

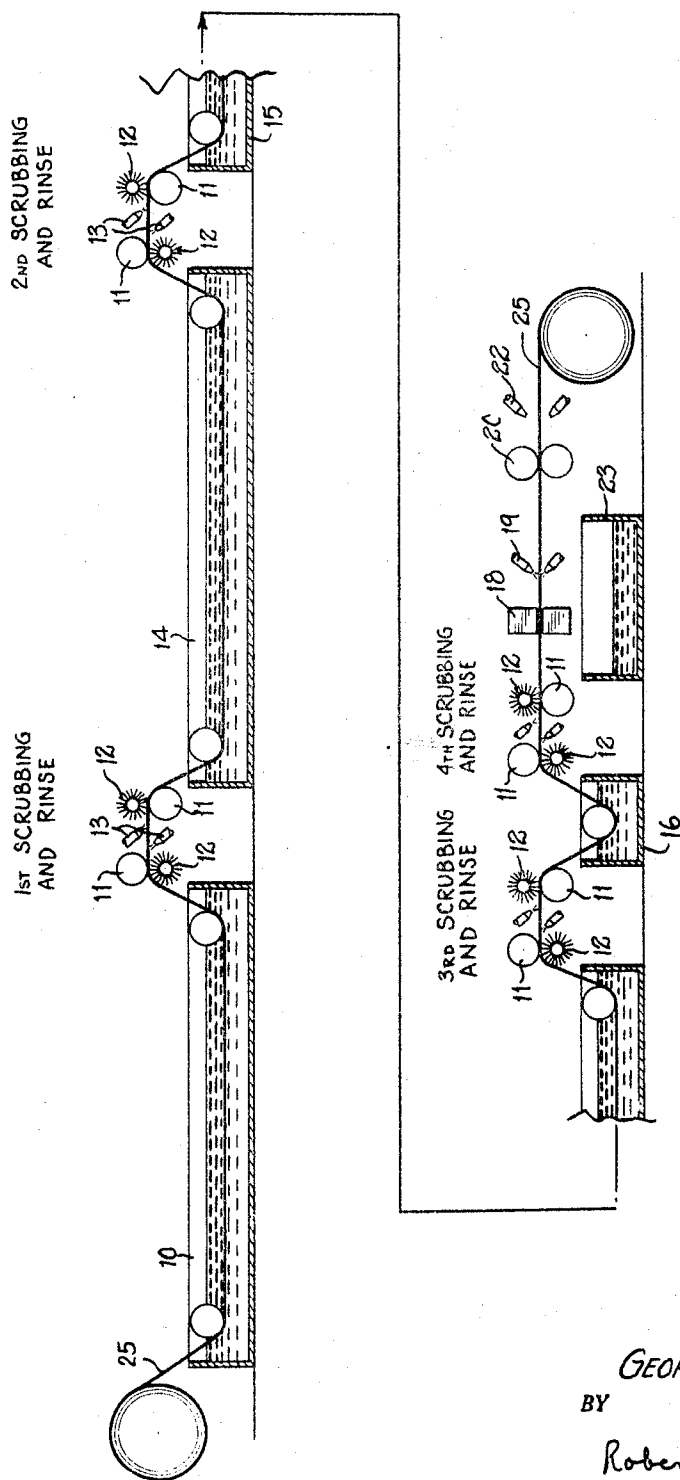
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ALUMINUM EVAPORATED COATING ON FERROUS METAL

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1

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ALUMINUM EVAPORATED COATING ON
FERROUS METAL

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This invention relates to the adherence of an aluminum coating on steel sheet by vapor deposition and more particularly, to the process of preparing steel sheet for the vacuum coating of aluminum.

Aluminum is coated on steel sheet by vapor deposition and adheres to the sheet by molecular adhesive forces attracting the aluminum to the steel. The aluminum does not alloy with the steel in the accepted understanding of alloying. The aluminum rather is in close adhesion with the steel surface and, therefore, satisfactory attachment of the aluminum coat to the steel is dependent upon close contact of the vapor deposited aluminum with the surface of the steel sheet.

Aluminum must be deposited upon a dry surface as well as a clean surface. If a wet cleaning method is used, there is presented the problem of drying the steel sheet to complete dryness without oxidation or the introduction of further soil. The sheet cannot be prepared simply by a cleaning method which results in a wet surface. Ordinary methods of drying will not produce the desired surface for the reception of a deposition of aluminum vapor.

Traces of oxides or precipitated matter which may be left on the surface of a cleaned strip of steel are objectionable in the vapor plating of aluminum onto the steel. There is no scrubbing action in the aluminum vapor plating process to remove such traces or minute amounts which are foreign in nature. The aluminum will condense over non-metallic surfaces as well as metallic surfaces and thus the presence of extraneous material will not prevent the laying down of the coating but will prevent its permanent adhesion. Also, methods of cleaning the steel surface which would destroy the original surface or which would destroy an important characteristic of the steel cannot be considered. For example, sand blasting which would scratch the steel surface, or bright annealing in hydrogen which would destroy the temper of the steel, are not available for preparing the dry steel surface for the vapor deposited aluminum.

It is an object of this invention to provide a method for coating aluminum on steel sheet by evaporation.

It is another object of this invention to prepare a dry surface of steel for the vapor deposition of an aluminum coating in close molecular adhesion thereon.

Still another object of this invention is the preparation of a surface of steel sheet dry and completely free from extraneous material which interferes with the close adhesion of a vapor deposited aluminum coating thereon.

These and other objects of this invention will become more apparent upon consideration of the following description, taken together with the accompanying diagram which shows in diagrammatic form the flow of the process of this invention.

In general, this invention provides a dry surface of a sheet of ferrous metal completely free from material deleterious to the attainment of a tenacious deposition

2

of aluminum by vacuum coating. The surface of the metal sheet, treated by the method of this invention is dry and is free from all grease and oil, oxides and precipitated compounds which have been found to defeat successful vapor plating of steel with an aluminum coat. The treated sheet will receive the aluminum coat with such adherence that no rupture of the adhesion of the coat is noted before the base steel is deformed to the point of rupture. A means of testing the adherence of the coating is provided by using a lacquer coating comparable to the coatings on tin plated steel sheets. The adherence of the aluminum coat is matched against the adherence of the lacquer coating to the aluminum coat under deformation of the metal sheet.

The diagram shows a system of cleaning a strip of steel sheet for aluminum deposition. This steel sheet may be any suitable steel such as black steel plate used in tin plating. Various other ferrous metals suitable for aluminum plating may also be cleaned by this method. As the purpose of the process is to provide the surface of the sheet for aluminum plating by vapor deposition dry and free of all extraneous matter, it is necessary in the process to remove oxides and residual solids and to avoid reoxidation of the cleaned, dry surface before the process of deposition. A sheet 25 is subjected to a succession of stepwise treatments. A tank 10 is provided with a solution of a cleaning composition for first cleaning the sheet 25 in a soak-cleaning. A soak-cleaner suitable for cleaning the black steel plate of sheet 25 has the following composition in a solution of 45 grams per liter:

	Percent by weight
Fused sodium orthosilicate	75.5
Dense soda ash	9.25
Tetrasodium pyrophosphate	5.00
Dresinate TX ¹	10.0
Igepon AP-75 ²	0.5
Pine oil	.75

¹ Dresinate TX is an alkaline metal salt of processed rosins and other resins which is the product of Hercules Powder Company.

² Igepon AP-75 is a fatty alkyl ester sulphonate which is the product of General Dyestuff Corporation.

The sheet 25 is cleaned in this soak-cleaner solution of tank 10 for 5 minutes at 205° F. This time period may be reduced to as short an interval as 30 seconds with sufficient agitation.

The sheet 25 leaving the tank 10 passes between a superposed back-up roll 11 and an underlying rotary brush 12 which, matched against the back-up roll 11, scrubs the lower surface of the sheet. Both surfaces of the sheet 25 are then rinsed by water sprays from nozzles 13 followed by a scrubbing of the upper surface with the rotary brush 12 against the back-up roll 11. The scrubbing of the upper and lower surfaces of the sheet 25 in each of the scrubber-type rinses in the illustrated cleaning apparatus is effected by rotary brushes 12 paired against a respective back-up roll 11.

Following the first scrubbing, the sheet 25 is subjected to a cathodic electro-cleaning in a tank 14 for 1 minute at 205° F.; less time (30 seconds) is sufficient with proper agitation. An electro-cleaning composition based on 90 grams per liter of solution is as follows:

	By weight
Na ₂ CO ₃	44.5
Na ₃ PO ₄	33.0
NaOH	22.5

In this electro-cleaning step, the sheet 25 is electrically connected as a cathode in the tank 14 and subjected to a current of 30-50 amperes per square foot of stripped area in the bath. Other suitable electro-cleaning composi-

tions may be employed as long as an adequate cathodic cleaning is achieved.

After electro-cleaning, the sheet 25 is subjected to scrubbing. Between scrubbing the lower surface and the upper surface, pairs of nozzles 13 spray both the upper and lower surfaces to provide a rinsing action. Following the second scrubbing and rinsing, the sheet 25 is treated with a muriatic acid pickling in a tank 15. The muriatic acid pickling solution is a hydrochloric solution ranging from 15% to 30% in concentration. The concentration of the acid solution and the time of immersion of the sheet 25 vary with the degree of soil or oxide on the sheet and the agitation of the solution. A 15 second dip is effective and a shorter time can be used with agitation if the soil on the sheet is not excessive. Following the acid pickling, the sheet is subjected to the third scrubbing and rinsing. Rotary brush 12, paired against the back-up roll 11, brushes the lower surface of the sheet 25 followed by the rinsing of both surfaces from a pair of nozzles 13. The rotary brush 12, paired against the back-up roll 11 scrubs the upper surface of the sheet 25.

The sheet 25 is substantially cleaned but is not satisfactorily prepared for the reception of vapor deposition of aluminum upon the cleaned surfaces. It is necessary to further prepare the surfaces of the sheet 25, according to this invention.

To obtain a satisfactory adherence of vapor deposited aluminum on the sheet 25 surfaces, the sheet is next subjected to an additional soak step in a tank 16 to provide the steel sheet surface with a passivity which inhibits or prevents reoxidation. Tank 16 contains a soak-cleaner composition the same or similar to the above-noted composition of the solution in tank 10. This composition in a solution of 45 grams per liter of water and at a temperature of 205° F. acts upon the surfaces of the sheet 25. This reaction of the cleaner composition on the cleaned surfaces of the sheet 25 provides the surfaces of the sheet with resistance to oxidation upon exposure to atmospheric oxygen for even a substantial period of time. The step of dipping the sheet 25 in the soak-cleaner tank 16 is followed by a fourth scrubbing and rinsing by a pair of rotary brushes 12 against respective back-up rolls 11 and oppositely applied sprays from nozzles 13.

The scrubbed and rinsed surfaces are wiped by a wiping means 18 by dragging the strip through a cotton fabric in the wiping means 18 which is moistened with a dilute cleaner, such as the soak-cleaner composition set forth above. This wiping serves to remove any residual precipitated material or loose smut still clinging to the steel surfaces. The dilute cleaning composition inhibits oxidation or rusting of the surface by providing an alkalinity thereto. After the wiping, the steel sheet surfaces are rerinsed with sprays from nozzles 19 and then quickly dried by a combination of rollers 20 and air blasts from nozzles 22. The rollers 20 are composed of a non-porous, non-wettable substance such as solid Tygon. The Tygon of the rollers is a modified halide polymer condensation resin and diene derivative compounded to produce a synthetic rubber-like material having non-porous and non-wettable physical properties. Tygon is the product of the United States Stoneware Company. A tank 23 is provided to receive the rinsing water from the nozzles 19.

The sheet 25 thus prepared is ready for the deposition of aluminum vapors. The surface of the sheet, free from grease and oil, oxides, precipitated compounds, and such residual solids or contaminants, may be passed to a chamber for plating with aluminum by vapor deposition without further treatment and with a handling procedure which avoids contamination. The combination of rapid drying of the cleaned and passivated surface enhances the receptivity of the surface for the aluminum plate. By cleaning the surface of all residual solids before drying, the surface is prepared so that the rapid drying will leave the surface free of residual contaminants after drying. These steps, together with the passivating accomplished in the soak-

cleaning after acid pickling, and the wiping of the surface by the mechanical means, combine to provide a satisfactory surface of this invention.

The deposition may take place in a standard vacuum system for the vacuum deposition of metals having a vacuum chamber into which the cleaned steel is passed immediately after the completion of the drying step. One such vacuum system may consist of a vacuum metallizer and a chamber. The pumps of the vacuum metallizer and the chamber are connected for operation by appropriate valves and connections.

It has been determined that a silicone type oil, which is chemically stable and is identified as silicone oil DC703 of the Dow Corning Company, is most suitable as a diffusion pump oil. The deposition of the vapor deposit aluminum coating by the vacuum system is assisted by the use of a diffusion pump oil, such as DC703, which has excellent stability to air and water vapor at the temperatures for pumping the vacuum.

The aluminum for deposition on the steel sheet surfaces is obtained from a clean and uncontaminated source to provide the best adherence to the surfaces. Aluminum vaporized from hot tungsten filaments, which are heated by an electrical resistance to evaporate the aluminum, has a high purity and deposits upon the surfaces with good adherence. The vaporized aluminum may be deposited on the steel surfaces in the vacuum in coatings of various thicknesses. Good adherence has been produced on steel surfaces, cleaned according to the method of this invention, in coating thicknesses up to 180×10^{-6} inches of aluminum. The normal thickness of the aluminum coat on steel cleaned according to this invention is of the order of 30×10^{-6} inches and is substantially in excess of a coating of 5×10^{-6} inches.

In the step of the deposition of aluminum, the vacuum chamber is evacuated prior to aluminum deposition by a four stage procedure which includes a first step of rough pumping to a vacuum of 200 microns of mercury. The vacuum is then further reduced by the use of the diffusion pumps. The residual atmosphere remaining after a pump-down must not contain organic vapors. A high voltage discharge step provides good adherence of the coating on the cleaned steel surface. This is effected while the vacuum chamber is being evacuated with the diffusion pumps. The surface of the steel plate is degassed by a high voltage discharge. After the high voltage discharge, the fourth step of the pump-down procedure carries the vacuum to about 0.5 micron of pressure. The aluminum is then vaporized from the tungsten filaments in a flash evaporation which may occur within 10 to 15 seconds after melting of the aluminum.

The resultant coating of aluminum on the steel, prepared according to this invention, has been found to be a tenacious coating having satisfactory adherence even under bending and other deformation of the plated product.

Referring again to the preparation of the steel sheet 25 as shown in the diagram for the vapor deposition, the dip in the soak-cleaner in tank 16 of the diagram passivates the surface to prevent or reduce to a minimum the contamination or modification of the steel surface subsequent to pickling. The further wipe of the surface, after the soak dip, aids in the removal of any tenaciously adhering substances such as a hydroxide, which are not rinsed off from the surface. The sheet is passed through a final water rinse before passing through the non-wetting, drying rollers 20. The final drying, by squeezing between the rollers 20 and the impingement of the air blast from the nozzles 22, leaves the surface of the steel in a passive condition. At the same time, the steel is not susceptible to rusting or other combination which would produce undesirable contamination of the surface before deposition of the aluminum vapor.

It has been discovered that drying the steel surface

with non-porous, non-wetting rollers, such as rollers 20 composed of Tygon, leaves the surface dry without oxidation and without the introduction of further contaminants or soil. The rinsing of the surfaces of the sheet 25 by sprays from the nozzles 13 and 19 is carried out so that a minimum of water is carried on the sheet 25 from one step to the next in the cleaning process.

Care is exercised to avoid contamination of succeeding steps with rinse waters from preceding steps. Similarly, care in the execution of the wipe by mechanical means at 18 avoids the deposition of undesirable foreign elements, such as lint or soap, onto the sheet.

A test has been devised for testing the adherence of the aluminum coating to the steel. In this test, a coating of lacquer, such as phenolic lacquer, is applied over the aluminum and baked. A ball is then pushed into the steel plate and the steel plate is drawn to the point of rupture. Pressure sensitive adhesive tape is then applied to the drawn area. When the tape is pulled away from the surface, poor adherence is indicated by spots of aluminum and lacquer coating adhering to the tape. The aluminum bond to the steel under the lacquer coating, if poor, gives way under the distortion and the area of aluminum and lacquer is stripped off. It has been found that the application of the lacquer coating over the aluminum affords a better test of poor adherence of the coating to the steel, than by trying to detect peeling of the aluminum on unlacquered samples. One lacquer which may be used is P-2 lacquer, formulated by the American Can Company, and is baked on the coated sample at 385° F. for 15 minutes. The lacquer coating is representative of any organic coating having good film strength and fair adherence to a metal base which may be applied to the coated sample for testing.

This test is related to the fabrication with the coated sheet steel. The aluminum coating must withstand forming tests after its application to the sheet steel. In practice, the coated sheet steel is lacquered and then formed. Therefore, the aluminum coating must be adherent on the sheet under forming tests after the application of the lacquer coating, such as is used in the manufacture of cans. If the sheet steel will withstand the described test, it will be susceptible to any forming operation. In the test described herein, after the aluminum coated steel sheet is lacquered according to the test procedure as set forth above, the aluminum coated steel sheet was deformed to the point of rupture. The aluminum coating could not be stripped from the steel surface upon removal of the tape. It is thus seen that the tenacity of the aluminum coating on the steel is satisfactory.

The steel sheet, coated with aluminum after preparation according to the method of this invention, is uniform in appearance and in adherence of the coat to the steel sheet. Cans fabricated from the material produced in this manner have passed all fabrication tests, which include shearing, drawing and the formation of double seams.

The aluminum plate of this invention provides a sacrificial protection of the steel surface under certain conditions. The original surface is retained without affecting the mechanical properties of the steel sheet. Further, good results have been obtained in the corrosion tests of steel sheet plated with aluminum according to this invention. There is no difference in the results of plating steel sheets of different gauges with aluminum by this invention. Aluminum plating by vapor deposition produces a surface having a good finish. The mechanical wiping step of this invention brightens the appearance of the aluminum plated surface. The results obtained by fabrication tests and corrosion tests indicate that the adherence of the aluminum coating on the steel sheet is of sufficient density to meet the requirements of use in the manufacture of cans and allied products.

The aluminum coating is provided by the method of

cleaning the ferrous surface for the reception of the deposition of the aluminum vapor. The cleaning system gives adherent plates of aluminum from a vapor deposition of the aluminum on the ferrous surface which are over 30×10^{-6} inches in thickness and may reach 180×10^{-6} inches in thickness. Thus, there is provided a dense pore-free aluminum coating on the surface. In addition, the aluminum coating exhibits adherence to steel sheets of the type used for container fabrication, such as black steel plate. This adherence is comparable to the adherence of tin to black steel plate. Accordingly, the aluminum coated steel sheet cleaned and prepared according to this invention is as satisfactory for fabrication as tinned black steel plate presently in use.

Various modifications of the above described steps of the process of this invention may be made without a departure from the spirit of the invention. As indicated, the soak-cleaner composition may be any cleaner which provides the inactive, passivated, dry surface on the treated steel sheet described herein. The rinses may be effected either by flowing water in a tank or by spray rinses. With either means of rinsing, the degree of rinsing must be adequate to remove the residue from the preceding step in a satisfactory manner. Ranges of concentrations of solutions and times of treatment in the acid pickle step have been indicated. Similarly, the electro-cleaner composition may be modified as long as adequate cathodic cleaning is obtained. Also, as indicated above, the rollers 20 may be any suitable non-porous, non-wetting material which quickly dries the surface without oxidation.

The specific embodiments set forth in the above description illustrate the spirit of the invention which is limited only by the scope of the appended claims.

What is claimed is:

1. The method of preparing a ferrous metal surface for vapor deposition of an aluminum coating which comprises treating the surface with an acid solution to remove impurities, passivating the acid cleaned surface with an alkaline solution to produce an elemental surface, wiping the elemental surface and squeezing and blowing the surface to dryness without oxidation for the deposition thereon of a coating of aluminum from a vapor of aluminum in a vacuum.

2. The method of preparing a ferrous metal sheet for coating by aluminum vapor deposition which comprises cleaning a ferrous metal sheet by acid pickling, treating the sheet surface with alkaline soak-cleaner to passivate the cleaned surface against re-oxidation, wiping the passivated surface with mechanical means to remove residual contaminants, rinsing the passivated and wiped surface to remove soluble contaminants and rapidly drying the rinsed surface without oxidation, by successively squeezing and blowing the surface.

3. In the method of preparing a ferrous metal surface for coating by aluminum vapor deposition after cleaning by acid pickling, the steps of treating the surface with an alkaline soak-cleaner to passivate the surface against re-oxidation, removing tenaciously adhering contaminants by mechanical wiping means, rinsing the surface free of all remaining contaminants, solid and dissolved, and quickly squeezing and blowing the surface to dryness without oxidation.

4. The method of forming an aluminum coating on a ferrous metal surface which comprises cleaning a ferrous metal surface, acid cleaning said cleaned surface, treating the acid cleaned surface with an alkaline solution to passivate the surface, rapidly drying the passivated surface without oxidation, and vapor depositing on said cleaned and dried surface a dense-pore-free coat of aluminum in a thickness of at least 30×10^{-6} inches, said rapid drying operation including the step of squeezing the passivated metal surface with a non-porous, non-wetting flexible roller of synthetic rubber-like material to rapidly dry said surface without oxidation.

5. In an apparatus for forming an aluminum coating

7

on metal surface, the combination with means for vapor depositing aluminum on a cleaned metal surface of cleaning means including means for rinsing said metal surface after final cleaning, means for wiping said cleaned rinsed surface, means for final rinsing said surface after wiping and a non-porous, non-wetting roller of synthetic rubber-like material for squeezing said final rinsed surface.

6. The method of preparing a ferrous metal surface for vapor deposition of an aluminum coating which comprises subjecting the surface to an alkaline soak-cleaning treatment and a cathodic electro-cleaning treatment, thereafter subjecting the surface to an acid pickling treatment, treating the acid-pickled surface with an alkaline solution to passivate the surface, each of the aforesaid treatments being followed by scrubbing and rinsing treatment of the surface, removing residual adhering contam-

8

inants from the passivated surface by mechanically wiping said surface, thereafter rinsing the passivated and wiped surface to free the same of all remaining contaminants, and rapidly squeezing and blowing the surface to complete dryness without oxidation, said squeezing step comprising engaging the surface with non-porous, non-wetting, flexible means to displace liquid.

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