

June 20, 1961

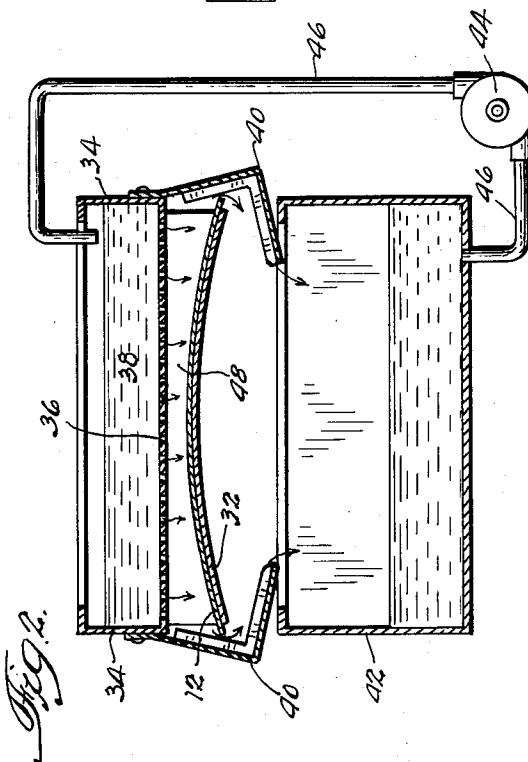
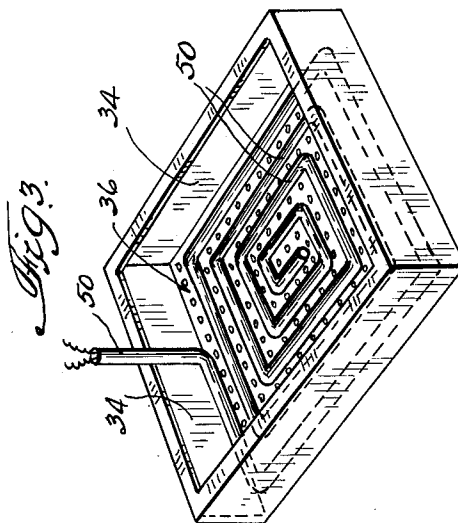
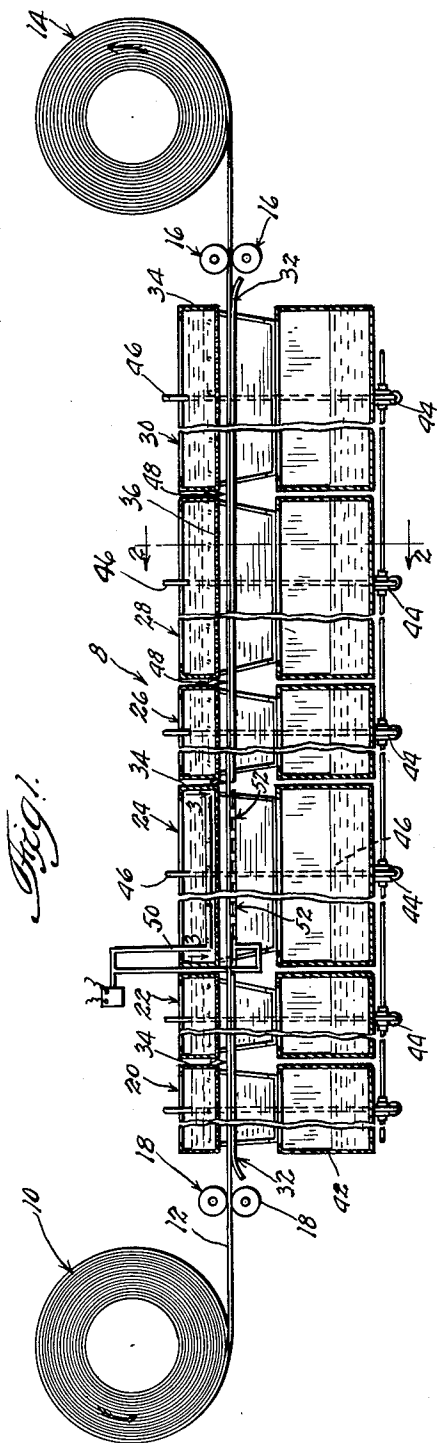
A. J. LLOYD ET AL

2,989,445

CONTINUOUS ELECTROLYTIC SURFACING OF METAL MEMBRANES

Filed Jan. 3, 1958

2 Sheets-Sheet 1



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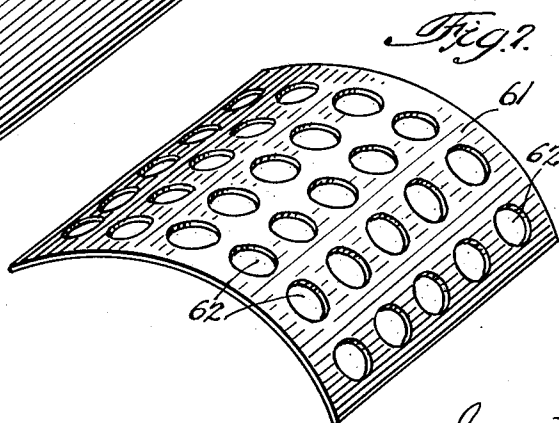
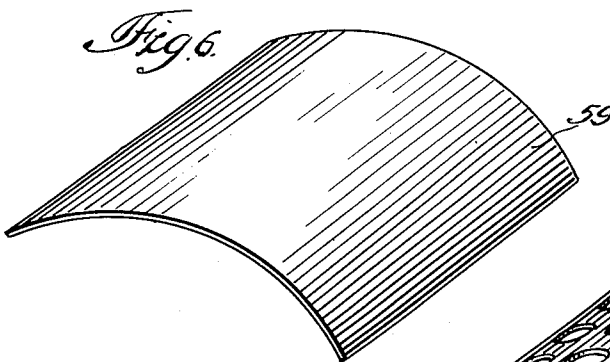
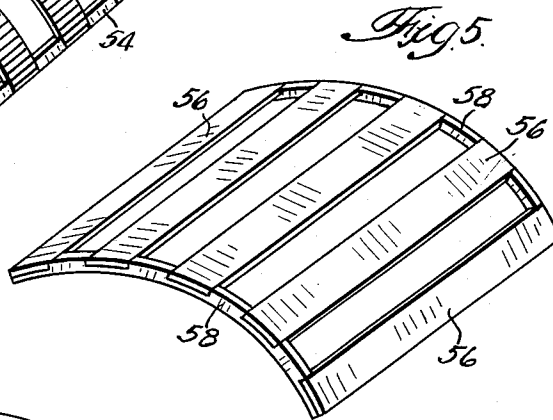
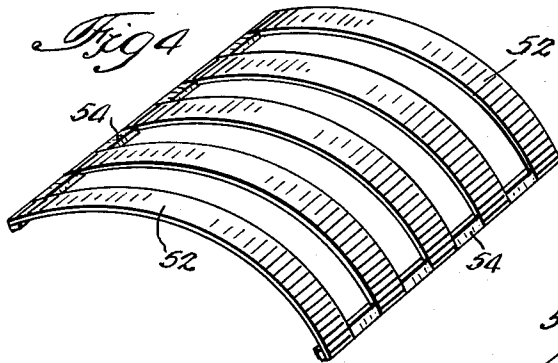
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2 Sheets-Sheet 2



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2,989,445

## CONTINUOUS ELECTROLYTIC SURFACING OF METAL MEMBRANES

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17 Claims. (Cl. 204-28)

This invention relates to a process for surfacing metallic membranes with a protective coating and, more particularly, to the continuous electrolytic surfacing of only one surface side of a metallic membrane, and to an apparatus for accomplishing the same.

This application is a continuation-in-part of our co-pending United States patent application Serial No. 603,710, filed August 13, 1956, now abandoned.

Processes for surfacing metallic membranes, as well as metallic objects in general, have heretofore been disclosed and used in the prior art. These well known prior methods involve primarily the plating of metal objects, sheets or the like, by complete immersion in an electrolyte tank while applying a current across the bath and object in a predetermined manner. The most classical examples of these methods are conventional electroplating or anodizing processes. When elongated sheets or strips of metal were to be electroplated, it has been a conventional procedure to pass the sheet through the electrolyte tank in a tortuous manner. With specific reference to the anodizing of aluminum, in such prior art methods, the anodization most commonly takes place between a web charged by a pair of contact rollers and an anodizing bath which carries the opposite charge. This technique involves charging the stock roll of aluminum sheet at a point outside the bath and passing the same through an electrolyte such as a sulfuric acid solution wherein the anodization takes place. In many instances in the continuous methods of processing of metallic membranes, localized high current densities are considered particularly detrimental since high temperatures usually result therefrom.

When relatively high current densities are evenly distributed over the surface of a metallic web undergoing processing, maximum benefits from the current utilized are achieved. These benefits are primarily that the rate of electrolytic surfacing is dependent upon the current density whether the process be that of anodization or electroplating, and therefore it is readily understood that using higher current densities results in more rapid anodization or electroplating. When the point of introduction of the current to the metallic membrane is relatively small with reference to the total area undergoing surfacing, the high localized current density results in fusing, overheating, and other detrimental effects.

For example, in one of the above described prior art techniques for continuous electrolytic surfacing of metallic membranes, the current is introduced into the web at a point removed from the contact area of the web and electrolyte. Since the area undergoing surfacing is limited to that portion of the web immersed in the electrolyte, the total charge or current introduced into the web is distributed over that interfacial area, which of course includes both surfaces contacting the electrolyte. Accordingly, the total charge that may be utilized is limited by the current carrying capacity of the web, which being relatively thin in cross section acts as a fuse when subjected to high current loads. Thus the total charge current which may be utilized is limited by the physical nature of the web being processed. To mitigate the possibilities of such fusion, overheating and the like, relatively low current densities were used and of course this

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unduly extends the time required to carry out the surfacing of that particular metal strip.

In addition to the problems associated with localized high current densities in these prior art processes, it has also been characteristic of these methods that the electrolytic surfacing has been carried out over all surfaces of the object or sheet and has therefore been costly and time consuming. Both surfaces were, of course, treated, since complete immersion in the electrolyte necessarily brought this about. In certain applications where the object required protection such as afforded by anodization on only one surface, the complete surfacing represented a needless and wasteful expense.

It is also true with respect to anodized aluminum that the colored effects which can be obtained by conventional dip-dyeing the anodized sheet are often wasteful in that only a single colored surface is exposed to view in applications such as in curtain walls of buildings, sheathing applications, machine housings formed from sheets, appliances, furniture, or the like. Therefore it would appear that coloring both surfaces of an anodized aluminum sheet amounts to unnecessary expense which in part has contributed to a less rapid acceptance and use than would ordinarily have been the case. It is also well known that aluminum sheet anodized on both surfaces is extremely brittle and, therefore, deep drawing or stamping thereof often results in fracture of the sheet so formed.

Accordingly, it is one of the objects of the present invention to provide a process for electrolytically surfacing only one side of a metallic membrane.

Another object of this invention is to provide a process for electrolytically surfacing only one side of a metallic membrane utilizing high current densities while eliminating the concentrating heavy current at localized portions of an aluminum web.

Another object of this invention is to provide a process for continuously electrolytically surfacing a theoretically infinite length of metallic membrane.

A still further object is the provision of a process for continuously anodizing an elongated strip of aluminum.

A still further object is the provision of a process for continuously anodizing, coloring, and sealing one surface of a continuous aluminum membrane in a unitized operation.

A still further object of the present invention is the provision of a novel method of forming an interface between a theoretically infinite length of aluminum membrane and an anodizing electrolyte.

A still further object of the present invention is to provide a process for the continuous and rapid electrolytic surfacing on one side of a moving continuous aluminum web without danger of overheating.

These and other objects of the present invention will appear from the description, accompanying drawings, and appended claims.

In one broad aspect of this invention there is provided a process for electrolytically treating one surface of a metallic membrane which comprises forming an interface with a flowing electrolyte over a substantial portion of said one surface of an elongated moving metal membrane and applying an electric potential across said interface between the electrolyte film and the metal membrane.

Referring particularly to a process for the continuous anodization of one surface of an aluminum membrane the present process broadly comprises moving an elongated aluminum membrane through a treating zone of substantial area, maintaining a flowing layer of electrolyte on said one surface substantially coextensive with said area, and applying an electric potential across said interface through the electrolyte and the metallic membrane in

contact therewith to uniformly distribute the electric charge over said area within the treating zone.

While the anodization of aluminum may be carried out at current densities of from 12 to 25 amperes per square foot, it is also true that utilizing the process of the present invention, current densities of up to about 200 or 500 amperes per square foot or above may be utilized resulting in very rapid anodization. However, it has been found for most purposes that current densities of from 50 to 180 amperes per square foot are satisfactory for rapid operation. It should be noted that high current densities in the present process result in anodic coatings that are superior to those formed by other methods, being more flexible, scratch-resistant and thicker. With regard to flexibility, anodic films on aluminum of up to 0.002 inch may be formed and the coated sheet subsequently drawn or shaped into a finished article. While heavy anodic coatings were known in the prior art, they were glass-like and extremely brittle. Similarly, while high current densities in the prior art methods often resulted in overheating of the sheet and the production of uneven and inferior coatings, the process of the present invention is superior in that respect since the flowing of electrolyte at the anodizing interface minimizes any heating effect that may occur and the coatings produced thereby are porous and of uniform thickness.

For a more complete understanding of this invention reference should now be had to the drawings, wherein:

FIGURE 1 is a vertical longitudinal schematic section of an apparatus for carrying out the process of the present invention, certain of the parts being shown in elevation.

FIG. 2 is a transverse sectional view of the apparatus taken along the lines 2—2 of FIG. 1, but on a larger scale.

FIG. 3 is a perspective view of a weir box and cathode shown in FIG. 1 and there designated by number 24.

FIG. 4 is a perspective view of the electrode utilized in the apparatus of FIG. 1.

FIG. 5 is a perspective view of another electrode similar to FIG. 4 but utilizing longitudinally disposed conductors.

FIG. 6 is a perspective view of still another embodiment of an electrode with the conductor in continuous sheet form.

FIG. 7 is a perspective view of an electrode similar to FIG. 6 but utilizing a perforated sheet as the conductor.

Referring now to the drawings and more particularly to FIG. 1, there is shown an apparatus 8 for the continuous electrolytic surfacing of metal membranes. A supply roll 10 is shown from which is drawn a sheet 12 or metal membrane which is horizontally drawn through the surfacing apparatus 8 and accumulated on a collection drum 14. A source of power may be provided at the drum 14 to draw the sheet or web 12 from the supply roll 10. For closer control of the membrane, powered rollers 16 contacting the sheet stock 12 may be used to draw the stock through the apparatus. Tensioning or idler rolls 18 are provided which maintain the sheet in proper tension and control the metal web being removed from the supply roll 10. There is provided, in this instance, a plurality of weir boxes 20, 22, 24, 26, 28 and 30, adapted to contain solutions suitable for use in the surfacing of the particular metal. The sheet is slidably supported on a plurality of supports 32 which (in FIG. 2) may be seen to be convex in cross-section, providing a crowning effect to the web which is drawn thereover. The purpose of the crowning effect on the web will become apparent from the discussion to follow. The weir boxes are of simple construction, being rectangular in shape and having sides 34 and a perforated bottom 36.

The weir boxes, 20 through 30, are situated in sequence above the metal web 12 and are adapted to discharge fluid contents thereof onto the top of the moving

web surface. Referring to FIG. 2, each weir box discharges fluid 38 on the upper or one surface of the sheet 12 (indicated by the arrows). The fluid flows off the sheet to either side due to the crowned shape of the metal membrane which is provided by the support 32. Troughs 40 or other suitable means collect and divert the fluid flowing from the surface of the sheet into collecting tanks 42. Each weir box is provided with a pair of these troughs which extend the length of the box and pass the fluid from said box into the tank 42. Wiping means 48 are disposed intermediate the weir boxes to contact the surface of the moving metal web and divert the surface liquids into troughs 40.

A pump 44 and pipe means 46 are provided for each weir box tank which circulate the fluid in the tanks 42 to the corresponding weir box. Thus, in operation, fluid passing from the weir box 24 through the perforated bottom 36 flows onto the sheet 12 in a continuous layer or film and off the sides thereof to collecting troughs 40 and into tanks 42. The fluid in the tanks is recirculated by a pump and pipe back to said weir box. The wiping means 48 divert fluid from the upper surface of the moving web to the sides thereof and into the collecting troughs 40 and, thus, prevent the intermixing of the liquids of adjacent weir boxes. It should be here noted that in the formation of the interface between the electrolyte and the metallic membrane in the electrolytic surfacing zone, the electrolyte should flow from the weir box 24 in such a manner as to provide a continuous layer of electrolyte over the surface of the web 12, as well as providing a continuous electrical contact from the cathode 50 disposed in the electrolyte in weir box 24.

In FIG. 3 is shown a perspective view of weir box 24 which is used for the electrolyte surfacing step and may be referred to for purposes of illustration as an anodizing step. A cathode 50 is shown disposed in the electrolyte fluid. For anodization, the cathode is attached to a source of direct current. Alternatively, a separate cathode need not be disposed in the fluid of the weir box, but the weir box may be constructed with a metal bottom such as lead sheet, which is a conductor and will serve as a cathode.

The under or other surface of the web is contacted by a plurality of elongated interconnected electrodes 52 formed in the same manner as the supports 32 to impart a crowned shape to the web 12. These electrodes are positioned in substantial opposition to the interface formed by the electrolyte 38 from weir box 24 flowing onto sheet 12. These electrodes are attached to the positive pole of the source of direct current. A sulfuric acid or other suitable anodizing electrolyte is provided in the weir box 24. In operation, the cathode contacts the sulfuric acid electrolyte which flows over the one surface of the moving metal web 12. The other surface of the metal sheet 12 contacts the electrodes 52 and it then becomes the anode of the system. When direct current is applied across said system, anodization takes place at the interface between the film of flowing liquid electrolyte which carries the negative charge and the one surface of the metal membrane which carries the positive charge. Thus, the current flow is substantially normal to the plane of the sheet 12 within the anodizing zone under weir box 24 and electrodes 52 since the positively charged electrodes or conductors 52 are in substantially opposed relation to the interface formed by the electrolyte on the one surface of sheet 12. The ultimate result is a substantially uniform distribution of the charge over the sector of the sheet within the anodizing zone.

In FIG. 4 is illustrated a perspective view of the electrode utilized in the apparatus of FIG. 1, showing the electrodes or conductors 52 laterally disposed across the direction of the sheet travel and in spaced side-by-side relation to each other. The conductors 52 are interconnected electrically at their ends by a conductor 54. While the individual electrodes or conductors are specifically

mentioned it should be understood that the term "electrode" also refers collectively to the group of electrodes through which the electric charge is transmitted to the under surface of the sheet in the electrolytic surfacing operation.

FIG. 5 shows an electrode similar to that of FIG. 4 but with the conductors 56 disposed longitudinally of the direction of travel of the sheet and in spaced, side-by-side relation to each other. These conductors are interconnected by a bus bar 58, so that they all carry the electric charge. It should be noted that while these conductors in the electrodes of FIGS. 4 and 5 are shown in spaced relation to each other, they may abut one another so as to present a substantially solid surface.

FIG. 6 is another electrode 59 in solid sheet form.

FIG. 7 is an electrode 61 similar to FIG. 6, having a number of uniformly spaced perforations 62 formed therein. In the foregoing FIGURES 4-7 the conductor may be formed from relatively heavy metal plate, such as copper or the like, or may be formed of non-metallic conducting media, such as graphite. In these figures the electrode is shown as crowned in the central portion and sloping symmetrically to either side in an arcuate manner as is preferred to provide for run-off of the electrolyte on the upper surface of sheet 12 when the latter is disposed thereupon. It should be noted that the electrodes illustrated are substantially the same area as the weir box disposed above the interface formed on the sheet 12.

Generally, the sequence of operation in the over-all process is a plurality of steps. Thus, with particular reference to anodization of aluminum, weir box 20 is provided with a caustic or other suitable cleaning solution. Box 22 contains water for rinsing; box 24 contains the anodizing electrolyte such as chromic acid, sulfuric acid or the like; box 26 contains water for rinsing; box 28 contains a suitable dye, such as Azorubine Extra, Quinoline Yellow P, Aluminum Blue A, etc., for coloring the anodized surface, and box 30 contains a sealing bath such as boiling water of about 212° F.

In one particular embodiment, the weir boxes are arranged and are of such size as to provide sufficient length of treatment for the passing aluminum membrane. For example, weir boxes 20, 22 and 26 are about 10 feet long. Box 30 is from 10 to 20 feet long. Box 24 (anodizing zone) may vary from 15 to 30 feet with about 18 feet optimum for most purposes. Box 28 is about the same size as box 24. The over-all length is approximately 100 feet, but may be adjusted for the needs of a particular process. Of course, the length of treatment in any one zone depends both on the rate of travel of the web and the length of the weir box in that zone. The current density maintained determines the rate at which the anodic coating is formed. Thus, in anodization of aluminum sheet using a current density of 60 amperes per square foot, a time of six minutes in the anodization zone produced an anodic coating 0.00044 to 0.00048 inch. The following table will illustrate other results obtained by this process.

Table

Electrolyte Temperature, ° F.	Current Density, amp./ft. <sup>2</sup>	Amp. minutes per square foot area	Coating Thickness (inch)
80.....	90	480	0.00068
80.....	120	600	0.00086
100.....	120	600	0.00094
100.....	120	480	0.00068
80.....	96	720	0.0014
100.....	120	720	0.00115
100.....	180	1,000	0.00167

In the foregoing experiment the electrolyte was about 35% aqueous sulfuric acid. Other sulfuric acid concentrations may be utilized, it only being necessary that the acid electrolyte can carry the current load satisfactorily. While the illustrations in the table show the use of temperatures of from 80° to 100° F. for the electrolyte, it

is true that temperatures approaching 32° F. and up to 120-125° F. may also be used to advantage. The electrolyte in anodization is not critical, but sulfuric acid is effective and also inexpensive. Generally oxygen-producing electrolytes are useful, such as aqueous sulfuric acid, chromic acid and also aqueous alkaline anodic baths, such as ammonium bicarbonate solution.

The current densities used may vary, but it is preferred to use densities of 50 to 180 although, as previously suggested, current densities as high as 500 amp./ft.<sup>2</sup> may be utilized. Of course, when high current densities are utilized, a greater amount of heat is generated and absorbed and carried away by the flowing electrolyte. Accordingly, it is desirable to cool the electrolyte by means of cooling coils in the collection tank 42 or cooling sectors in the conduit 46 which recirculates electrolyte to weir box in the anodizing zone. This cooling helps regulate the anodic coating deposited and assures better control and a more uniform product.

As may be readily seen from the foregoing, the prior art processes necessarily militate against the utilization of high current densities in the continuous anodization of aluminum webs. In the process of the present invention the charge is applied uniformly to the aluminum sheet over a relatively broad surface, and the area of the interface being quite large eliminates the possibility of high current densities at any local portion of the aluminum web and eliminates the line contact heretofore found in those processes for anodization of aluminum wherein a portion of the web was completely immersed in a charged electrolyte anodizing bath.

Generally the aluminum membrane must be sealed to make the protective anodized coating impervious. The sealing operation may take place immediately after anodizing step or may take place after the anodized film has been colored with a suitable dye, as aforementioned. While the dyeing operation is not essential, it provides a variety of attractive finishes. Generally the dyeing is carried out at somewhat elevated temperatures for periods of from about fifteen to thirty minutes, although shorter or longer periods may be desirable in specific instances. After dyeing, the sheet may be sealed in the same manner as if the anodized film were uncolored.

The sealing process is carried out by the use of a hot water bath, usually at the boiling temperature (212° F.).

While the anodizing electrolyte for aluminum has heretofore been described, other electrolytes for use in anodizing other metallic membranes may be used such as sodium hydroxide solution, sodium metaborate solution, sodium metasilicate solution, or the like.

Generally, it may be said that electrolytic anodization may be carried out using the process of the present invention on all those metals which form on proper treatment a protective oxide that prevents further oxidation or corrosion of the underlying metal.

The current densities may be adjusted for each plating operation. Agitation is not necessary when utilizing the process of the present invention, because the electrolytes utilized are constantly flowing in an uninterrupted manner from the weir boxes disposed above the metal membrane onto the surface of said membrane to collecting troughs and tanks and are recirculated.

While several particular embodiments of this invention are shown above, it will, of course, be understood that the invention is not to be limited thereto, since many modifications may be made and it is contemplated, therefore, by the appended claims to cover any such modifications as fall within the true spirit and scope of this invention.

We claim:

1. A process for continuous anodization of only one surface of an aluminum sheet which comprises passing an elongated aluminum sheet longitudinally and unidirectionally through a processing zone, treating one surface of said sheet with a cleaning solution, rinsing and wiping

said surface, passing said sheet through an anodizing zone wherein a substantial predetermined area of said one surface is contacted with a flowing anodizing electrolyte to form an interface thereon, contacting the other surface of said sheet with an electrode disposed in substantially opposed and coextending relation to said interface, passing an electric current of uniform density through said interface by means of the electrolyte and electrode to anodize the one surface of said sheet, removing the electrolyte from said sheet after anodization, and sealing the anodic coating formed on said surface.

2. The process of claim 1 wherein the anodized surface is colored prior to sealing.

3. The process of claim 2 wherein the current at the interface is maintained at a density of up to 500 amperes per square foot.

4. A process of anodizing only one surface of an anodizable metallic membrane which process comprises continuously moving an elongated anodizable metallic membrane through an anodizing zone, applying a flowing layer of anodizing electrolyte continuously over a predetermined portion of said one surface within said anodizing zone to form an interface thereon of an area substantially coextensive with said predetermined portion, and applying an electric potential across the entire interface area by contacting the other surface of the membrane with an electrode in substantially opposed and coextending relation to said interface, whereby the charge is distributed substantially uniformly thereover and flows in a direction substantially normal to the plane of said membrane to anodize said one surface.

5. A process of anodizing only one surface of an anodizable metallic membrane which process comprises continuously moving an elongated anodizable metallic membrane through an anodizing zone, applying a flowing layer of anodizing electrolyte continuously over a predetermined portion of said one surface within said anodizing zone to form an interface thereon of an area substantially coextensive with said predetermined portion, and extending entirely across said membrane, and applying an electric potential across the entire interface area by contacting the other surface of the membrane with an electrode in substantially opposed and coextending relation to said interface, whereby the charge is distributed substantially uniformly thereover and flows in a direction substantially normal to the plane of said membrane to anodize said one surface.

6. A process of continuous anodization of only one surface of an anodizable metallic membrane, which process comprises continuously moving an elongated anodizable metallic membrane through an anodizing zone, continuously flowing an anodizing electrolyte on said one surface to form an interface thereon of an area substantially coextensive with said zone and extending laterally entirely across said metallic membrane within said anodizing zone, applying an electric potential across the entire interface area by contacting the other surface of the metallic membrane with an electrode in substantially opposed and coextending relation to said interface and maintaining a substantially uniform current density thereover, wherein said metallic membrane is the anode of the system, and whereby the current flow is substantially normal to the plane of the metallic membrane to continuously anodize the said one surface.

7. A process of continuous anodizing of only one surface of an anodizable metallic membrane which comprises moving an elongated anodizable metallic membrane longitudinally through an anodizing zone extending laterally across and longitudinally of said one surface of said membrane, flowing a layer of anodizing electrolyte continuously over a predetermined portion of said one surface in the anodizing zone to continuously form an interface thereon of an area substantially coextensive with said predetermined portion and extending entirely across said membrane, simultaneously applying an electric po-

tential to the electrolyte by contacting said electrolyte with a cathode substantially coextending therewith and contacting the other surface of said membrane with an electrode-anode in substantially opposed and coextending relationship to said interface in a manner so as to substantially distribute the charge impressed thereby uniformly over said interface to continuously anodize said one surface.

8. A process of continuous anodizing of only one surface of an aluminum membrane, which process comprises moving an elongated aluminum sheet longitudinally through an anodizing zone extending laterally across and longitudinally of said one surface of said membrane, flowing a layer of anodizing electrolyte continuously over a predetermined portion of said one surface in the anodizing zone to continuously form an interface thereon extending entirely across said membrane, simultaneously applying an electric potential to said anodizing electrolyte and contacting the other surface of said membrane with an electrode in substantially opposed and coextending relationship to said interface in a manner so as to distribute the charge impressed thereby uniformly over said interface and anodize said one surface.

9. A process for the continuous anodization of only one surface of an aluminum membrane, which process comprises moving an elongated aluminum membrane through an anodizing zone, continuously flowing a layer of anodizing electrolyte upon one surface of said aluminum membrane within said anodizing zone while said membrane passes through said zone to form an anodizing interface thereon, extending laterally across the entire width of said moving membrane, contacting the other surface of said aluminum membrane with an electrode in substantially opposed relationship to said interface and substantially coextensive therewith, passing an electric current through said electrode and across the interface so formed on said moving aluminum membrane, whereby the current flows through said aluminum membrane in a direction substantially normal to the plane of said interface area and at a substantially uniform density.

10. The process of claim 9, wherein the current density at the interface in the anodizing zone is up to 500 amperes per square foot.

11. The process of claim 9, wherein the anodizing electrolyte is sulfuric acid.

12. The process of claim 9, wherein the temperature is maintained below about 120° F.

13. The process of claim 9 wherein the temperature is maintained between about 80 and 100° F.

14. A process for continuous anodization of only one surface of an aluminum sheet which comprises passing an elongated aluminum sheet longitudinally and unidirectionally through a processing zone, treating one surface of said sheet with a cleaning solution, rinsing and wiping said surface, passing said sheet through an anodizing zone wherein a substantial predetermined area of said one surface is contacted with a flowing anodizing electrolyte to form an interface extending entirely across the width of said sheet, contacting the other surface of said sheet with an electrode disposed in substantially opposed and coextending relation to said interface, passing an electric current through said interface by means of the electrolyte and said electrode to maintain a uniform current density throughout the anodizing interface to continuously anodize the one surface of said moving sheet, removing the electrolyte from said sheet after anodization, and sealing the anodic coating formed on said surface.

15. The process of claim 14, wherein the anodized surface is colored prior to sealing.

16. The process of claim 14, wherein the current density at the interface and normal thereto is maintained at a density of up to 500 amperes per square foot.

17. The process of claim 14, wherein the anodization

is carried out at a temperature between about 80° and about 100° F.

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