252. COMPOSITIONS) Cross Reference Examiner

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METHOD OF PICKLING CHROMIUM-IRON ALLOYS.

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This invention relates to chromium iron alloys and more particularly to a method of pickling or removing the scale from such alloys.

The chromium iron alloys to which this invention is particularly directed are those known as "rustless" or "stainless" iron and "stainless" steel. In such alloys, the chromium content may vary between 8% and 60% and the carbon content may vary depending upon the particular use to which articles made therefrom are to be put. The alloy may contain silicon and other metalloids.

In one alloy with which I have had experience, the chromium content is approximately 12% and the carbon content is approximately .12%. The scale produced on such alloy when the same is either box annealed or open annealed is extremely difficult to remove. The pickling solutions most commonly used contain sulphuric acid or hydrochloric acid and certain brightening agents and these not only will not remove the scale from these alloys but they have a tendency to dissolve the alloy itself in preference to the scale.

An object of this invention is to produce a method of pickling or removing the scale from such iron chromium alloys without detrimentally etching or otherwise attacking the alloy itself.

I have discovered that if hydrofluoric acid is used as a preliminary pickle and then after the alloy has been rinsed is followed by a nitric acid pickle the scale will be removed. I have discovered, however, that if hydrofluoric acid is used alone as the preliminary pickle, it has a tendency to over-etch or pit the alloy itself. The hydrofluoric acid apparently activates the scale or puts it in a state where it can and will be acted upon by the nitric acid pickle. After many trials and experiments, I have discovered that the scale can be properly activated without over-etching the alloy itself, if a metal or a metal compound that is positive to both iron and chromium is added to the hydrofluoric acid pickle. Potassium, sodium, calcium, aluminum and zinc are examples of such metals.

As a cheap metal that is positive to both iron and chromium and that satisfactorily prevents over-etching of the hydrofluoric acid, I prefer to use commercial nitre cake. The preliminary pickle which I have found satisfactory contains from 1% to 6% hydrofluoric acid and from ½% to 3% nitre cake. The solution is preferably used cold and the alloy is immersed therein for a length of time which varies according to the heat treating method to which the alloy or the articles made therefrom have been subjected.

A hot rolled sheet can be pickled in a short time, while a hot rolled open annealed sheet takes somewhat longer, and a hot rolled box annealed sheet takes still longer. A hot rolled box annealed sheet requires about three hours in a cold solution. After the preliminary pickling and after the alloy or articles made therefrom have been rinsed, about 15 to 20 minutes pickling in a warm nitric acid solution of from 2% to concentrated will be sufficient to remove the activated scale.

The preliminary pickling can be shortened if the solution is somewhat warmed, but I do not advise a temperature of over 140° F. If the percentage of nitre cake in the preliminary pickle is reduced, the temperature of the solution may be somewhat raised.

The nitre cake contains about 28% of sulphuric acid but its high positive sodium content tends to prevent this from dissociating thereby preventing overpickling or over-etching by this sulphuric acid content.

When metals that are positive to iron and chromium are added to the hydrofluoric acid pickle, fluorides of these metals are formed and instead of adding such metals to the pickle, it is possible to add fluorides of the metals above mentioned.

An over addition of chlorides must be avoided in order to prevent a too violent action of the pickle.

If it is desired, a combination of sulphides and chlorides of metals that are positive to iron and chromium may be added to the hydrofluoric acid pickle.

The preliminary or scale activating pickle must always be followed by a nitric acid pickle. The theory that I have proceeded upon is that if the scale is activated, it is made soluble in nitric acid and I believe that in the step of activating the scale, it is converted into a chromium-iron-fluoride which is readily soluble in nitric acid.

Having thus described my invention, what I claim is:—

1. The method of removing the scale from chromium iron alloys, which consists in first activating the scale on such alloys by immersing the same in a solution containing hydrofluoric acid and a metal or salt thereof that is positive to iron and chromium and then in dissolving the scale in a solution containing nitric acid.

2. The method of removing the scale from chromium iron alloys, which consists in activating the scale thereon in a solution containing hydrofluoric acid and the salt of a metal positive to iron and chromium and then in dissolving the scale in a solution containing nitric acid.

In testimony whereof, I have hereunto subscribed my name this 26th day of May, 1926.

LOUIS SCHULTE.