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2,736,640

CHEMICAL POLISHING BATH

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No Drawing. Application December 1, 1954,
Serial No. 472,520

5 Claims. (Cl. 41—42)

This invention pertains to chemical polishing of metals and more particularly to an improved method for chemical polishing of aluminum.

Bath compositions for the chemical polishing of aluminum have been described in the literature. Most of these compositions, although efficient polishing baths, are hazardous to handle, being ordinarily composed of mixtures containing nitric acid, hydrofluoric acid and the like. The use of such chemicals requires definite precautions on the part of the operator and the fumes generated by such polishing baths are a health hazard.

The chemical polishing method described in Cochran in U. S. 2,613,141, represents a substantial improvement over the older methods involving the use of hazardous acid mixtures. This method makes use of a solution of phosphoric acid, containing hydrogen peroxide. Such a solution does not give rise to objectionable fumes and is much safer to handle than the acid solutions previously described in the art.

At the proper operating temperature, that is between 190° F. to 230° F., the phosphoric acid-hydrogen peroxide bath will polish aluminum to a mirror finish. In this temperature range and even at somewhat lower temperatures the bath is, however, somewhat unstable as far as the hydrogen peroxide component is concerned. That is to say that hydrogen peroxide in the operating bath is not only used up by the polishing action proper but also lost by decomposition. The polishing action depends to a large extent on hydrogen peroxide concentration in the bath. Although minor variations in hydrogen peroxide concentration in the bath will not materially affect the polishing action, this action will cease if hydrogen peroxide concentration drops substantially below a certain level.

It is possible to reduce hydrogen peroxide decomposition by the addition of suitable stabilizers such as acetanilide, dipicolinic acid and others. But even a properly stabilized bath will lose its polishing efficiency at about the rate at which hydrogen peroxide concentration in the bath drops. Within the effective range of hydrogen peroxide concentration, polishing will take place at a rate determined by operating conditions, particularly by temperature. The economic efficiency or working capacity of the bath is therefore largely determined by two reaction rates; one, the rate of chemical decay and two, the rate of polishing. The combination of these two rates will determine the surface area of aluminum that can be polished by a given volume of bath.

There are practical limits below which the rate of chemical decay can not be reduced in an operating bath of this type. If it is desired to increase the working capacity of the bath, the only way in which this can be done is to increase the rate of polishing. With a given rate of chemical decay, a higher polishing rate will permit to polish a larger surface area with a given volume of bath and in doing so will correspondingly decrease polishing cost per unit of surface area.

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It has now been found, in accordance with the present invention, that the polishing rate of the phosphoric acid-hydrogen peroxide bath can be increased substantially without increasing the rate of chemical decay, if relatively minor amounts of finely divided graphitic carbon are dispersed in the bath. It is very surprising that graphitic carbons dispersed in the phosphoric acid-hydrogen peroxide solution are able to improve bath performance. It is well known that hydrogen peroxide solutions are easily decomposed by finely divided solids dispersed in the solution even if these finely divided solids are of a material which, in bulk form, has little or no effect on hydrogen peroxide stability. For example, fused quartz is inert towards hydrogen peroxide and the latter will be completely stable in a quartz vessel. However, finely divided quartz dispersed in a hydrogen peroxide solution will very often cause noticeable decomposition of the hydrogen peroxide. It was, therefore, quite unexpected to discover that graphitic carbon, even in finely divided form, does not promote decomposition of the phosphoric acid-hydrogen peroxide bath but actually promotes bath efficiency.

In accordance with the method of the present invention, the chemical polishing bath for aluminum considered here will contain, in addition to the dispersed carbon, about 60% to 80% of phosphoric acid, about 1% to 10% of hydrogen peroxide and water. If desired, the bath may further contain a hydrogen peroxide stabilizer, for instance about 0.5% of dipicolinic acid. These percentages, all of which are percent by weight, may be varied somewhat.

The finely divided graphitic carbon is dispersed in the bath to the extent of 1% to 3% by weight on the weight of dispersing solution. Examples of graphitic carbon suitable for this use are graphite itself, both natural and synthetic, and carbon blacks such as furnace black, channel black and acetylene black.

Carbons other than graphitic carbons do not perform satisfactorily. Therefore, carbons such as active carbon, charcoal and the like are not within the scope of this invention.

After the bath is made up it is brought to the proper starting temperature. Because of the exothermic nature of the polishing reaction, temperatures tend to rise during operation of the bath. Operating temperatures are within the range of about 160° F. to 230° F. The aluminum articles to be polished are merely solvent cleaned and then immersed in the bath for about 1/2 to 5 minutes, withdrawn, rinsed with water and dried.

In chemical polishing, the polishing rate can be measured conveniently by measuring the rate of metal removal by the bath. A reasonably high rate of metal removal is a necessary pre-requisite in chemical polishing but is not the only factor determining the polishing ability or polishing efficiency of a chemical polishing bath. Still, metal removal rate can be considered as a practical measure of bath efficiency. The higher the rate of metal removal or polishing rate, the greater the surface area that can be polished with a given volume of a bath of a given chemical decay rate. Whereas a given volume of a given bath may permit polishing of, say, 1 square foot, the same bath at a higher polishing rate may permit polishing of, say, 4 square feet, thus reducing chemical polishing cost by three-quarters.

The following examples will serve to illustrate the principle of this invention. In these examples the polishing rate is expressed throughout in terms of rate of metal removal, the higher values indicating a higher polishing rate and, therefore, correspondingly lower polishing cost.

Example 1

A bath was prepared containing, by weight, 70%

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H_3PO_4 , 5.3% H_2O_2 and 0.5% dipicolinic acid, in aqueous solution, and heated to the proper starting temperature of about 195° F. The bath volume was 200 ml. A solvent cleaned strip of 2S-aluminum of 6 square inch total surface was then dipped into the bath for about 3 minutes then withdrawn, rinsed off with water, and dried.

The treated aluminum strip was slightly brighter in appearance than before but did not have a mirror finish or polish. Measurement of the strip after treatment indicated that metal removal rate by this bath was about 0.3 mil per minute.

Example 2

A bath was prepared exactly as in Example 1 but the bath contained in addition, 1% by weight of acetylene black. An aluminum strip was treated as in Example 1 and showed, after treatment, a mirror finish. Metal removal rate in this case was 0.8 mil per minute.

Example 3

This corresponds exactly to Example 2 but 3% acetylene black were added to the bath. A mirror finish was again produced and the metal removal rate was 1.1 mil per minute.

Example 4

To a bath containing 70% H_3PO_4 , 5.3% H_2O_2 , in aqueous solution, was added 1% of a furnace black. The bath was heated to a starting temperature of about 195° F. and a solvent cleaned strip of 2S-aluminum was immersed in the bath for 4 minutes. A mirror finish was obtained and the metal removal rate was 0.5 mil per minute.

Example 5

This corresponds to Example 4 but 1% of powdered graphite was added to the bath. The starting temperature was 205° F. A mirror finish was obtained by treating a solvent cleaned strip 2S-aluminum for 2 minutes in that bath and metal removal rate was 0.6 mil per minute.

Example 6

This corresponds to Example 4 but 1% of a channel black was added to the bath and starting temperature was about 195° F. A mirror finish was obtained by treating a solvent cleaned strip 2S-aluminum for 5 minutes in that bath and metal removal rate was 0.46 mil per minute.

The following examples will further serve to illustrate the improvement in working capacity of a standard polishing bath obtained by the addition of a graphitic carbon suspension to the bath.

Example 7

A bath was prepared containing 70% H_3PO_4 , 5.3%

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H_2O_2 , in aqueous solution, heated to a starting temperature of 195° F. and then used to treat solvent cleaned strips of 2S-aluminum. Bath volume was 200 ml. and the surface area of the individual aluminum strips was 6 square inches each.

This bath produced a mirror finish on one strip and etched subsequent strips, indicating a very low working capacity. Rate of metal removal was about 0.4 mil per minute.

Example 8

A bath was prepared as in Example 7, stabilized with 0.5% dipicolinic acid, and 1% of acetylene black was added to it. The bath was again brought to a starting temperature of 195° F. and then used to treat solvent cleaned strips of 2S-aluminum. Bath volume was again 200 ml. and the surface area of the individual aluminum strips was 6 square inches each.

This bath produced a mirror finish on 4 strips, indicating a substantially increased working capacity. Rate of metal removal was about 0.8 mil per minute.

What is claimed is:

1. In the method of chemically polishing aluminum with a bath containing phosphoric acid and hydrogen peroxide, the improvement which comprises having present in said bath from about 1% to about 3% of graphitic carbon.

2. In the method of chemically polishing aluminum with a bath containing phosphoric acid and hydrogen peroxide, the improvement which comprises having present in said bath from about 1% to about 3% of acetylene black.

3. In the method of chemically polishing aluminum with a bath containing phosphoric acid and hydrogen peroxide, the improvement which comprises having present in said bath from about 1% to 3% of channel black.

4. In the method of chemically polishing aluminum with a bath containing phosphoric acid and hydrogen peroxide, the improvement which comprises having present in said bath from about 1% to about 3% of furnace black.

5. In the method of chemically polishing aluminum with a bath containing phosphoric acid and hydrogen peroxide, the improvement which comprises having present in said bath from about 1% to about 3% of graphite.

References Cited in the file of this patent

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