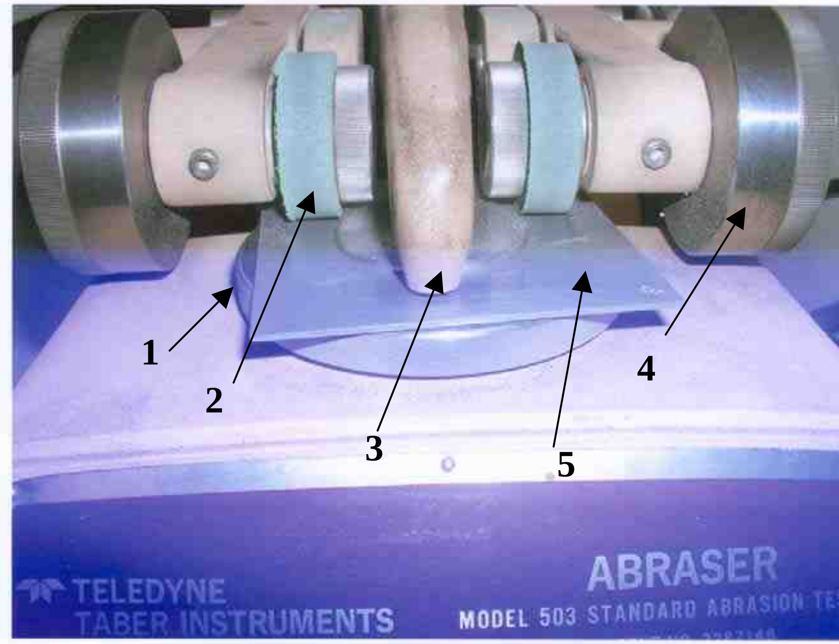
B

1- Bakelite mount

2- Anodized layer

3- 2024 aluminum body

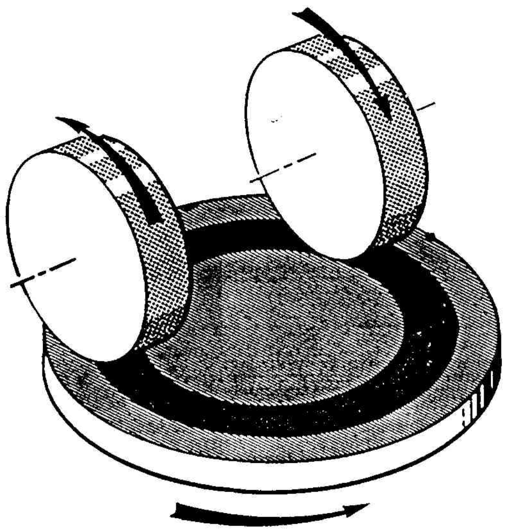
APPENDIX 6
View of the Taber wear testing apparatus.

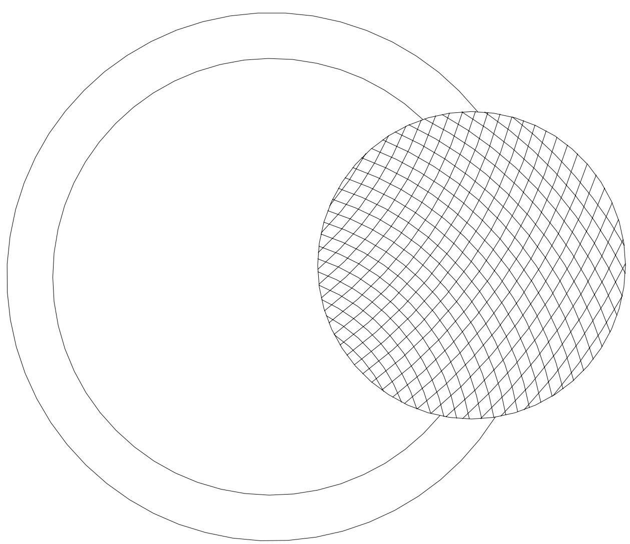


The following are the components of the Tabor Abraser:

1. A 70 RPM rotating table on which the anodized aluminum piece is fixed.
2. A pair of non-engaging rubber wheels. In order to obtain reliable wear test results the wheels are refaced after each test.
3. Vacuum nozzle, which absorbs the debris.
4. A pair of auxiliary wheels.
5. The anodized aluminum testing plate.

APPENDIX 7
View of the wear track generated by the Taber Model 503 Abraser.





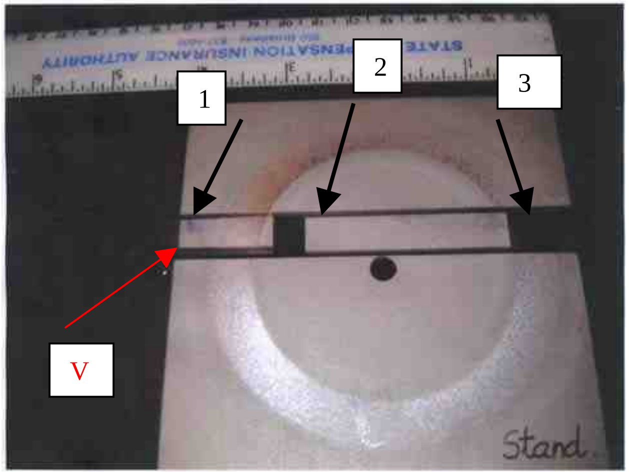
The 30 cm^2 track wear is generated by the two non engaged wheels. Theoretically the tensile stress produced by each wheel is intersecting and forms a mesh pattern.

APPENDIX 8

View of the dissected pattern employed to investigate the area losses in the 2024 anodized coating with and without cryogenic treatment after competing the 1000 cycles Taber test.

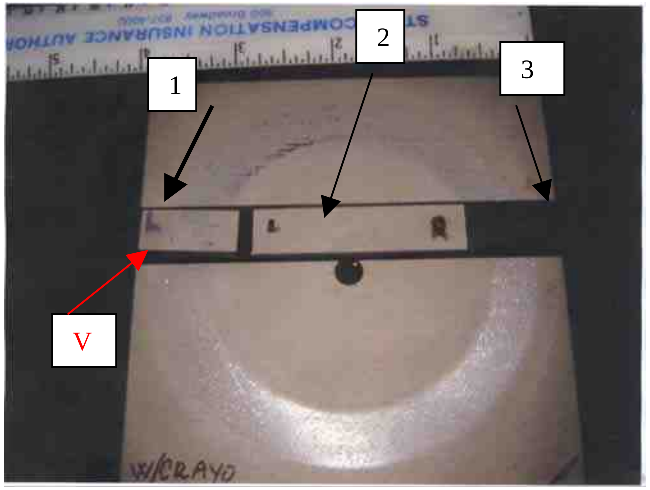
The missing piece identified with Arrow 3 was glued on piece 1.The two pieces were polished together and viewed from the same direction – see arrow V.

A-Standard anodized 2024 tested plate.



B-Standard anodized 2024 tested and dissected plate.

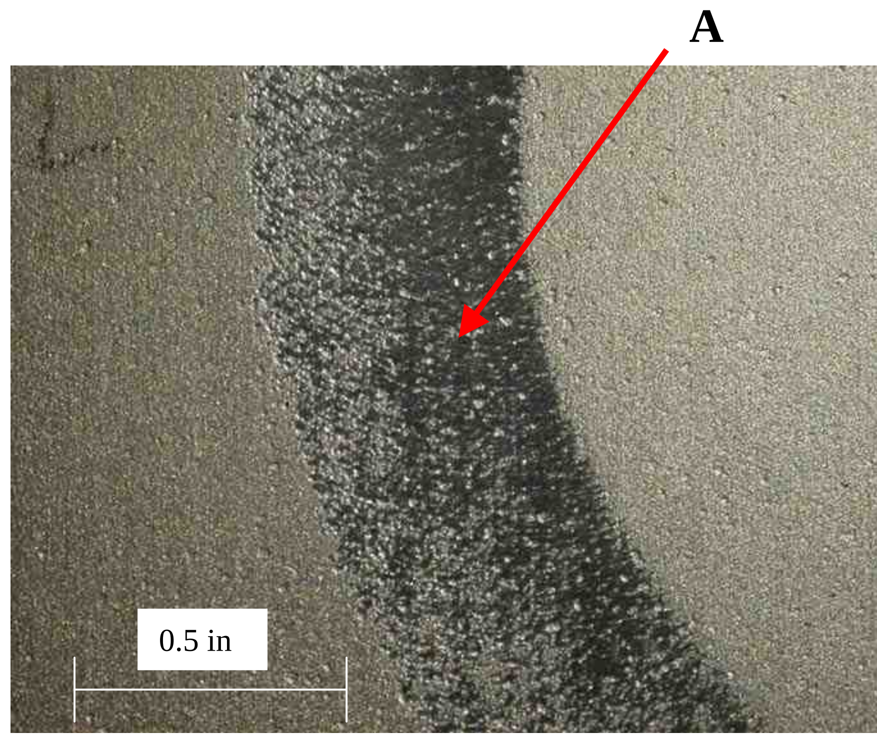
C-Additionally cryogenic treated 2024 tested plate.



D-Additionally cryogenic treated and dissected 2024 tested plate.

APPENDIX 9

A

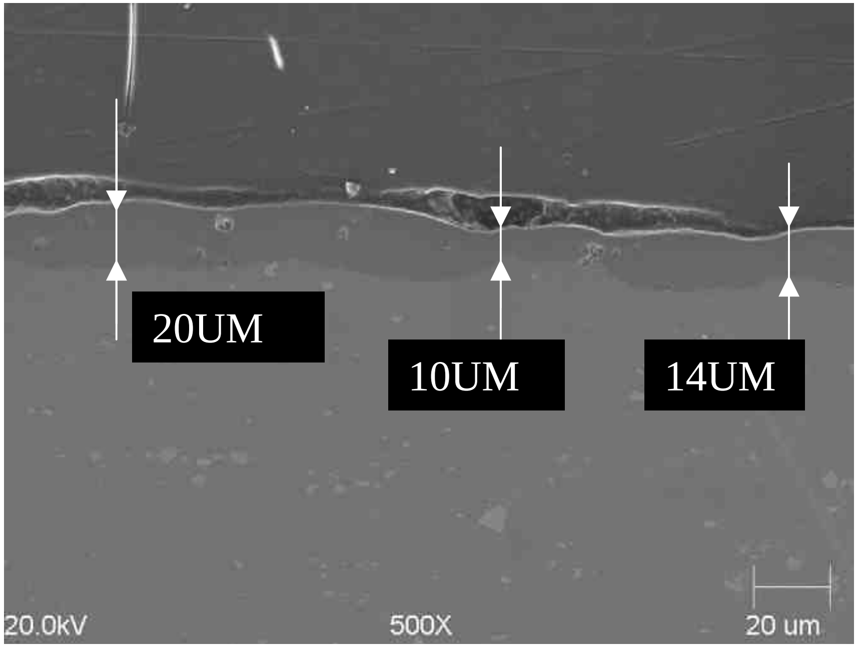


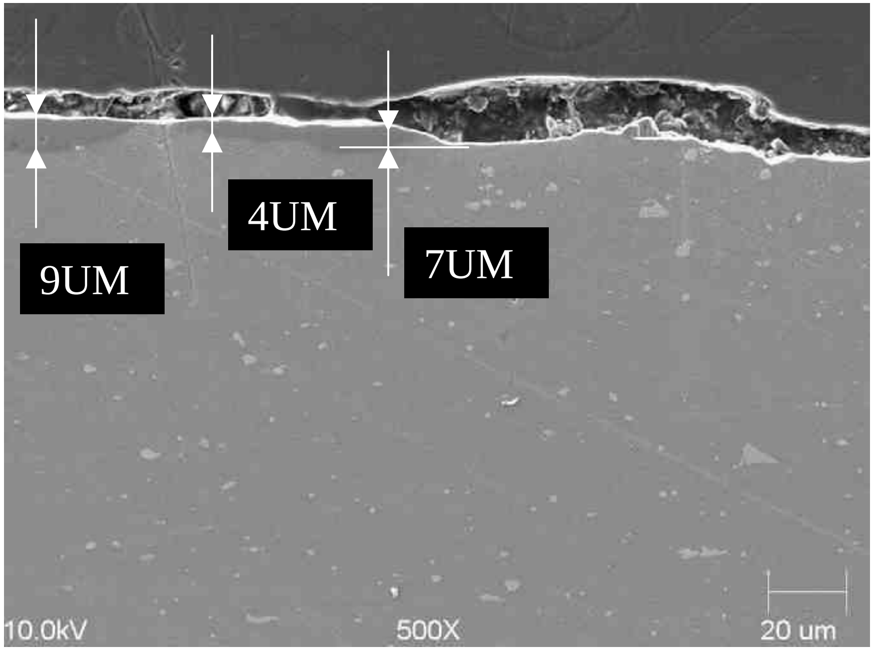
B

Low magnification photographic views of the 2024 anodized wear tracks after completing the 1000 cycle Taber abrasion test. Photo A shows the cryogenic treated plate which has significantly less damage than the standard anodized plate shown in Photo B. The standard anodized coating broke through earlier and smeared aluminum on top of wear track – see Arrow A. The wear damage on both plates is higher toward the ID of the wear track. The two photos were taken in the immediate vicinity of the transversally cut shown with Arrow V in Appendix 8.

APPENDIX 10

SEM views of the OD wear track anodized coatings after completing 1000 cycles with the Taber apparatus. Photo A shows the cryogenic treated anodized sample while Photo B presents the standard anodized processed sample. The average anodized area of both samples before starting the Taber test is 3205 UM^2.



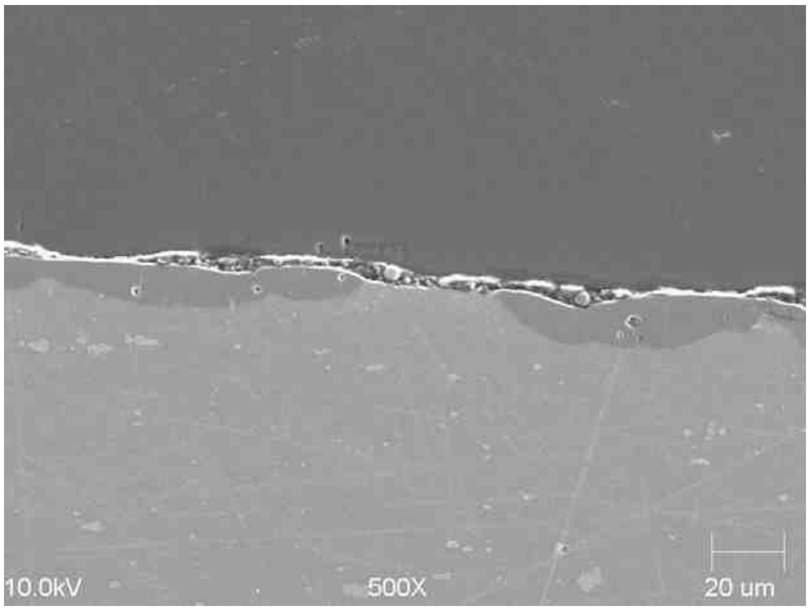


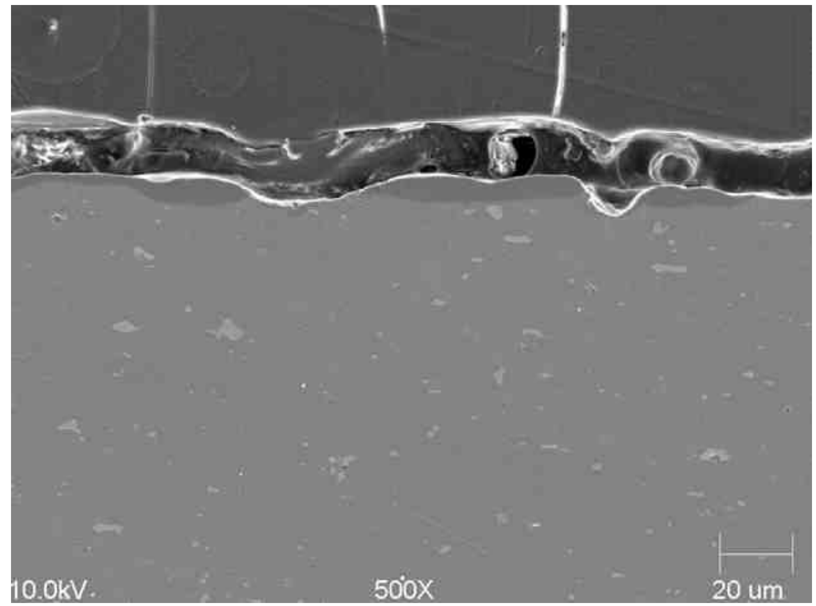
A **B**

The wear removed area of the cryogenic treated 2024 anodized layer shown in Photo A is minimum. Only 118 UM^2 were lost versus 2715 UM^2 the removed area of the standard anodized coating. The remaining area of the standard anodized coating is 490 UM^2 while the remaining cryogenic treated anodized area is 3090 UM^2. This represents a 630% wear improvement of the anodized layer obtained through cryogenic treatment.

APPENDIX 11

SEM views of the center anodized wear tracks after completing 1000 cycles with the Taber test. Photo A shows the 2024 cryogenic treated coating while Photo B shows the 2024 standard anodized coating. The average of both anodized areas before starting the Taber wear test is 3205 UM^2.





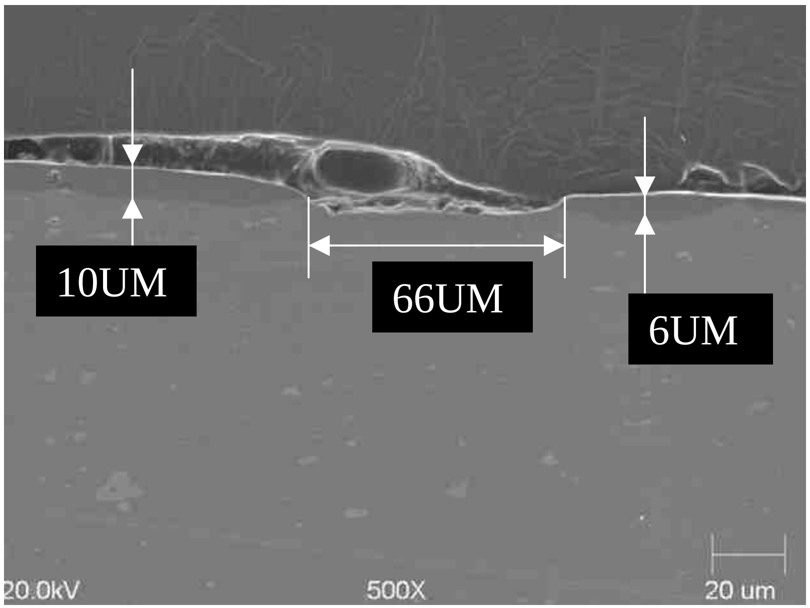
A

B

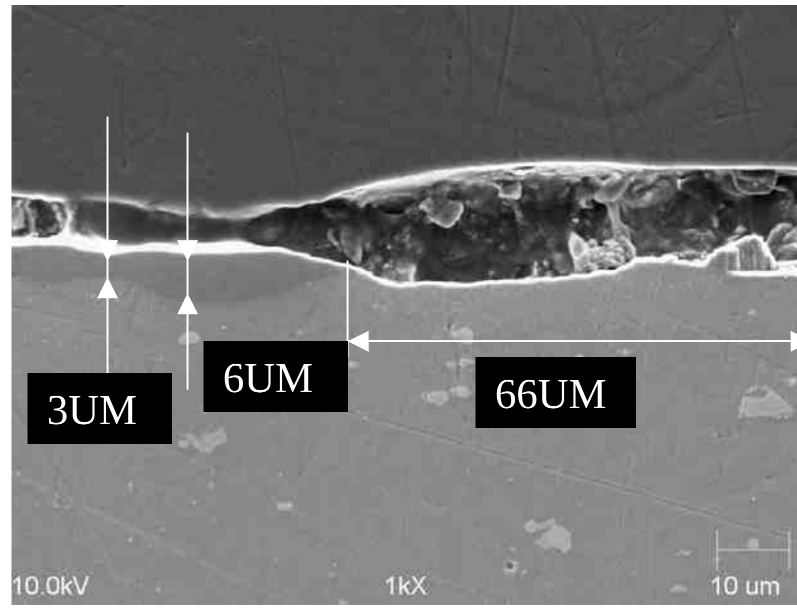
The remaining area of the 2024 cryogenic anodized coating is 1704 UM^2 versus 825 UM^2 the remaining area of the standard 2024 anodized coating. The wear loss of the cryogenic anodized coating is 1501 UM^2 while the wear loss of the standard coating is 2380 UM^2. Based on the ratio of the remaining anodized areas the wear performance of the cryogenic anodized coating is 207% better than the standard anodized coating.

APPENDIX 12

SEM views of the ID wear tracks after completing 1000 cycles with the Taber tester. Photo A shows the 2024 cryogenic treated anodized coating at X500 magnification while Photo B shows the 2024 standard anodized coating at X1000 magnification. This area was extrapolated to X500 magnification as well.



A

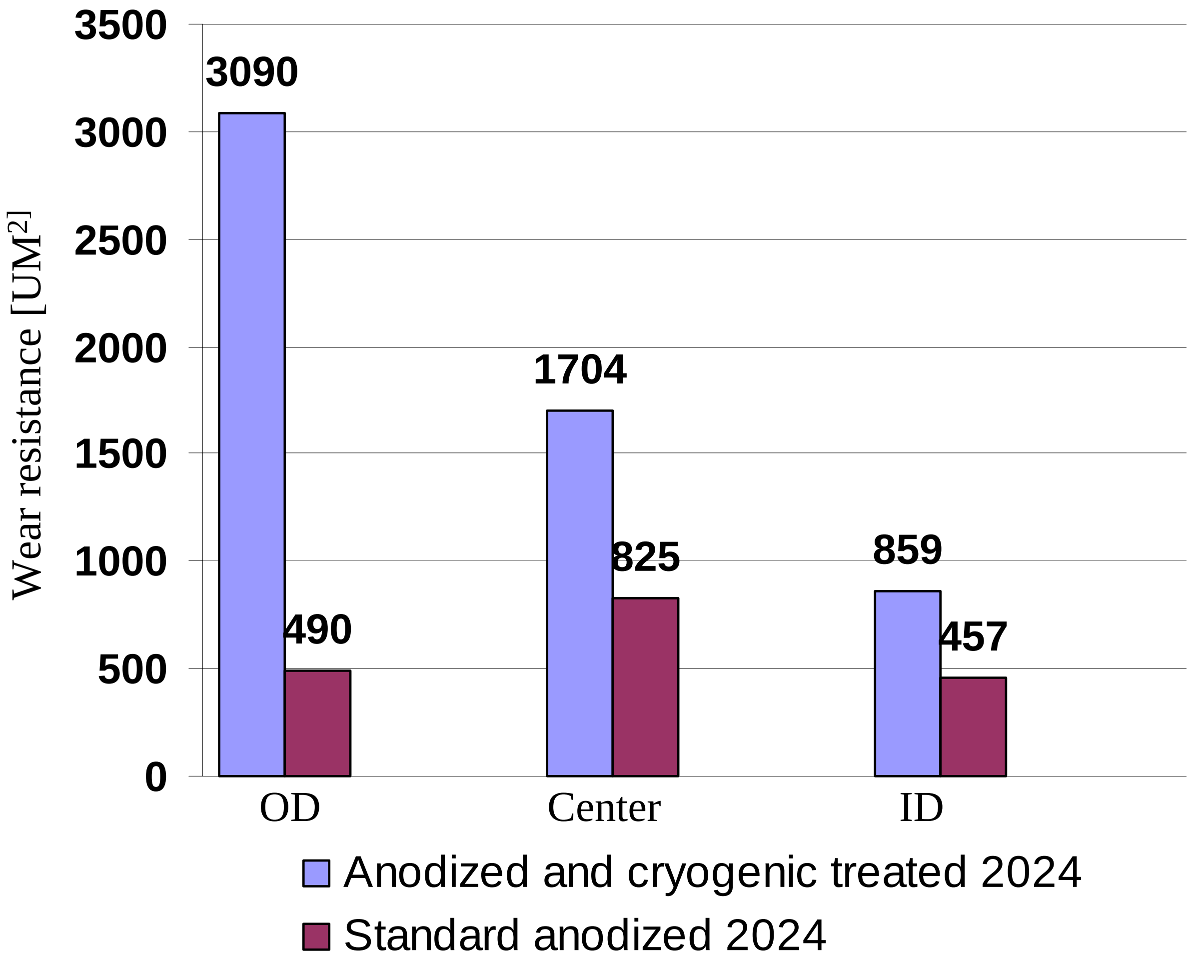


B

The remaining area of the 2024 cryogenic anodized coating is 859 UM^2 while the remaining area of the 2024 standard anodized coating is 228 UM^2. Indeed the ID of the wear track has the highest damage. The cryogenic anodized sample has 88% improved wear resistance versus the standard anodized sample. Likewise mentioned in Appendix 10 and Appendix 11 the area of both coatings before staring the Taber test was 3205 UM^2.

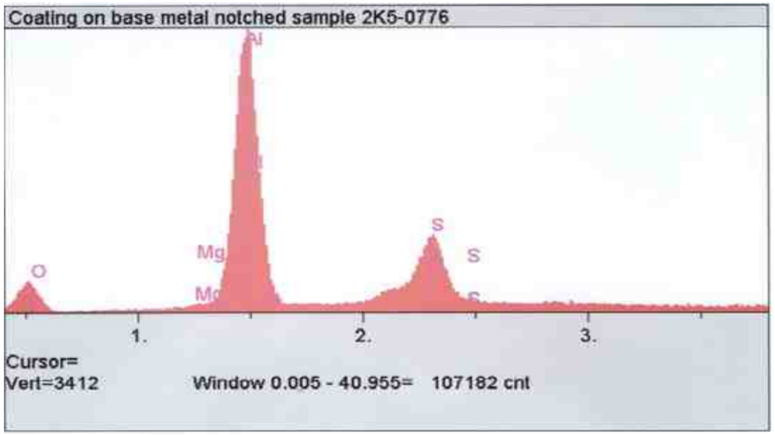
APPENDIX 13

Comparative wear resistance evaluated through the remaining areas of the standard and cryogenic treated anodized 2024 coatings at the OD, center and ID of the 1000 cycles Taber tested wear tracks.

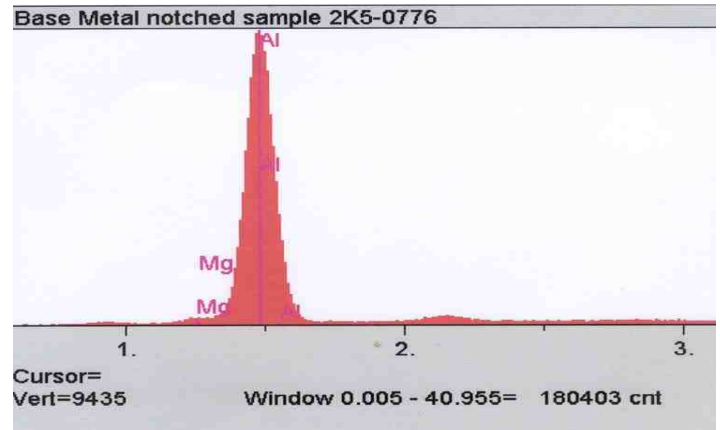


APPENDIX 14

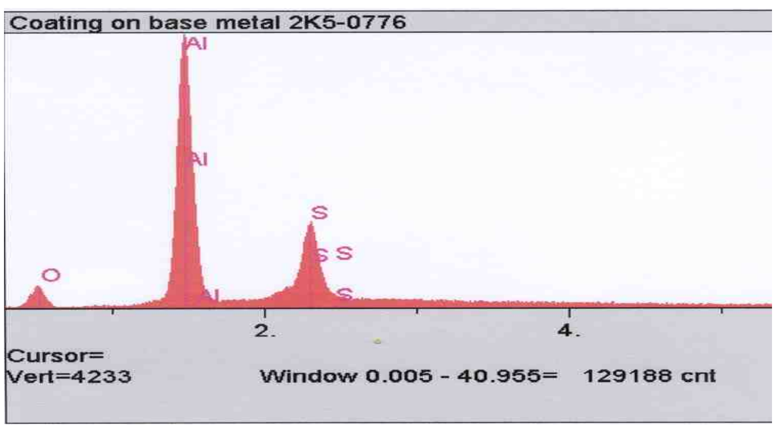
EDAX qualitative chemical investigation of A – 2024 cryogenic anodized coating while B is the 2024 body, C – 2024 standard anodized coating with D the 2024 body.



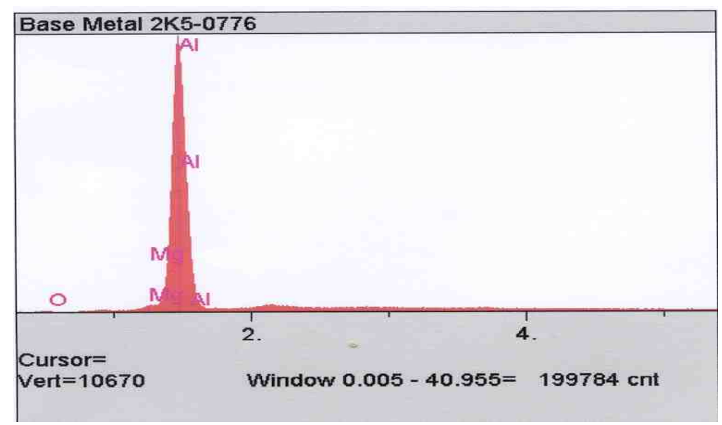
A



B



C



D

APPENDIX 15

View of a cryogenic treated basket loaded with wear resistant castings used in Wilfley centrifugal pumps. The castings are covered with a thin layer of frost.

APPENDIX 16

Rear view of the Wilfley cryogenic processing unit. The size of the cryogenic box is 10 ft x 10 ft x 5 ft and it was designed to provide a uniform cooling temperature within the processing chamber.