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PROCESSES OF ELECTROLYTIC POLISHING OF METALS

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8 Claims. (Cl. 204-140.5)

The invention relates to processes for electrolytic 15 polishing of metals.

The main object of this invention is to devise a process for the electrolytic polishing of aluminum articles, or articles formed from an aluminum-rich alloy so that a lustrous mirror-like surface having a high specular reflec- 20 tivity free from pitting is obtained.

A further object is to provide such a process which is effective to produce lustrous effects in recessed surfaces.

A process of producing a smooth, lustrous surface on articles of aluminum or aluminum-rich alloy according to 25 the invention is characterized by immersing the articles in a heated electrolyte containing phosphoric acid, sulphuric acid, a phosphoric acid ester of a polyhydric alcohol or salt thereof and water, whereby etching of the articles is effected and then subjecting the immersed articles to 30 direct current voltage, the article being made anode in the bath.

An electrolyte for use in producing a smooth lustrous surface on aluminum or aluminum-rich alloys according to the invention is characterized by comprising phosphoric 35 acid in predetermined amount, sulphuric acid, a phosphoric acid ester of a polyhydric alcohol or salt thereof and water.

The electrolyte may have a total acid content of from 45 to 90 per cent by weight. Phosphoric acid, preferably 40 orthophosphoric acid may constitute 45 to 80 per cent by weight of the electrolyte, sulphuric acid from 5 to 20 per cent by weight and the phosphoric acid ester of a polyhydric alcohol may be present in proportion of from 1 to 5 per cent by weight. 45

In carrying the invention into effect in a preferred manner an electrolyte is prepared from the acids specified and from 1 to 5 per cent of glycero phosphoric acid or its sodium or potassium salt and is heated to a temperature from 70° to 90° C. The article to be polished, 50 which may be given a cleaning treatment and a light mechanical polishing, is then immersed in the electrolyte. After a period of from 30 to 60 seconds during which etching of the article occurs, direct current potential at 7 to 25 volts is applied, the article being made the anode. 55 The voltage is regulated to give a current density at the anode of from 100 to 180 amperes per square foot. Controlled agitation of the electrolyte is beneficial during the treatment and is preferably produced by giving the article a to-and-fro motion in the bath through the medium of 60 a reciprocatory movement of the anode rod supporting the article. A time of electrolytic treatment of from 2 to 20 minutes may be given according to the degree of lustre required in the final product. The article is then removed from the bath and rapidly washed. A thin 65 surface film usually present on the polished surface may be removed by immersion in a suitable solution and the article may then be anodized and, if desired, dyed.

The following is a preferred embodiment of the invention, as applied, by way of example, to the production of 70 a lustrous surface on electric torch cases manufactured by impact extrusion from aluminum or aluminum alloy,

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one end of the torch being permanently closed, while the other is preferably flared to receive a reflector and a transparent cover.

An electrolyte is prepared from the following:

Per cent by w	eight
Orthophosphoric acid	67.5
Sulphuric acid	15.5
Sodium glycerophosphate	1
Water	16.0

The electrolyte is heated to 70° to 90° C. and the torch case after having received preparatory treatment for removal of grease and dirt is immersed therein for a period of 30 to 60 seconds without application of a potential. Direct current voltage is then applied, the torch case being made the anode, an anode current density of approximately 150 amperes per square foot being employed. Cathodes of lead or graphite, preferably lead may be used. They should be substantially greater in area than the projected area of the torch case being treated; a ratio of 6 to 1 has been found to be satisfactory.

The time of treatment varies according to the smoothness of the original surface of the torch case, but varies from approximately 2 to 20 minutes.

After this period the torch case is removed from the bath and is rapidly swilled, e. g. in water which may be hot or cold or contain suitable known wetting agents such as those known under the registered trademarks "Teepol" and "Lissapol" to render swilling more rapid and effective. It then has a brilliant appearance free from surface scratches and with a high specular reflectivity.

After polishing, the articles have a thin surface film which it is desirable to remove before the subsequent anodizing process. This film can be removed in known manner by immersion of the article in a solution of a mixture of chromic and phosphoric acids or chromic and sulphuric acids, or solutions of the salts of these acids. The time of immersion in such solutions should be no longer than is necessary to remove the surface film and may for example vary from 30 seconds to 5 minutes.

After further swilling the torch case may be, in accordance with the invention, then subjected to anodic treatment in a bath containing either chromic acid (CrO₃) or sulphuric acid (H2SO4) according to whether an opaque or transparent protective film respectively is desired. A suitable electrolyte for anodic oxidation is one consisting of a 15 per cent by volume solution of sulphuric acid employed at a temperature of 20° to 22° C. and an elec-tric potential difference of 12 volts. The anodic film can subsequently be dyed, and, if desired, sealed for example by immersion in hot water to render the absorptive anodic film impermeable non-absorptive and non-staining, and also to render the torch case unaffected by finger marking. The anodized and dyed film has a very satisfactory fastness to light. Alternatively, if a clear highly polished metallic finish is desired, the dyeing may be omitted and the coating sealed immediately after the said further swilling.

The composition of the electrolyte and conditions of electrolysis cited above can be modified considerably without departure from the scope of the invention. For example, the percentage by weight of orthophosphoric acid may be from 45% to 80%, and the percentage by weight of sulphuric acid may be from 5% to 20%, with the total acid concentration 45% to 90% by weight, whilst the temperature range may be between 60° C. to 100° C. without detrimental effect. Under these conditions satisfactory results can be obtained with current densities varying between 80 and 250 amperes per square foot, corresponding to electrical potentials of between 7 and 25 volts, a preferred current density range being 100 to 150 amperes per square foot.

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By the present invention shaped articles of aluminum or alloys of aluminum can be given a lustrous surface even though the surface of the article before treatment is uneven, scratched or mildly scored and the mirror-like finish is obtained without deformation of the article from the shape possessed before the process was commenced. This is particularly important as regards extruded or deep-drawn articles of the metals mentioned because of their liability to deformation, e. g. with radial pressure.

However, in accordance with the present invention a 10 light mechanical polishing may be advantageously effected on the article before being subjected to electrolytic polishing by the process described, and subsequently anodic treatment may be effected in a chromic acid-type or sulphuric acid-type bath to obtain an opaque or transparent 15 protective film as desired, which film may be dyed.

The electrolytes of the invention have good throwing power, the inner surfaces of recessed and cup-like articles, such as for example reflectors, taking on a satisfactory polish even on the deepest portions.

By the present invention a highly reflective surface on aluminum or aluminum-rich alloys may be obtained which, after anodizing and dyeing, has a superior fastness to light or resistance to fading as compared with any results obtained by mechanical polishing.

What is claimed is:

1. A process for producing a smooth, lustrous surface on an article of aluminum or an aluminum-rich alloy, said process comprising immersing said article in an electrolyte, said electrolyte comprising, by weight, 45% to 80% phosphoric acid, 5% to 20% sulfuric acid, the total of the acids being not more than 90% of said electrolyte, 1% to 5% in the aggregate of at least one of the phosphorus containing material in the group consisting of a phosphoric acid ester of a polyhydric alcohol and a salt of said electrolyte, and, subsequently, subjecting said article to a direct current voltage while making said article an anode in said electrolyte.

2. A process according to claim 1 wherein said phosphorus containing material is glyceryl phosphate.

3. A process according to claim 1 wherein said phosphorus containing material is the sodium salt of glyceryl phosphate.

4. A process according to claim 1 wherein said phosphoric acid is orthophosphoric acid.

5. A process according to claim 4 wherein said phosphorus containing material is glyceryl phosphate.

6. A process according to claim 4 wherein said phosphorus containing material is the sodium salt of glyceryl phosphate.

7. A process for producing a smooth, lustrous surface on an article of aluminum or an aluminum-rich alloy,

said process comprising immersing said article in an electrolyte at a temperature of from 70° C. to 90° C., said electrolyte comprising, by weight, 45% to 80% phosphoric acid, 5% to 20% sulfuric acid, the total of the radius temperature from 00% of said electrolyte 1%

acids being not more than 90% of said electrolyte, 1% to 5% in the aggregate of at least one of the phosphorus containing materials in the group consisting of a phosphoric acid ester of a polyhydric alcohol and a salt of said ester, the remainder water, etching said article in said electrolyte, and, subsequently, subjecting said article

20 to a direct current voltage while making said article an anode in said electrolyte.

8. An electrolyte for use in producing a smooth, lustrous surface on an article of aluminum or an aluminum-rich alloy which comprises by weight 45% to 80%
25 phosphoric acid, 5% to 20% sulfuric acid, the total of the acids being not more than 90% of said electrolyte, 1% to 5% in the aggregate of at least one of the phosphorus containing materials in the group consisting of a phosphoric acid ester of a polyhydric alcohol and a salt 30 of said ester, the remainder water.

References Cited in the file of this patent UNITED STATES PATENTS

2,708,655	Turner May 17, 1955	5
	FOREIGN PATENTS	
54,614	France July 7, 1950	0
558,925	Great Britain Jan. 27, 1944	4
606,240	Great Britain Aug. 10, 1948	8
	54,614 558,925	FOREIGN PATENTS 54,614 France July 7, 195 558,925 Great Britain Jan. 27, 194

OTHER REFERENCES

"The Iron Age, Sept. 5, 1946, pages 50-53, paper by Mason.

"Transactions of the Electrochemical Society," vol. 78 (1940), pages 265–272, paper by Uhlig.