

Nov. 23, 1943.

C. L. FAUST

2,334,698

POLISHED METALS AND METHODS OF MAKING THE SAME

Filed July 9, 1938

3 Sheets-Sheet 1

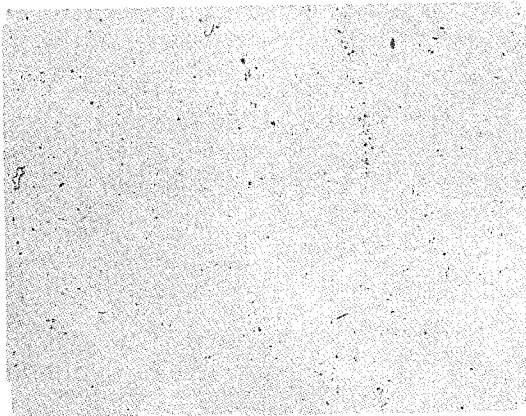


Fig. 1



Fig. 2.

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3 Sheets-Sheet 2

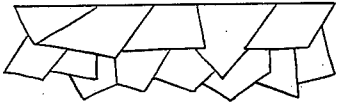


Fig. 7



Fig. 8

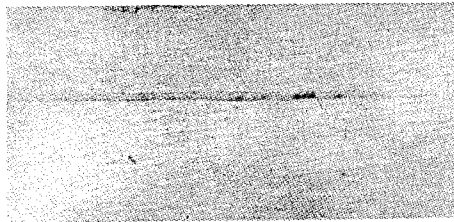


Fig. 3.

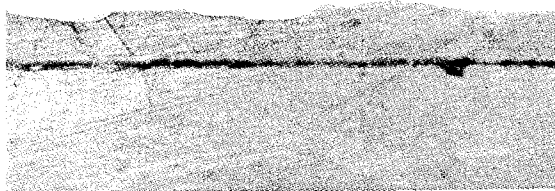


Fig 4.

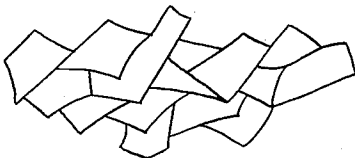


Fig. 9

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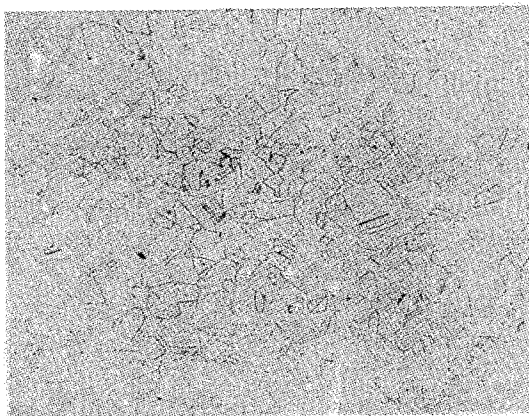


Fig. 5



Fig. 6.

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## UNITED STATES PATENT OFFICE

2,334,698

POLISHED METAL AND A METHOD OF  
MAKING THE SAME

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Application July 9, 1938, Serial No. 218,388

6 Claims. (Cl. 204-140)

My invention relates to polished metals and a method of making the same. It is particularly applicable to stainless steel, although it is not necessarily limited thereto and may be applicable to various other metals.

Metals are most commonly polished by mechanical rubbing and buffing and this practice has many drawbacks. For example, the mechanical polishing of stainless steel is one of the most expensive steps in its manufacture. As a result, polished stainless steel has hitherto sold for a price which is very high, when the cost of materials alone is considered. There are certain difficulties in rolling and other steps of manufacture that account, in part, for this high cost but, in the main and particularly with certain types of objects, the mechanical polishing is one of the largest items in the total cost.

In the prior art, stainless steel has been polished mechanically only and the operation has not only been expensive but it has been difficult because of the properties of the material. For instance, stainless steel is a poor conductor of heat and, in consequence, relatively high speeds of polishing tend to burn the surface thereof. This greatly limits the speed of polishing. Furthermore, it is hard and tough and has a tendency to "pile" so that the surface produced is cold worked and strained. Likewise, commercially mechanically polished stainless steel is marked by microscopic scratches.

Manufacturers of stainless steel have wished vainly for other methods of polishing. Polishing by chemical agents has not been possible heretofore. As a matter of fact, chemical attack by the more powerful chemical reagents serves only to etch the surface and to develop a matte appearance of a type that is useless, so far as the demands of the trade for a polished surface is concerned.

Some effort has been made to clean ferrous alloys and other metals by electrolytic treatment. However, these efforts have been directed to cleaning only and definitely do not produce a polished surface. Such surfaces are sometimes described as etched, but it will be obvious that an etched surface is not a polished one.

For example, one prior art patentee deals with an electrolytic process for cleaning ferrous metals in preparation for nickel plating thereon and states that he obtains a product which is uniformly etched. But a polished surface of the type with which I am concerned would not normally be covered up with nickel plating or other plating. Moreover, the type of polish with which I am concerned is, in the main, antithetical to an etched surface.

Other prior art patentees have suggested the electrolytic treatment of such metals as stainless steel for the purpose of removing or loosening

scale preparatory to the subsequent treatment of the metal. But these patentees do not suggest that a polished or lustrous surface can be produced upon such materials by electrolytic treatment.

One object of my invention is to provide an effective method of imparting such a high luster to a metal or metal alloy that no further treatment will be necessary to render it highly ornamental and pleasing to the eye.

Another object of my invention is to provide a method of treating stainless steel which will produce thereon a surface which will be highly lustrous and free from the scratches and "piled" layers characteristic of mechanically polished surfaces of stainless steels.

Another object of this invention is to provide a method for polishing metals which will be less expensive than the mechanical polishing thereof while, at the same time, producing surfaces thereon having relatively superior characteristics.

Another object of this invention is to provide a metal having a surface thereon which is highly lustrous.

Another object of this invention is to provide a stainless steel having a surface thereon which is highly lustrous.

Other objects of my invention will appear as this description progresses and from the appended claims.

This application is a continuation in part of my application, Serial No. 160,890, filed August 25, 1937.

Generally stated, my method consists of treating the metal in an electrolyte bath of suitable composition by making it an anode and passing a current of sufficient density and for a sufficient length of time to produce a high luster or polish on the metal. I have found that with my method, I can produce a lustrous surface that is, at least, equal in appearance to surfaces obtainable by mechanical polishing and that is superior in other characteristics, such as freedom from "piling" and scratches characteristic of mechanically polished surfaces, so that all necessity for mechanical polishing is obviated while, at the same time, superior results are obtained. As a matter of fact, my method permits me to obtain almost mirror-like surfaces that are very much superior to the results obtainable by ordinary mechanical polishing. The results which I obtain transcend a mere electrolytic cleaning process in that there is produced a highly lustrous or polished surface. This high polish and luster is an important feature of my invention and sharply distinguishes it from prior art methods used in the electrolytic cleaning of stainless steel.

More specifically, I have discovered a method by which the surfaces of the grains of the metal being treated may be rendered substantially level

and that this results in a highly lustrous surface. Thus, the treatment of stainless steel by the method which I shall now describe more in detail results in a surface free from any amorphous layer and of extremely high luster.

In carrying out my process, I provide a suitable electrolyte and make the stainless steel or other metal to be cleaned the anode of the cell. The cathode may be made of any inert conducting material. The electrolyte may be widely varied in composition, although not all electrolytes are suitable. A great many substances may be used to produce the result. However, certain electrolytes are much more desirable than others and some of these electrolytes will be enumerated hereinafter.

I prefer to use sulfuric and phosphoric acids in substantial amounts in this electrolyte mainly because of their low cost. Sulfuric acid may, however, be used without phosphoric acid in conjunction with any one of a wide variety of organic materials such as glycerol, some alcohols, ethylene glycol, ammoniated glycyrrhizin, glycyrrhiza extract or licorice, thiourea and its substitution products. These, when combined with sulfuric acid in suitable concentration, produce electrolytes which are effective in producing lustrous surfaces. Most of the materials I have found useful when added to sulfuric acid belong to the class of chemical compounds which are known to form complex ions with such ions as iron, chromium, nickel, etc.

In choosing materials for the electrolytic bath, I have discovered certain principles that offer some guidance in the selection. The formation of the highly polished and lustrous surface that is a distinguishing feature of my invention is undoubtedly associated with the presence of a polarizing film over the surface of the metal during the process of anodic attack. The nature of this film is such that selective attack of the various phases present in the alloy is minimized. Anodic dissolution apparently takes place at a relatively high rate at high anodic polarization values. The result of the anodic solution of the metal under these conditions is to level the crystal surfaces of the metal and to produce a smooth and mirror-like finish. These conditions do not prevail in the simple electrolytic cleaning treatments known to the prior art.

I have found that it is preferable to keep the water content of the electrolytic bath relatively low. While I have been able to produce polishes on stainless steel with mixtures of sulfuric and phosphoric acids containing as much as 50 per cent water I find that, in general, baths containing less water will polish at lower current densities.

The mixtures of sulfuric and phosphoric acids which are suitable for carrying out my process cover a wide range of compositions. I prefer to have the total acid in excess of 50 per cent and not over 90 per cent, the balance in any case being water or organic addition materials such as I have enumerated previously. Sulfuric acid when supplemented by phosphoric acid should be in excess of 5 per cent to produce the best results; phosphoric acid is not essential to my process, but its use permits polishing at lower current densities and I prefer to use it in excess of 50 per cent of the total bath composition.

In order to obtain the results I desire in a reasonable length of time I prefer to use relatively high current densities. With the preferred mixtures of sulfuric and phosphoric acids the proc-

ess may be operated with anode current densities of from 50 to 1000 amperes per square foot, though it will be understood that lower current densities may be used, in which case a longer time will be required. Higher current densities than this range may also be used though such high current densities are necessarily costly.

The length of time to effect the desired results will depend on the current density used and to some extent on the nature of the initial surface. Rough surfaces will obviously require a longer time to polish than relatively smooth ones. The treatment will also depend to some degree on the previous heat treatment of the steel. The condition of the carbon in the steel affects the attack upon the grain boundaries and in some cases it is necessary to increase the current density.

As examples of electrolytic baths and the conditions under which they produce good polishes on stainless steel, I cite the following:

#### Bath No. 1

	Parts of weight
Sulfuric acid.....	15
Phosphoric acid.....	63
Water .....	22

With this bath excellent polishes are obtained on 18-8 (Cr-Ni), 25-12 (Cr-Ni) and 12 to 18 per cent Cr and other straight chromium stainless steels when made the anode therein at current densities of 50 amperes per square foot for one hour. At higher current densities proportionally less time will be required.

#### Bath No. 2

	Parts of weight
Sulfuric acid.....	60
Water .....	40
Ammoniated glycyrrhizin.....	0.01
Methanol .....	0.55

This bath is particularly adapted to polishing of 18-8 type (Cr-Ni) stainless steel when it is made the anode therein at current densities of 2000 per square foot for 1½ to 2 minutes. This time suffices to produce a very brilliant surface at a bath temperature of 80 to 175° F.

#### Bath No. 3

	Parts of weight
Sulfuric acid.....	50
Glycerol .....	40
Water .....	10

This bath when used with current densities of 300 to 1000 amperes per square foot for the anodic treatment of 18-8 (Cr-Ni), 24-12 (Cr-Ni) and chromium stainless steel develops a very brilliant surface. The time required at temperatures of 90 to 200° F. ranges from 3 to 9 minutes, depending somewhat upon the current densities used, and the original state of the surface.

#### Bath No. 4

	Parts of weight
Sulfuric acid.....	16
Phosphoric acid.....	13
Glycerol .....	56
Water .....	15

This bath is very satisfactory for polishing 18 per cent Cr-type stainless steel at current densities of around 1000 amperes per square foot.

#### Bath No. 5

	Parts of weight
Sulfuric acid.....	60
Phosphoric acid.....	30
Water .....	10

In a bath of this composition excellent polishes were obtained on 18-8 (Cr-Ni) and 24-12 (Cr-Ni) stainless steel at current densities of 30 amperes or more per square foot.

It will be understood that the relative amounts of materials in the baths cited as examples may be varied considerably.

With my process I am able to produce stainless steel having a surface which is different from that produced by prior art methods of polishing this material. All polished stainless steel articles hitherto available commercially show microscopic scratches on their surfaces which are produced by the mechanical process of buffing and polishing. The surfaces produced by mechanical polishing are characterized further by the presence of a layer of material which has been altered by the polishing process. Electron diffraction studies which I have made on 18-8 (Cr-Ni) indicate this to be a surface of ferritic material produced, presumably, by the mechanical working of the surface by polishing. Surfaces produced by my method on the contrary are free from the scratches and altered layer which characterize these mechanically polished surfaces.

Surfaces produced by my method are also very different and greatly superior to those produced by any electrolytic cleaning or pickling treatment hitherto used. The electrolytic treatments of stainless steel by the methods of the prior art develop matte surfaces which, when examined microscopically, show an attack of the crystal boundaries and a strong development of the crystalline structure of the metal. The surfaces resulting from my method show no trace of the crystalline structure when examined microscopically or at most a faint suggestion of this structure. This feature of my method which results in a suppression of the development of the crystal structure also helps to produce the high polish which I obtain.

Still further evidence of the novelty of surfaces produced by my method is found in metallographic studies which I have made of stainless steels treated by my process and by ordinary methods of mechanical polishing. These studies are exemplified in the accompanying photolithographs wherein:

Figure 1 shows the result of nickel plating a mechanically polished surface of stainless steel, magnification 100 diameters.

Figure 2 shows the result of nickel plating a stainless steel surface polished by my method, magnification 100 diameters.

Figure 3 shows a cross section through a nickel plate on surfaces of stainless steel which have been polished mechanically, magnification 500 diameters.

Figure 4 shows a cross section through a nickel plate on surfaces polished by my method, magnification 500 diameters.

Figure 5 shows the etching action of glycer-regia on the surface of mechanically polished stainless steel, magnification 100 diameters.

Figure 6 shows the etching action of glycer-regia on the surface of stainless steel polished by my method, magnification 100 diameters.

Figure 7 is a diagrammatic sketch illustrating the type of surface which is obtained by electrolytically polishing stainless steel in accordance with my method.

Figure 8 is a diagrammatic sketch illustrating the type of surface obtained by mechanically polishing stainless steel and further illustrating

the so-called amorphous layer on said surface which results from mechanical polishing.

Figure 9 is a diagrammatic sketch illustrating the type of surface obtained by etching stainless steel.

Figures 1, 2, 3 and 4 collectively show the difference between the surfaces produced by my method and by methods of mechanical polishing. Figure 1 shows that nickel plated on to a metallographically polished specimen of stainless steel shows no structure at ordinary magnifications. This is what would be expected from the nature of the stainless steel surface produced by mechanical polishing. Such a surface will be characterized by the presence of a layer of amorphous or pseudo amorphous material produced by cold working the surface, and nickel, plated on to a layer of this nature, would not be expected to show a structure at ordinary magnifications, as is actually found to be the case. I refer to the layer of worked material on the mechanically polished stainless steel as amorphous without meaning to imply that such is its actual state since I am aware that much controversy has been waged over the actual nature of this layer of worked material.

Surfaces produced by my method are free from this amorphous layer. The polish produced by my process is characterized by the presence of level surfaces of the crystals forming the stainless steel. Since the lattice constants of nickel are approximately those of austenite, isomorphism is possible and nickel plated on to such a surface would be expected to deposit in such a way as to continue the crystalline structure already present and to make it more apparent. That this actually occurs is shown by Figures 2 and 4. Figure 2 shows the result of plating nickel on 18-8 stainless steel polished by my method. The crystal structure present on the stainless steel surface is sharply brought out by such a plate.

That the structure exhibited by the nickel plate is actually the crystal structure of the underlying stainless steel is shown by Figure 4. This figure is a cross section through the nickel plate and the stainless steel. The continuity of crystal growth from the surface to the plate is clearly shown in this photomicrograph. It will be noted that such continuity of crystal growth is not shown in Figure 3, where the nickel has deposited on the amorphous layer produced by mechanical polishing.

Not only is the metallic surface produced by my method different from that resulting from mechanically polishing stainless steel, but the film covering the surfaces is also different. This is proven by Figures 5 and 6 which show the difference in behavior of the two surfaces of 18-8 Cr-Ni stainless steel when subjected to an etching treatment with glycer-regia. Mechanically polished surfaces are subject to attack which immediately develops the crystal structure of the material, as shown in Figure 5. Specimens polished by my method on the other hand are apparently covered by a film which permits attack only at discrete points which results in a pin hole structure, as shown in Figure 6. Substantially similar results are obtained with straight chromium stainless steels.

Referring to Figures 7, 8 and 9, which are sketches showing in exaggerated form the grain structures of stainless steel and the types of surfaces produced thereon by my method, by mechanical polishing and by etching, it will be noted

that the surface illustrated in Figure 7 which results from electrolytic polishing by my method is one wherein the surfaces of the grains are substantially level and are not covered by any layer of amorphous material. In Figure 8, it will be noted that on mechanically polished stainless steel the polished surface is a layer of amorphous material. In Figure 9, it will be noted that the etched surface is made up of grains whose surfaces are not level. This condition is characteristic of the etched surfaces such as those produced by electrolytic cleaning.

From this description of my invention it will be apparent that I have provided a novel and effective way of producing a polish on stainless steel. My method avoids all the disadvantages of the old method of mechanical polishing and allows me to produce stainless steel articles with surfaces which are free from mechanical strain, dragging, and piling, and which are better than those made by mechanical polishing methods.

By the term "stainless steel" I mean those steels which are made corrosion-resistant by the addition of such alloying elements as chromium or chromium and nickel and which are exemplified by the examples I have given.

Having thus described my invention, what I claim is:

1. The method of imparting a lustrous surface to stainless steel which comprises making such stainless steel the anode in an electrolytic bath of substantially the composition: sulfuric acid, 15 per cent; phosphoric acid, 63 per cent; water, 22 per cent, and passing a current therethrough at an anode current density in excess of 50 amperes per square foot for a period of time sufficient to impart the desired luster.

2. The method of imparting a lustrous surface to stainless steel which comprises making such stainless steel the anode in a solution of 5 to 85 per cent sulfuric acid, 5 to 85 per cent phosphoric acid, the combined acid concentration being between 50 and 90 per cent and the balance being substantially water, and passing a current of at

least 30 amperes per square foot anode current density therethrough for a period of time sufficient to impart the desired luster to said surface.

3. The method of imparting a lustrous surface to stainless steel which comprises making such stainless steel the anode in an aqueous electrolyte containing at least 5 per cent of sulfuric acid and at least 50 per cent of phosphoric acid, the total acid concentration being not over 90% and the balance being largely water, and passing an electric current therethrough of sufficient density and for a sufficient period of time to effect such lustrous surface.

4. The method of imparting a lustrous surface to stainless steel, which comprises making such stainless steel the anode in an aqueous electrolyte containing at least 5% of sulfuric acid and at least 50% of phosphoric acid, the total acid concentration being not over 90% by weight and the balance being largely water, and passing an electric current therethrough of a density of from 30 to 2000 amps./sq. ft. for from 1½ minutes to 1 hour, the time being shorter for the higher current densities and longer for the lower current densities while maintaining the electrolyte at a temperature between 80 and 175° F.

5. A new article of manufacture having a stainless steel surface, said surface being anodically polished to remove metal therefrom and render said surface free from mechanical strain and from amorphous layers and showing substantially no crystalline structure microscopically.

6. The method of imparting a lustrous surface to stainless steel, which comprises making such steel the anode in an aqueous acid electrolyte consisting initially of about eleven per cent water by weight, sulfuric acid, and phosphoric acid, each of said acids being present in substantial effective amounts, and passing an electric current therethrough of sufficient density and for a sufficient period of time to obtain such lustrous surface.

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