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## CHEMICAL BRIGHTENING OF ALUMINUM

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This invention relates to the chemical brightening of aluminum and its alloys containing a major percentage of aluminum which will be hereafter referred to generically as aluminum.

This application is a continuation-in-part of my application, Serial No. 229,216, filed October 8, 1962.

The invention relates particularly to the brightening process disclosed in my Patent 2,729,551, dated January 3, 1956. In accordance with that patent, aluminum surfaces are treated with a mixture comprising, by weight, 40% to 98% of free phosphoric acid, 1/2% to 50% of free nitric acid, and 2% to 35% of water, as the essential ingredients, with the action taking place at an elevated temperature in the range from 150° F. to the boiling point of the mixture. In the event that specular finishes are not required and diffuse finishes are adequate, the nitric acid may be omitted. Times of immersion range from a few seconds to several minutes depending upon the alloy being treated, the temperatures used and the initial condition of the metallic surface, such shorter times of immersion being involved in the higher temperatures for given initial surface conditions. To these baths are sometimes added heavy metal salts, such as those of copper, which seem to have some catalytic action. Other materials are sometimes added for different purposes, such as sulphuric acid, alkali salts, aluminum salts, or the like. The present invention is concerned with the use of such treating baths in general. As will appear, the invention relates to modification of such baths which, apparently, affects their physical properties and actions rather than their chemical actions.

In the use of such baths several matters arise which would desirably be modified or improved. First, the time of immersion is often critical in that if immersion is carried out too long there may be produced etching or other action producing uneven finishes, or where specular finishes are desired, diffuse finishes may result. This occurs despite the fact that the desired action is that of selective solution of portions of the surface originally taking place to eliminate irregularities on the original mill finished surfaces. One aspect of the present invention is that it maintains the smoothness or uniformity of action of the baths over more extended periods of treatment. This is particularly desirable because due to mechanical manipulations it is sometimes difficult to remove a large amount of material from a bath sufficiently rapidly to prevent the detrimental extended period of action.

Even more detrimental than extended action of the bath, however, is the action taking place between the time of removal of work from a bath and initial rinsing which terminates the chemical action. What occurs may be readily understood from the fact that those bath compositions which, at elevated temperatures, produce specular and uniform finishes will, if cooled, produce diffuse finishes or uneven finishes. Here again the mechanical aspects of handling of the material enter the picture: the materials being treated must be handled by the use of hoists not only because of weight but because of the physically dangerous nature of the baths; and after the work is lifted from a bath it is cooled in the ambient atmosphere and since it carries the bath solution the chemical action will continue but at a much lower temperature than that

existing in the bath. Hence it has heretofore been necessary to effect the transfer from the polishing solution to a rinsing bath of water at a rate which is rather difficult to achieve. In accordance with the present invention a much longer transfer time becomes tolerable without detrimental effect.

When work is withdrawn from the bath polishing solution adheres thereto, and the polishing solution containing a high percentage of phosphoric acid is quite viscous. Excessive dragout of the polishing bath accordingly occurs with excessive loss of bath materials merely by adhesion. In accordance with the present invention the dragout is minimized, the bath composition draining more readily from the work.

It is also found that in accordance with the present invention the fuming of the bath is considerably lessened. During the operation of baths used heretofore nitric oxide fumes are emitted as the result of solution of the aluminum when nitric acid is a component of the bath. These are noxious and highly objectionable.

In accordance with the present invention there are added to a bath and dispersed therein small quantities of substances falling in the group of oils, fats and waxes. This, apparently, seems to have primarily physical, rather than chemical, actions.

The fact that the action is primarily of a physical type seems to be borne out by the highly divergent characters, from a chemical standpoint, of the materials of this group which may be used. As examples of the oils, fats or waxes, or materials derived therefrom, there may be cited the following:

- heavy mineral oils;
- fatty alcohols such as octyl, decyl, lauryl, myristyl, cetyl and stearyl alcohols;
- paraffin;
- soaps (which are probably decomposed by the acids in the bath to provide soap acids);
- diglycol stearate;
- coconut fatty acids;
- lanolin;
- peanut oil;
- linseed oil;
- lauric acid;
- tall oil fatty acid;
- hydrogenated castor oil;
- tallow;
- dimer acid;
- distearyl amine;
- coconut oil amine;
- okra gum;
- cottonseed oil;
- lard oil;
- sperm oil.

It is possible that in some instances, as where unsaturated fatty acid groups are involved, there may be some oxidation by the nitric acid in the bath but the products are fatty or oily in themselves and hence the additions function in the bath.

The oils added to the bath should be non-emulsifiable, that is, they should not be "soluble oils" of the type used, for example, in rolling aluminum.

The amounts of these materials added to the bath may be quite small to secure highly effective results. The amounts added may range from about 0.0005% to 1%, by weight of the bath, the bath being of one of the type described in my aforementioned patent. Less than 0.0005% is initially effective, but may result in rather rapid deterioration of effectiveness. More than 1% is not required since no better action is secured by using more, and too large a percent is objectionable because of

the difficulty of maintaining the material dispersed, and it may float on the surface adhering to the work as it is put into the bath, thus interfering with the chemical brightening action.

It is important that the oil, fat or wax material which is added should be dispersed and maintained so during operation as by mechanical stirring or by agitation such as produced by injection of air bubbles. Under such conditions the bath has a milky appearance due to the dispersed material. However, as in cessation of work at the end of a working period, the agitation is stopped, the material added will generally separate and float on the bath surface. Before resuming operations the agitation is then desirably carried out to re-establish the dispersed condition.

All other aspects and conditions of operation are similar to those described in my patent.

A typical composition which has been found highly satisfactory is composed, by weight, of 79.00% of 100% phosphoric acid, 6.84% of sodium nitrate (which by the action of phosphoric acid forms free nitric acid), 0.26% lauryl alcohol, and 14.00% water. This composition is typical of compositions utilizing others of the materials listed above in place of the lauryl alcohol, and any of these materials or mixtures thereof may be present in total amounts ranging, as already indicated, from around 0.0005% to 1% or more. It has also been found that various derivatives of the materials of the type enumerated may be used though it has been found desirable not to use chlorinated derivatives because chlorine seems to have a highly detrimental effect on proper polishing by baths of the type under consideration.

A composition which is particularly effective and has the advantage of low cost is one identical with that given but with lard oil substituted for lauryl alcohol in the same amount.

The action of the oily or similar materials is not fully explainable, but one aspect of operation which would seem to indicate that surface tension effects enter the picture is the quite radical reduction in the amount of dragout. It has been found by measurement that operation with baths in accordance with the invention reduces the dragout by as much as 40% as compared with prior baths, based on the treatment of flat sheets which may drain without entrapment of the solution. It seems probable that an oily film is formed on the surface of the metal as it is withdrawn to isolate it from the clinging liquid so that rapid drainage back into the treating bath is effected.

This isolation may also account for the considerable reduction of rate of action as the work is transferred from the polishing bath to the rinsing bath. Apparently the chemical action is abruptly stopped, or at least slowed down, by a film of the oily material, and this makes tolerable much more extended time of transfer, increasing the facility of handling. This action may also be involved in lessening the deterioration resulting if work is permitted to remain too long in the bath. If a film is formed, however, it is apparently not so continuous in the bath that it greatly inhibits the desired action. This is in

contrast with the inhibiting of the polishing action if, for example, oily material is floating on the bath and the work is lowered therethrough in which case a substantially continuous film tends to isolate the work from the acids of the bath.

The slowing down of the emission of nitric oxide fumes is also perhaps due to the slowing down of the reaction by the oily material, though this may well not be an accurate explanation of the result.

The action is apparently physical rather than chemical since, as will be apparent from the listing of useable materials, they have no general resemblances in chemical composition. Since at the temperatures of the baths the fats, waxes and other material added, or the substances resulting from action of the baths on added materials, are "oily" as well as added substances which may be called oils, it appears that the resulting baths may be most aptly defined as acidic baths having dispersed therein oily materials which are insoluble in the baths.

It will be evident from the foregoing that various modifications may be made in accordance with the invention without departing from the scope thereof as defined in the following claims.

What is claimed is:

1. A bath for the chemical brightening of aluminum when used at a temperature in excess of 150° F. comprising as essential constituents phosphoric acid and water, with the free phosphoric acid content in the range from 40% to 98% by weight, said bath having dispersed therein, at its operating temperature in the range 150° F. to boiling, an oily material insoluble in, and non-emulsifiable in, the bath, the content of the oily material being substantially in the range from 0.0005% to 1.0%.

2. A bath for the chemical brightening of aluminum when used at a temperature in excess of 150° F. comprising as essential constituents phosphoric acid, nitric acid and water, with the free phosphoric acid content in the range from 40% to 98% and with the nitric acid content in the range from ½% to 50%, by weight, said bath having dispersed therein, at its operating temperature in the range 150° F. to boiling, an oily material insoluble in, and non-emulsifiable in, the bath, the content of the oily material being substantially in the range from 0.0005% to 1.0%.

3. A bath according to claim 2 in which the oily material is lauryl alcohol.

4. A bath according to claim 2 in which the oily material is lard oil.

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