# 2,692,853

# UNITED STATES PATENT OFFICE

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#### PROCESS AND COMPOSITION FOR ELEC-**TROPOLISHING STAINLESS IRON AND** STEEL

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# 8 Claims. (Cl. 204-140.5)

This invention relates to the electrolytic polishing of stainless iron and steel. Stainless iron and steel are defined as iron base alloys containing up to 30% chromium, with or without substantial proportions, up to 40%, of nickel, and 5 optionally, supplemental small proportions of manganese, silicon, molybdenum, columbium, titanium, etc., for special purposes.

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According to this invention, stainless iron or stainless steel is electrolytically polished by mak- 10 ing it the anode in an electrolyte comprising an acid of the group consisting of sulfuric and orthophosphoric acids and mixtures thereof, water, and a material of the group consisting of humic acid and alkaline salts of humic acid. Through- 15 out the specification and claims, the alkaline salts of humic acid include the ammonium salt as well as the salts of the alkali metals, and the term phosphoric acid is used to refer to orthophosphoric acid. Temperature, voltage, current den- 20 only for those compositions in which the mineral sity, polishing time, and the use of enhancing materials, in this invention, vary within the usual limits covered by these factors in conventional electropolishing processes.

Humic acids are the complex acids derived 25 from the decomposition products of vegetable matter, e. g. peat, lignite, and humus. In the examples of this application, the use of humic acid derived from peat is illustrated, but humic acids from other sources are equally suitable. Processes for the preparation of humic acid are 30 described by Thiessen and Engelder, "Isolation of Humic Acids," Industrial and Engineering Chemistry, 22:1131 (1930), and by Esh and Guha-Sircar, J. Indian Chem. Soc., 17:326-31 (1949).

Since humic acid is insoluble in water and 35 most dilute acids, it is usually prepared by precipitation with strong mineral acid from an alkaline aqueous solution. The alkaline salts of humic acid can be prepared by evaporation of the water from an alkaline aqueous solution of 40 humic acid. Humic acid precipitates are somewhat colloidal in nature and difficult to filter, hence the alkaline salts of humic acid, which are more easily isolated, are usually used, and when necessary, used in acid media to form humic acid 45 preferred temperature range is from 30° to 80° in situ

The proportions of ingredients used in the electrolyte of this invention may be varied within fairly wide limits. The current density required for polishing is governed by the composition of 50 the electrolyte, but in all cases should be at least 0.2 amp. per square inch on the anode, and may be up to 20 or more amps. per square inch. Excessively high current densities are undesirable because of consequent local overheating of elec- 55

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trolyte, excessive generation of gas, and increased tendency for pitting caused by local galvanic action. When sulfuric acid is used, the electrolyte should contain about 35% to 70% of sulfuric acid, about 10% to 60% of water, and about 0.2% to 40% of humic acid or alkaline salt of humic acid, the larger proportions of humic acid or its salt being required with the larger proportions of water. When phosphoric acid is used, the electrolyte should contain about 40% to 80%phosphoric acid, about 10% to 55% water, and about 0.2% to 25% humic acid or alkaline salt of humic acid. When both sulfuric acid and phosphoric acid are used in the electrolyte, any relative proportion of the two is satisfactory, the proportion of humic acid or its alkaline salt being in the range between 0.2% and 25%, the proportion of water being between 60% and 1%, the part of this latter range below 10% being suitable acids are present in nearly equal proportions, and the mineral acids making up at least 30% of the electrolyte composition. The higher proportions of humic acid or alkaline humate make the electrolyte viscous, hence the lower proportions of humic acid or its salt are preferred. In all the suitable electrolyte compositions, the preferred proportion of humic acid or its alkaline salt is 2% to 10% by weight of the composition.

The electrolytes may also contain small proportions of additional agents added to enhance the polishing effect of the electrolysis, for example alcohols, which have been used in the prior art to enhance the lustre of the polished metal. Methanol has been used to replace water up to 10% by weight of the electrolyte composition of this invention.

The time required for polishing depends primarily on the condition of the original surface. Polishing times as short as three minutes are often sufficient for a satisfactory polish, and longer times are required at lower temperatures, lower current densities, and lower rates of action of the electrolyte of the material being polished.

When using sulfuric acid in the electrolyte, the C., and the preferred current density on the anode is from 0.5 to 2.0 amps. per square inch. The most desirable composition of the essential ingredients of the electrolyte has been found to be 50% to 60% by weight sulfuric acid, 2% to 4% sodium humate, remainder water. Sodium humate, the sodium salt of humic acid, forms a colloidal precipitate of humic acid in the strongly acidic electrolyte.

When using phosphoric acid in the electrolyte,

the preferred temperature range is from 80° to 100° C. and the preferred current density on the anode is from 0.5 to 2.5 amps. per square inch. The most desirable composition of the essential ingredients of the electrolyte has been found to 5 be about 60% by weight phosphoric acid, about 5% sodium humate, remainder water. Higher proportions of phosphoric acid and sodium humate increase the viscosity of the electrolyte and favor the formation of froth during elec- 10 10% to 60% water and 0.2% to 40% of an alkaline trolysis. Proportions of sodium humate within the operating range, i. e. up to 25% by weight of the electrolyte, are soluble in the phosphoric acid, but the electrolyte functions as effectively as when humic acid is present as a precipitate, as 15 extent of 10% by weight of the electrolyte. it is in the sulfuric acid electrolyte.

#### Example I

A sample of A. I. S. I. type 304 cold rolled stainless steel was polished at a current density of 20 0.66 amp. per square inch and 4.5 volts, in an electrolyte consisting of 55% H2SO4, 42% water, and 3% sodium humate held at a temperature of about 60° C. The sample was polished for 5 25minutes, at the end of which time it had a bright mirror-like, polished surface.

#### Example II

A sample of type 304 cold rolled stainless steel 30 was polished at a current density of 1.7 amps. per square inch at 5 volts, in an electrolyte consisting of 80% phosphoric acid, 18% water, and 2% sodium humate held at a temperature of about 80° C. The sample was polished for 5 minutes, 35 in which time it attained a bright mirror surface.

#### Example III

A sample of type CF-8M cast, partially ferritic, stainless steel was polished at a current 40 density of 4 amps. per square inch at 7 volts in an electrolyte consisting of 57% water, 18.5%sulfuric acid, 18.5% phosphoric acid, and 6%sodium humate, held at a temperature between  $80^{\circ}$  and  $90^{\circ}$  C. The sample attained a bright 45mirror surface with five minutes electropolishing.

Although the proportion of humic acid or alkaline humate required in the electrolyte of this invention may be small, its presence is critical none-the-less. An electrolyte containing just 50 sulfuric or phosphoric acid and water will produce a polish on a stainless iron or steel anode over only a narrow range of temperature and current density, outside of which it will merely etch the anode. Hence the action of humic acid 55 the anode in an electrolyte consisting essentially or alkaline humate in producing a bright polish over a wide range of acid concentration, temperature, and current density is remarkable in view of the small proportions which are satisfactory. An aditional benefit attained by the use  $^{60}$  acid. of humic acid or its alkaline salt in electropolishing is an unexpected and marked increase in the "throwing power" of the current, which is the ability of the current to polish in the recesses of irregularly shaped objects. A further advantage 65 achieved is the ability to use electrolytes containing higher proportions of water than are possible without humic acid or alkaline humate, thereby reducing the consumption of relatively expensive 70 acid.

I claim:

1. An electrolyte for use in the electrolytic polishing of stainless iron and steel articles wherein the article is employed as an anode, said 75electrolyte consisting essentially of an acid of the

group consisting of sulfuric and orthophosphoric acids and mixtures thereof, water, and an amount of a material of the group consisting of humic acid and alkaline salts of humic acid effective to produce a bright polish over a wider range of acid concentration, temperature, and current density than in the absence of the member of said group.

2. An electrolyte, as claimed in claim 1, consisting essentially of 35% to 70% sulfuric acid,

salt of humic acid, all percentages being expressed on a weight basis.

3. An electrolyte, as claimed in claim 2, wherein methanol replaces part of the water up to the

4. An electrolyte as claimed in claim 1, consisting essentially of 40% to 80% orthophosphoric acid, 10% to 55% water, and 0.2% to 25%of an alkaline salt of humic acid, all percentages being expressed on a weight basis.

5. An electrolyte for use in the electrolytic polishing of stainless iron and steel articles wherein the article is employed as an anode, said electrolyte consisting essentially of 0.2 to 25% of a material of the group consisting of humic acid and alkaline salts of humic acid, 10% to 60% of water, and a mixture of sulfuric and orthophosphoric acid, said mixture comprising at least 30% of said electrolyte, all percentages being expressed on a weight basis.

6. The art of electropolishing a stainless iron or steel product which comprises (1) making the product the anode in an electrolytic cell containing an electrolyte consisting essentially of the electrolyte claimed in claim 2, and (2) while maintaining the temperature of the electrolyte in the range from 30° to 80° C., (3) applying a voltage to the electrolytic cell to maintain a current density of at least 0.2 amp. per square inch on the surface of the anode, whereby the product is electrolytically polished.

7. The art of electropolishing a stainless iron or steel product which comprises making the product the anode in an electrolytic cell containing an electrolyte consisting essentially of the electrolyte claimed in claim 4, and, while maintaining the temperature of the electrolyte in the range from 80° to 100° C., applying a voltage to the electrolytic cell to maintain a current density of at least 0.2 amp. per square inch on the surface of the anode, whereby the product is electrolytically polished.

8. A process for electropolishing a stainless iron or steel article comprising making the article of (1) an acid of the group consisting of sulfuric and orthophosphoric acids and mixtures thereof, (2) water, and (3) a material of the group consisting of humic acid and alkaline salts of humic

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