IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant : **Analytical Specialties, Inc.**
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Examiner : **Ho-Sung Chung**
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Customer No. : **21,901**
For : **Method and Composition for Metal Finishing**

RULE 132 DECLARATION
(37 C.F.R. § 1.132)

Upon being duly cautioned, we, Keith R. Eidschun and Joshua Cloakey, joint inventors of the invention of "Method and Composition for Metal Finishing", hereby declare individually that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that false statements and the like so made are punishable by fine, imprisonment or both under Section 1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the application and any patent issued pursuant thereto.

The present patent application discloses an anodizing system for use in anodizing of aluminum, magnesium or alloys thereof. A series of experiments were conducted to support unexpected results with respect to the cited prior art of Windsor-Bowen (G.B. 396,743, 1932) (hereinafter "Windsor") as well as current conventional anodizing systems.

Experiment 1

In the first experiment, we attempted to determine the anodic coating thickness on a 7075 aluminum panel as the anode and cathode using the parameters set forth in Windsor. Windsor requires a solution containing a proportion of sulfuric acid and sodium sulfate equivalent to 25% to 55% with the proportions of each being 1 of sulfuric acid to 3.2 of sodium sulfate. A 40% solution was prepared which contained 400 g/L of sulfuric acid and 1280 g/L of sodium sulfate, which is within the parameters given in Windsor. Windsor also requires use of an oxidizing agent, such as sodium persulfate, at a proportion of 1% or less. Applicant used between 0.1% and 0.5% (0.2g/L to 0.5 g/L) sodium persulfate, which is within the parameters disclosed in Windsor. Windsor discloses that treatment is conducted by current of 80 V DC or more with the current density being 1 to 5 amperes per square foot. Applicant applied 90 V DC, which is within the parameters listed by Windsor. Amperes were not able to be measured as discussed below for safety reasons. Temperature is not discussed in Windsor thus a temperature of 65°F was used as it is known in the art to use temperatures of between 62°F to 68°F. Table 1 below summarizes the parameters of Windsor and the parameters used in the experiment:

TABLE 1

	Windsor	**Experiment 1**
Solution concentration	Between 25% to 55%	40%
Solution composition	Ratio of 1:3.2 of sulfuric acid to sodium sulfate	Sulfuric acid 400 g/L Sodium sulfate 1280 g/L Ratio 1:3.2
Oxidizing agent concentration	$\leq$ 1%	0.1%
Oxidizing agent composition	Na or K nitrate Na or K persulfate Na or K perborate	Na persulfate
Voltage	>80 V DC	90 V DC
Amps	1-5 amps/sq. ft.	Unable to measure due to safety concerns
Temperature	Not given Known in the art to use between 62°F to 68°F	65°F
Substrate type	Aluminum and Al alloys	Aluminum
Processing time	Not given	< 1 minute due to safety concerns

The test was conducted on a 7075 aluminum panel as the anode and cathode with a 40% sulfuric acid solution, containing a ratio of 1:3.2 of sulfuric acid and sodium sulfate, and 0.1%

sodium persulfate used as the electrolytic media. The solution was cooled to 65°F and a potential of 90 V DC was applied to the test specimen.

At the conclusion of the experiment the test specimen showed no signs of any anodic coating present, lost material thickness (0.001 inches lost), and had a significant amount of residue on the surface of the material.

It is noteworthy that the solution boiled in less than 1 minute (in about 15 seconds) and began melting the wires so that the experiment had to be stopped for safety reasons before amperes could be measured. At 90 V DC the electrical potential is so high that the rate of dissolution of aluminum exceeds the rate of growth, thus no coating is able to form as it is dissolved as it is trying to be produced.

We submit that the type of equipment used to anodize aluminum in the 1930's was substantially different from that available today, particularly given that current rectifiers used for anodizing aluminum today run at a much lower voltage of around 10-15 V DC. We were unable to obtain particular machinery that would have been used in the 1930's to conduct the experiment. As such, while the method described in Windsor may have been successful using equipment known in the 1930's, recreating the experiment using current DC power supplies at 90 V, and inferring parameters such as temperature based on current anodizing systems, results in no anodic coating.

Additional Experiments

We conducted additional experiments using the parameters in Table 2 below:

TABLE 2

	Windsor	**Control**	**Persulfate additive**	**Permanganate additive**
Solution concentration	Between 25% to 55%	10%	10%	10%

Solution composition	Ratio of 1:3.2 of sulfuric acid to sodium sulfate	Sulfuric acid	Sulfuric acid	Sulfuric acid
Oxidizing agent concentration	≤1%	N/A	0.1%	0.1%
Oxidizing agent composition	Na or K nitrate Na or K persulfate Na or K perborate	N/A	Na persulfate	K permanganate
Voltage	>80 V DC	10 V DC	10 V DC	10 V DC
Amps	1-5 amps/sq. ft.	N/A	N/A	N/A
Temperature	Not given Known in the art to use between 62°F to 68°F	Between 62°F to 68°F	Between 62°F to 68°F	Between 62°F to 68°F
Substrate type	Aluminum and Al alloys	7075 Aluminum	7075 Aluminum	7075 Aluminum
Processing time	Not given	3 minutes	3 minutes	3 minutes

The 10% solution concentration for sulfuric acid was chosen because this is a concentration known to be used in current conventional anodizing of aluminum. As discussed below, sodium sulfate additive was not used in the above experiments. The amount of oxidizing agent was chosen to be within the parameters of Windsor. The voltage was chosen to correspond to a voltage known to be used in current conventional anodizing of aluminum in light of the high voltage used in Windsor boiling the solution and melting the wires. The temperature range was chosen to correspond to a range known to be used in current conventional anodizing of aluminum.

Results

The control, which replicates the conditions of conventional anodizing, resulted in no measurable anodic coating. A continuity test was performed on the specimen and was found to still have continuity which is an indication of no anodic coating formation on the surface of the aluminum.

The persulfate additive yielded no measurable coating and when a continuity test was performed the specimen was shown to still have continuity. This indicates that the coating did not have a non-conductive coating on the surface of the aluminum.

The permanganate additive yielded no measurable coating but when a continuity test was performed it yielded no continuity. This indicates that the surface of the aluminum panels has a non-conductive coating present.

Sodium sulfate additive was not used in the above experiments as the comparison to current conventional anodizing, using only sulfuric acid, was being made. Addition of sodium sulfate in the presence of permanganate is not beneficial. In conventional anodization, the hydrogen ion is a critical reaction for the cathode of the solution. Sulfuric acid is diprotic meaning that it releases 2 hydrogen ions into solution. The first hydrogen ion disassociates readily into solution, but the second hydrogen ion does not readily disassociate. This gives two parameters that are typically monitored in sulfuric acid anodization bath, free and total acid (hydrogen ions). The free acid readily reacts with the cathode to increase cathode efficiency, while the portion of the hydrogen ions that have not disassociated have a tendency to migrate to the anode where the electric field allows for them to disassociate and undergo multiple other potential reactions. By adding sodium sulfate to an anodization solution, the amount of free acid would reduce due to its binding to the sulfate ion. The use of potassium permanganate requires a high free acid content since the hydrogen ion is a critical component of the half-cell reduction reaction of the permanganate ion. Thus, the addition of sodium sulfate would slow the reaction as the sodium sulfate would compete with permanganate for the free acid. As such, the addition of sodium sulfate, as required by Windsor, would not be desirable when permanganate is used.

Conventional Anodizing versus Claimed System

Experiments were conducted between current conventional anodizing and the claimed invention according to the parameters in Table 3 below:

TABLE 3

	Current Conventional Anodizing	**Claimed System**
Solution concentration	10% to 15%	10% to 20%
Solution composition	Sulfuric acid	Sulfuric acid
Oxidizing agent concentration	N/A	0.01% to 0.05%
Oxidizing agent composition	N/A	K permanganate
Voltage	10-15 V DC	10-15 V DC
Amps	18-40 amps/ft^2 (dependent on total surface area of substrate)	10-50 amps/ft^2 (dependent on total surface area of substrate)
Temperature	Between 62°F to 68°F	Between 62°F to 68°F
Substrate type	2024, 6061, or 7075 Aluminum	2024, 6061, or 7075 Aluminum

Results of experiments described in the specification comparing conventional anodizing to the claimed system with respect to various aspects are summarized in Table 4 below:

TABLE 4

	Current Conventional Anodizing	**Claimed System**
Processing time	15 minutes to reach about 0.0002 anodic thickness	< 5 minutes to reach about 0.0002 anodic thickness (See Figure 10 of specification)
Dimensional change	Dimensional growth increases substantially as anodic coating thickness increases	Very little dimensional growth change as anodic coating thickness increases

		(See Figure 11 of specification)
Corrosion resistance	336 hours with pit formation	800 hours with no pit formation (See Example 6 of specification)
Abrasive wear	Fails taber abrasion	Coating with less than half the standard anodic thickness withstands at least two back to back taber abrasion tests (See Example 7 of specification)
Weight of coating	200-500mg/ft² for an anodic coating that is 0.0001-0.0003 inches in thickness and a minimum of 1000mg/ft² for anodic coatings that are 0.0004 – 0.0006 inches in thickness; For Hard Coat anodizing - anodic coating that is 0.0018 – 0.0022 inches in thickness the anodic coating weight should be a minimum of 4000mg/ft²	2639.52mg/ft², which is five times the weight required by specifications at a thickness of 0.0001 – 0.0006 inches; Compared to hard coat anodizing – novel solution exhibited over half the coating weight with only 1/6 of the anodic coating thickness (See Example 8 of specification)
Pore formation/microfractures	Very porous, several microfractures	No pore formation or microfractures (See Example 12 and Figures 25-26 of specification)

As shown in Table 4 above, the novel conventional anodizing solution claimed in the instant application demonstrates several superior unexpected results as compared to current standard conventional anodizing systems. These unexpected results include the novel system producing an anodic coating faster than that produced by the standard solution; producing a coating allowing for very little dimensional change; producing a coating having superior corrosion resistance; producing a coating having superior abrasive wear; producing a coating having a higher weight; and producing a coating having no pore formation or microfractures.

Summary

In conclusion, we have obtained unexpected results as compared to both Windsor as well as current conventional anodizing systems with regard to several aspects described above, including processing time, dimensional change, corrosion resistance, abrasive wear, weight of coating and pore formation.

Very respectfully,

Dated: 10/26/2018

Keith E. Eidschun

Dated: 10/26/2018

Joshua Cloakey