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3,061,485

RESIDUAL LUBRICANT REMOVAL FROM ALUMINUM FOIL

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5 Claims. (Cl. 148—13.1)

This invention relates to a method for the treatment of aluminum strip in coil form to remove residual coatings of lubricant from the surface of the strip. More particularly, the invention concerns a novel method for the removal of rolling oil from aluminum foil in the as-rolled condition by applying to the exposed ends of the tightly wound coil a stream of gas containing free oxygen, leaving the coil freely unwindable. This is a continuing application with respect to my copending application Serial No. 726,613, filed April 7, 1958, now abandoned.

In this field, problems of sticking have long been recognized, and are attributed to the presence of residual lubricant during the heat treatment of aluminum foil subsequent to rolling. Such sticking becomes particularly acute when the aluminum foil, after coiling, is subjected to heat treatment.

In a dry annealing process, an objective is to remove the residual rolling lubricant, and temperatures as high as about 1000° F. have been employed. There is also known a process called slick annealing, in which the aluminum foil is heated in an inert atmosphere, but the lubricant is not substantially removed. Dry annealing conventionally involves the use of higher temperatures than slick annealing, since such higher temperatures were regarded in the art as necessary in order to drive off volatile materials by distillation. Prior to the present invention, no technique was known whereby lubricant could be removed at reduced temperatures, while still resulting in substantially complete removal of lubricant and elimination of sticking.

Such sticking was considered in the art to be attributable to oxidation of the lubricant and of the aluminum foil surfaces. Hence, in prior art methods, the avoidance of an oxidizing atmosphere during the annealing, whether dry or slick, was emphasized. Thus, in U.S. Patent 2,506,364, it is proposed to reduce oxidation by carrying out the treatment of aluminum foil in an atmosphere having an extremely low oxygen content, consisting of the incomplete combustion products of a mixture of gaseous hydrocarbons and containing less than 1% oxygen, and preferably less than about 0.6% oxygen, by volume.

In accordance with the present invention, it has been found, surprisingly and unexpectedly, that a coil of aluminum foil having a residual surface coating of rolling oil or lubricant can be freed from this oil or lubricant by heat treating the coil in the annealing range of temperatures up to about 850° F., and at the same time exposing the ends of the coil to the forced circulation of a stream of gas containing substantial amounts of free oxygen.

While not wishing to be limited to a particular theory of action, it is presently believed that the method of this invention accomplishes removal of residual lubricant deposits by creation of a concentration gradient within the coil.

In the prior art procedures, which emphasized merely volatilizing the lubricant, certain high-boiling fractions were necessarily unaffected, even at temperatures appreciably higher than those permissible in the present teaching. Of course, within a given temperature limitation, the unvolatilizable fractions are completely secure from the

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physical purging action of a gas stream which cannot penetrate between adjacent turns of a tightly wound coil.

In the method of the invention, however, a vastly increased purging effect is achieved by intentionally oxidizing those portions of the lubricant which are not readily volatilized. This is accomplished not by injecting the oxidant between the coil layers, but by passing the oxidizing gas stream across the exposed edges of the coil to create a concentration gradient between the innermost surfaces and the edge portions of the coil. Since a lesser proportion of the lubricant need be volatilized, a lower temperature will suffice; and the lower operating temperature itself contributes to the avoidance of sticking, by minimizing polymerization and similar undesirable effects.

The use of a substantial oxygen content in the purging gas stream is essential to the development and full utilization of the desired concentration gradient. The action resulting from such a gradient is similar to other diffusion mechanisms, such as those involving metallic alloys. See, for example, the definition of "diffusion" given at page 5 of the "Metals Handbook" (American Society for Metals, 1948).

Additionally, the action of a circulating fresh gas stream containing free oxygen, flowing past the ends of the coil, results in oxidation of the volatilized lubricant fractions to form carbon dioxide and water vapor, which are readily exhausted.

The result achieved by use of the present teaching does not depend upon penetration of the gas stream between the coil layers, although such penetration would be helpful. Rather, the action is that of producing continuous diffusion of residual oil or lubricant from between the coil surfaces to the coil edges, from which the emerging products are immediately swept away. It is to be emphasized that the lubricant products emerging from the coil are not only the volatilized fractions, but also included are the oxidation products of the unvolatilized residue which was previously immune to removal. In this manner, there is established a concentration gradient previously mentioned, until ultimately all lubricant has migrated to the coil edges and the inner surfaces are completely dry and free of any tendency to stick.

In accordance with the present invention, it is the forced circulation of a stream of gas containing free oxygen over the exposed ends of the coil which produces the removal of lubricant and leaves the coil freely unwindable without sticking. This is a result which could not be achieved by simply heating the coil in a non-oxidizing atmosphere and without forced circulation, even if the atmosphere contained a minimal amount of free oxygen, for without the gas circulation, the components of the lubricant would attain a condition of equilibrium with the surrounding atmosphere and would simply remain on the coil surfaces. Hence, while the avoidance of lubricant oxidation in a slick annealing process would promote non-sticking, there would be no removal of lubricant as is required for subsequent converting operations (such as printing).

Another advantage of dry annealing at low temperatures is that wrinkling of the foil due to thermal variations during heating is minimized.

The novel procedure of treatment of a coil of aluminum foil in the as-rolled condition to remove a residual surface coating of lubricant by exposing the ends of the coil to a stream of oxidizing gas containing free oxygen until the lubricant has been eliminated, leaving the coil freely unwindable, and preferably at a moderate range of temperature, runs entirely counter to the teachings of the prior art, and the remarkable results achieved are entirely unpredictable. It was always firmly believed by those skilled in the art that oxidizing conditions should be

avoided at all cost, particularly during annealing, in order to prevent sticking caused by oxidation or polymerization of the rolling oil. It is theorized that the sticking of foil encountered in prior art methods was attributable to the rise of a condition of stagnation, in which lubricant which was volatilized was permitted to attain a state of equilibrium with the atmosphere of the annealing furnace, whereby little oil was removed and much residue remained. Furthermore, the higher maximum annealing temperatures employed in prior art methods to anneal the coils in a given period of time were apparently responsible for the formation of intercrystalline aluminum oxide, which intensified the sticking. This oxide formation phenomenon is described, for example, in U.S. Patent 1,996,379. The forced circulation of fresh gas containing free oxygen, and the lower treating temperatures employed in the method of the present invention, minimize intercrystalline aluminum oxide formation and consequently eliminate sticking.

As conducive to a clearer understanding of certain features of the invention it may be noted at this point that aluminum foil, whether of substantially pure aluminum grade such as commercially pure aluminum, or of any of the aluminum-base alloy grades, is in widespread demand in the annealed condition for conversion by such subsequent operations as printing, ornamenting, bonding, laminating, or the like, and the foil made available for this is often referred to as converter foil or stock. Foil, as ordinarily defined, has a web thickness of about 0.006 inch or less. The gauge of course may range downward to 0.00017 inch or considerably less depending upon such factors as what purpose the product is to serve.

Insofar as accomplishing recrystallization of the metal is concerned, it is only necessary to heat at temperatures of about 850° F. or very much lower temperatures, for not much more than the period of time necessary to bring the whole coil up to temperature. However, the annealing treatment alone, even if prolonged within tolerable limits beyond recrystallization, does not produce a free unwinding product. Prolonged heat develops a dry cohesive sticky effect between the convolutions especially along marginal areas which accordingly take the form of bands lengthwise of the coil and when unwinding of the annealed convolutions is attempted a considerable resistance is encountered often leading to tearing. Foil also is easily stretched or wrinkled, as when efforts are made to unwind a coil having poor unwinding qualities. Wrinkling or stretching can at times be so severe as to be ruinous especially where a smooth, undistorted product surface is demanded. At moderate annealing temperatures heretofore employed the convolutions of the coils are separated by a slick zone which remains between outer dry areas, indicating incomplete drying. Such converter uses as printing the foil with printer's ink are not possible on the slick surface. The tendency for the product to remain slick after heat treatment becomes all the more pronounced at lower annealing temperatures such as in the vicinity of 650° F. in according with previous practice. Certain annealing procedures heretofore used have led to serious development of stains and discolorations at the annealing temperatures and the treated metal therefore is of poor surface quality.

The general object of the present invention, therefore, is the provision of a method for producing annealed aluminum foil in the form of a coil, the annealing being applied to the coil in the as-rolled condition with rolling lubricant still present on the metal surface, and which method yields an annealed coil product which is substantially completely dry, has minimum cohesion at the edges, is of good surface quality and may be unwound without tearing or wrinkling of the sheet material.

In accordance with the present preferred practice of this invention, coils of aluminum foil are placed in the as-rolled condition in a furnace and brought to a tempera-

ture within the range of about 575° F. to 725° F., while the coil, or at least its ends where the side edges of the foil appear, are exposed to air or other gas containing sufficient free oxygen to eliminate the oil slick on the foil surfaces. This temperature is maintained until the foil is annealed and a substantially dry product surface is obtained throughout the coil. There is a minimum hold-time at temperature required to complete the anneal and to eliminate slick, depending on the operating conditions and the width of the coil (5 hours is an average preferred minimum for standard coils, but much less time is required for very narrow-width coils), but this time can be increased without limit, except for economic considerations. Within the ranges of temperature and time set forth, it sometimes is beneficial to increase temperature or time or both for increased widths of the foil being treated. Thus, coils of strip up to about 14 inches in width are quite effectively treated at temperatures and for periods of time close to the 5 hour minimum, but coils of wider strip may require a little more time or temperature, or a combination of both.

During the heat treatment, the coils of aluminum foil are supplied with gas having a free-oxygen content which is sufficient to expedite elimination of the oil. The gas preferably has a free-oxygen content of at least about 5% when introduced into the furnace, as a practical matter, but it would be possible to reduce this figure if the gas should be moved rapidly past the coil without recirculation. A gas having a greater oxygen content or even pure oxygen itself may be supplied, but small amounts of oxygen down to about 5% by volume seem to be equally effective when coupled with the above-mentioned heating temperatures and periods of time used for treating the coils. Ordinary air is preferred for the purpose for it is quite effective in the furnace and is an economical source of oxygen in the amounts needed. The gas supplied around the coils advantageously is kept fresh such as by continuously feeding it through the furnace throughout the heat treatment, preferably without recirculation. In general, the preferred minimum rate of feed of gas containing 5% free oxygen, for example, is about 0.005 cubic foot (based on standard temperature and pressure) per hour per square foot of foil area (measuring the area on both sides of the foil in the coil) for purposes of elimination of slick during a five hour treatment period. This minimum rate of feed varies, of course, with variations of the period of treatment and with variations of free-oxygen content of the gas. In the case of air, for example, which contains 20% free oxygen, the minimum rate is four times less than the above-mentioned rate for gas containing 5% free oxygen. There is no upper limit on the rate of feed of the oxygen-containing gas, except as a matter of convenience and cost, since an excess of oxygen over that required to eliminate the slick will do no harm.

For the purposes of the invention, "aluminum foil" refers to foil composed of pure aluminum or any of the alloys composed principally of aluminum which are rolled into foil. Most of the foil rolled in the United States is 1235 alloy (Aluminum Association alloy designation, containing about 99.35% aluminum), and foil of that alloy is the foil referred to in the examples given below of the practice of the invention.

The residual oils found on rolled foil before annealing are of mixed composition, because the foil is passed through several successive roll stands as it is rolled down to gauge, and different rolling oils are usually applied at different stands. Moreover, rolling oils are seldom composed of a single constituent, but instead are usually made of a mixture of ingredients determined on a largely empirical basis, and these ingredients do not remain in constant proportions as the rolling oil is used during rolling operations. In general, the rolling oils with which the invention is concerned are composed of a mixture of at

least about 80% of a light petroleum oil (e.g., kerosene) and not more than about 20% of additive materials selected from vegetable and animal oils and acids having saponification values of about 148 to about 220 as determined by ASTM Standard D-94-52T entitled "Saponification Number of Petroleum Products by Color Indicator and Titration."

Residues of such mixed rolling oil are found on the bright side of foil. In some cases foil is rolled bright on both sides, but it is also common to fold the foil into two layers and then roll it so that each of the layers has a bright side and a matte side, the matte side being the surface folded against the other layer of foil. In some cases no lubricant is used on the matte side during rolling, and in other cases a lubricant is used, such as a light petroleum oil, usually without additive.

The amount of residual oil in the coil before annealing is typically about 5 to 7 milligrams per square foot of foil surface in the coil, calculated in terms of the sum of the area on both sides of each segment of strip in the coil, and such amount was present in the case of the following examples of the practice of the invention.

Example 1

A coil of 30 pounds of aluminum foil, matte one side, 0.00035 inch thickness, 13 $\frac{1}{8}$ inches wide, coiled on a 3 inch diameter aluminum core and having a build-up or thickness of approximately 2 inches and combined surface area on both sides of the foil of about 12,166 square feet, is mounted with the horizontally projecting ends of the core on a rack and is placed in a muffle-type electric furnace. The furnace is energized to raise the temperature of the coil at a rate of approximately 300° F. per hour until it reaches a temperature of about 600° F. and it is held at that temperature for about 5 hours. During the entire time the foil is in the furnace, ambient air is continuously supplied in substantially constant amount through the furnace without recirculation at a rate of about 450 cubic feet per hour with the coil exposed to the furnace atmosphere thus created. Finally the coil is removed from the furnace and allowed to air cool. The resulting foil is soft, of good surface quality, and dry with no excessively cohesive edge bands and accordingly is free unwinding from the coil.

Example 2

Same as Example 1 except that the ambient air is preliminarily dried to a dewpoint of less than -25° F. before being delivered to the furnace. The treated coil and the foil in it have substantially the same properties as those noted in Example 1, with negligible improvement.

Example 3

Same as Example 1 except that ambient air is delivered through the furnace at about 240 cubic feet per hour rather than at about 450 cubic feet per hour. Favorable results like those in Example 1 are obtained.

Example 4

Same as Example 1 except that about 24 cubic feet of ambient air per hour is circulated through the furnace. Favorable results like those in Example 1 are still obtained.

Example 5

(a) Same as Example 1 except that 10 liters per minute or approximately 21 cubic feet per hour of commercially pure oxygen is used in lieu of ambient air. Results still are excellent.

(b) On the other hand, special high purity dry nitrogen gas having a free-oxygen content of less than 0.1% or "Exogas" having a like free-oxygen content, when used in place of the air of Example 1, with all other conditions the same, produces poor results, and the surface of the foil in the treated coil is slick and not suitable for such further operations as printing the foil with printer's ink.

(c) However, when the free-oxygen content of such gas is raised to 5%, other conditions remaining the same, favorable results like those in Example 1 are obtained.

Example 6

A coil of 95 pounds of aluminum foil of 0.0004 inch gauge, shiny both sides, approximately 22 inches in width and about 8 inches in diameter (2 $\frac{1}{2}$ inch build-up) on a 3 inch core is placed in a small production-type resistance furnace modified with a compressed air inlet. The combined surface area of both sides of the foil is about 33,600 square feet. The foil is brought to a temperature of about 700° F. and held at that temperature for about 10 hours under compressed air flow of approximately 750 cubic feet per hour. The resulting foil is soft, of good surface quality and has free unwinding properties.

Example 7

A gas fired, radiant tube Swindell furnace preheated to 700° F. and equipped with a blower is charged with 5000 pounds of 0.00035 inch aluminum foil, matte on one side, in 22 coils having different web widths (19 $\frac{1}{8}$ inches to 27 $\frac{3}{8}$ inches), individual foil weights of 148 pounds to 310 pounds, 3 inch cores and over-all diameters of 10 inches to 12 $\frac{1}{4}$ inches, with a combined surface area (both sides) of about 20,000,000 square feet. After bringing it up to temperature, the charge is maintained at a temperature of 670° F. to 720° F. for about 5 $\frac{1}{2}$ hours in an air flow through the furnace from the blower of approximately 200 to 400 cubic feet per minute. Thereafter the load is pulled and allowed to cool in normal plant atmosphere. The coils have the good characteristics described in Example 1.

Example 8

A coil of 285 pounds of aluminum foil, 0.00035 inch web thickness, 22 $\frac{3}{8}$ inch width, and coiled on a 3 inch diameter aluminum core to an overall diameter of about 13 inches (5 inch build-up), having a combined surface area on both sides of the foil of about 1,140,000 square feet, is heated in a muffle-type electric furnace at a rate of approximately 300° F. per hour to about 650° F. and held at that temperature for about 5 hours. During that time ambient air is continuously supplied through the furnace at the same rate as in Example 1 with the coil exposed to the furnace atmosphere thus produced. As a result of the treatment the coil is soft, of good surface quality and has free unwinding properties.

Example 9

Same as Example 8 except that the holding temperature in the furnace is increased to 700° F. Results as compared with those in Example 8 are equally favorable.

Example 10

Same as Example 8 except that the coil is held for about four hours at temperature of 600° F. followed by about one hour at 650° F. Results are satisfactory and are as good as those in Example 8.

While present preferred embodiments of the invention, and methods of practicing the same, have been described, it will be understood that the invention may be otherwise embodied and practiced within the scope of the following claims.

I claim:

1. Method for the treatment of aluminum foil in coil form in the as-rolled condition to remove residual coatings of lubricant from the surfaces thereof, which comprises annealing said foil at a temperature up to about 850° F. while exposing the ends of the coil to circulation of a stream of gas containing a substantial amount of free oxygen and sufficient to oxidize and remove said lubricant, thereby oxidizing and removing substantially all traces of the lubricant, including fractions thereof which are not necessarily volatilized.

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2. The method of claim 1, in which the stream of gas is air.

3. The method of claim 1, in which the stream of gas is fresh air which has not been previously used in said stream.

4. Method for the treatment of aluminum foil strip in coil form in the as-rolled condition to remove residual coatings of lubricant from the surfaces thereof, which comprises annealing said coil at a temperature up to about 850° F. in an atmosphere containing at least about 5% oxygen by volume, while continuously circulating the oxidizing atmosphere across the ends of the coil, thereby oxidizing those portions of the lubricant coating adjacent

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to the edges of the coiled strip and causing the remainder of the lubricant to progressively migrate toward the edges whence it is likewise removed.

5. The method of claim 4, in which a temperature range of about 575° F. to about 725° F. is employed.

References Cited in the file of this patent

UNITED STATES PATENTS

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2,506,364	Jarvie et al.	May 2, 1950