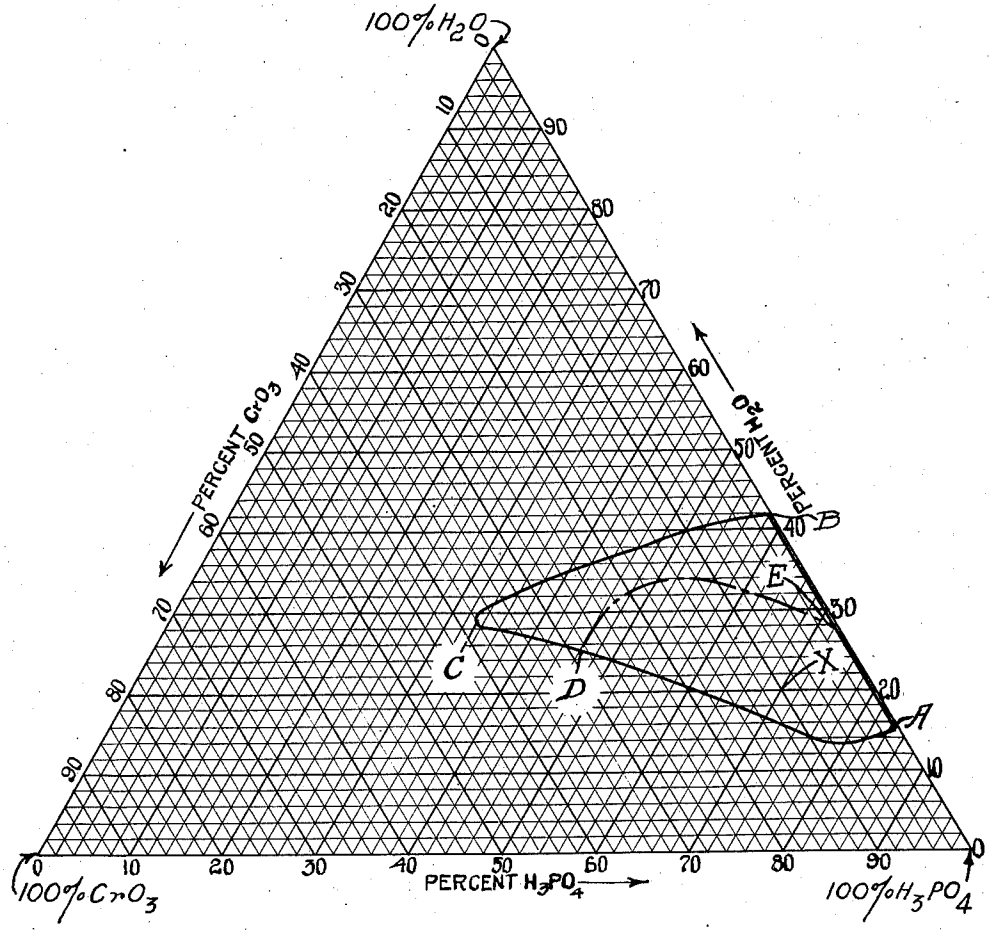
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METHOD OF ANODICALLY POLISHING NICKEL

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METHOD OF ANODICALLY POLISHING NICKEL

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This invention relates to a method of and an electrolyte for anodically polishing nickel, electrodeposited nickel, non-ferrous nickel alloys and the like. More particularly the invention relates to an aqueous electrolyte comprising phosphoric acid, chromic acid and water and to the use of such electrolyte in the anodic polishing of nickel.

I have now found that an aqueous electrolyte comprising phosphoric acid, chromic acid and water, as the essential ingredients, can be satisfactorily used in the anodic polishing of nickel. If the relative proportions of these ingredients be kept within certain limits, which I have determined, very satisfactory polishes can be produced in the case of nickel, while at the same time the surfaces obtained are relatively superior as regards the absence of amorphous layers and freedom from mechanical strain, to those obtainable by mechanical polishing or buffing operations.

It is therefore an important object of my invention to provide an electrolyte containing phosphoric acid, chromic acid and water within certain well defined limits as to their relative proportions, for use in the anodic polishing of nickel, electrodeposited nickels, non-ferrous nickel alloys and the like, to impart thereto a highly lustrous surface.

It is a further important object of this invention to provide a method of anodically polishing nickel, electrodeposited nickel, non-ferrous nickel alloys and the like, using an electrolyte of novel composition that is operative throughout a wide range of anode current densities and temperatures.

Other and further important objects of this invention will become apparent from the disclosures in the specification and the accompanying drawing.

This invention (in its preferred form) is illustrated in the drawing and hereinafter more fully described.

On the drawing:

The figure represents a triaxial diagram indicating the relative proportions of phosphoric acid, chromic acid and water in compositions of electrolyte coming within the scope of my invention.

In the accompanying diagram, the respective sides of the triangle indicate the percentages of phosphoric acid (H_3PO_4), of water (H_2O), and of chromic acid (CrO_3), from 0 to 100%. On the basis of experimental data, I have determined the relative proportions of phosphoric acid, chromic acid and water that give compositions of electrolyte that are operative for the anodic polishing of nickel. The area representing operative compositions of electrolyte is defined on the accompanying diagram by the solid lines ACB and BA. Within the area so defined, any composition selected will be found to be operative in the method hereinafter described for the anodic polishing of nickel.

In order to get the best polishing results, however, I have found that the range of proportions of phosphoric acid, chromic acid and water should be kept within somewhat narrower limits, and these narrower limits are represented on the accompanying diagram by the area defined by the lines AD, dot and dash line DE, and EA. The preferred compositions of electrolyte with respect to the relative proportions of phosphoric acid, chromic acid and water, lie within this lesser area.

The reading of a triaxial diagram such as the accompanying one is well understood, but the following will be given for purposes of illustration.

The point on the diagram represented by the letter A, for instance, indicates the composition comprising 85% of phosphoric acid, a small but significant proportion, say 0.1% of chromic acid and the balance, somewhat less than 15%, water; the point indicated by the reference letter B a composition comprising about 58% of phosphoric acid, about 0.1% of chromic acid and the balance, somewhat less than 42%, of water; the point indicated by the reference letter C a composition comprising 33% of phosphoric acid, 38% chromic acid and 29% of water; and the point indicated by the reference letter D a composition comprising 46% of phosphoric acid, 29% of chromic acid and 25% of water.

It will be seen that the maximum phosphoric acid content is 85% and the minimum 33%; the maximum chromic acid content 38% and the minimum about 0.1%; and the maximum water content about 42% and the minimum 13%. The preferred composition limits are from 46 to 85% phosphoric acid; from 0.1 to 29% chromic acid; and from 13 to 34% water. The percentages of the three ingredients, however, are inter-dependent and should lie within the areas above described for best results.

It should be understood, also, that while the accompanying diagram serves to indicate the relative percentages of phosphoric acid, chromic acid and water in a system consisting only of these three components, an electrolyte suitable for the anodic polishing of nickel may also include other ingredients, such as other acids and/or metallic

salts. Regardless, however, of what other ingredients may be present in the bath, the bath should contain phosphoric acid, chromic acid and water in such relative percentages as to lie within the preferred or less preferred areas defined on the accompanying diagram.

In making up an electrolyte for the anodic polishing of nickel, one might, for instance, select the point X on the accompanying diagram as being a bath within the area of preferred compositions. The composition represented by the point X would be 70% phosphoric acid, 10% chromic acid and 20% water. During the continued use of such a bath in the electropolishing of nickel, the bath composition would necessarily change, owing to the anodic dissolution of nickel into the bath. There might also be some change in the water content, such as an increase due to absorption by the bath of moisture from the air, or a decrease due to evaporation of water from the surface of the bath, to decomposition of water by electrolytic action or to loss of water from the bath by entrainment thereof in gases given off from the bath.

Notwithstanding such changes in composition as may occur during continued use, if the relative percentages of phosphoric acid, chromic acid and water, expressed in percentages by weight of the total weight of only these three ingredients in the bath composition, remain within the area defined by the lines AD, DE and EA, the bath will continue to operate satisfactorily. Even if the bath composition be so altered during continued operation as to fall within the less preferred area defined by the lines DE, EB and BCD, on the accompanying diagram, the bath will continue to function, although not so satisfactorily.

Consequently, where the relative percentages of phosphoric acid, chromic acid and water in a given bath composition lie within either the preferred or less preferred areas defined on the triaxial diagram, such bath composition is intended to come within the scope of my invention, even though it may contain other acids than phosphoric and chromic and even though it may contain a substantial quantity of metallic salts.

Instead of chromic acid, soluble chromates and bichromates may be substituted therefor and are to be considered the equivalent of chromic acid on a stoichiometric basis. The term "chromic acid equivalent," as used in this specification and in the claims, is therefore intended to include chromic acid itself (CrO_3) and stoichiometrically equivalent weights of soluble chromates and bichromates.

Similarly, in place of orthophosphoric acid, other phosphoric acids, such as meta- and pyrophosphoric acids, may be used and are to be considered as included within the term "phosphoric acid."

In the method of anodically polishing nickel, electro-deposited nickel, non-ferrous nickel alloys and the like, using a bath of a composition indicated as suitable by reference to the accompanying triaxial diagram, the metal, or an article made therefrom, is made the anode in a bath of the selected composition and an electric current is passed therethrough of sufficient density and for a sufficient length of time to produce the desired high degree of luster, or polish, on the nickel surface. By employing an electrolyte having a composition within the preferred areas defined on the accompanying triaxial diagram, a highly lustrous, mirror-like surface can be readily obtained. The highly lustrous surface obtainable by my method, using an electrolyte of preferred composition, is an important feature of my invention and one that sharply distinguishes it from prior art finishes produced in the electrolytic cleaning of nickel.

The formation of highly polished and lustrous surfaces is undoubtedly associated with the presence of a polarizing film over the surface of the nickel during the process of anodic dissolution. The nature of this film is such that selective attack on the various phases present in nickel, or non-ferrous nickel alloys, is minimized. Anodic dissolution apparently takes place at a relatively high rate and at a high anodic polarization value, with the result that the anodic dissolution of the metal acts to level the crystal surfaces thereof and to produce a mirror-like finish. These conditions do not prevail in the simple electrolytic cleaning treatment known to the prior art.

In order to obtain the best results in a reasonable length of time, it is preferable to use relatively high current densities, such as those of the order of magnitude of from 100 to 500 amperes per sq. ft. It will be understood, however, that lower current densities, even as low as 50 amperes per sq. ft., may be employed with consequent prolongation of the time of treatment. Higher current densities, up to as high as 2000 amperes per sq. ft., may also be used, but such high current densities imply, in general, larger currents, which require more expensive equipment. The length of time to effect the desired results depends upon the magnitude of the current densities employed and to some extent upon the characteristics of the nickel, its grain size and the like. Rough surfaces, of course, require a longer time to polish than relatively smooth ones.

With any of the compositions of electrolyte lying within the preferred area defined by the lines AE, ED and DA on the accompanying triaxial diagram, excellent polishes are obtained on surfaces of nickel, of electrodeposited nickel, or of non-ferrous nickel alloys, if such surface is made the anode therein at current densities of 125 to 250 amperes per sq. ft. and the treatment carried out for a period of from 4 to 12 minutes at a bath temperature in the neighborhood of 100 to 155° F. In general, the temperature of the bath may be maintained at any point between room temperature and the boiling point of water but temperatures of around 100° F. are found very satisfactory.

From the foregoing description of my invention, it will be apparent that I have provided a novel composition of electrolyte and a method whereby the same may be used to produce highly lustrous polishes on nickel, electrodeposited nickel, non-ferrous nickel alloys and the like. My electrolyte and method avoid the disadvantages of the old methods of mechanical polishing, and enable the production of nickel surfaces that are free from mechanical strain, dragging and "piling," and which are superior to those obtainable by mechanical polishing methods.

It will, of course, be understood that various details of the process may be varied through a wide range without departing from the principles of this invention and it is, therefore, not the purpose to limit the patent granted hereon otherwise than necessitated by the scope of the appended claims.

I claim as my invention:

1. The method of anodically polishing an article having a surface of nickel or of a non-ferrous nickel alloy, which comprises making

said article the anode in a solution containing from 33 to 85% of phosphoric acid, from 0.1 to 38% of CrO_3, and from 13 to 42% of water, the total acid concentration in said solution being from 58 to 87%, all of the percentages being by weight of said solution and the relative proportions of said three solution ingredients lying within the area defined on the accompanying diagram by the line ACB and the straight line BA, and passing therethrough an electric current of sufficient density and for a sufficient duration of time to effect a polish on said surface.

2. The method of anodically polishing an article having a surface of nickel or of a nonferrous nickel alloy, which comprises making said article the anode in a solution containing from 46 to 85% of phosphoric acid, from 0.1 to 29% of CrO_3, and from 13 to 34% of water, the total acid concentration being from 66 to 87%, all of the percentages being by weight of said solution and the relative proportions of said three solution ingredients lying within the area defined on the accompanying diagram by the line AD, the line DE, and the straight line EA, and passing therethrough an electric current of sufficient density and for a sufficient duration of time to effect a polish on said surface.

3. The method of anodically polishing an article having a surface of nickel or of a nonferrous nickel alloy, which comprises making said article the anode in a solution containing from 33 to 85% of phosphoric acid, from 0.1 to 38% of CrO_3, and from 13 to 42% of water, the total acid concentration being from 58 to 87%, all of the percentages being by weight of said solution and the relative proportions of said three solution ingredients lying within the area defined on the accompanying diagram by the line ACB and the straight line BA, and passing therethrough an electric current of between 125 and 250 amperes per square foot at a bath temperature between 100° and 155° F. and for a sufficient duration of time to effect a polish on said surface.

4. The method of anodically polishing an article having a surface of nickel or of a nonferrous nickel alloy, which comprises making said article the anode in a solution containing from 46 to 35% of phosphoric acid, from 0.1 to 29% of CrO_3, and from 13 to 34% of water, the total acid concentration being between 66 and 87% by weight of said solution, all of the percentages being by weight of said solution and the relative proportions of said three solution ingredients lying within the area defined on the accompanying diagram by the line AD, the line DE, and the straight line EA, and passing therethrough an electric current of between 125 and 250 amperes per square foot at a bath temperature of between 100° and 155° F. and for a sufficient duration of time to effect a polish on said surface.

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