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PROCESS FOR THE PROTECTIVE TREATMENT OF IRON

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This invention relates to a process for the protective treatment of iron and has particular relation to a process of this type in which metallic aluminum is applied to and alloys with the iron surface to be protected.

The main object of the present invention is to provide an improved process for the protective treatment of iron by the application of aluminum chloride in the vapor phase to the surface to be protected in order to obtain a more efficient and durable protection of the iron, than obtainable by the hitherto known processes using aluminum.

Another object of my invention is to provide an anhydrous gaseous mixture, in which vaporized aluminum chloride is present in intimate mixture with hydrogen and which is particularly adapted to react quickly with the iron and to produce an improved corrosion-resistant surface layer.

A further object of my invention consists in providing a process for the protective treatment of iron with a mixture of aluminum and copper.

It is also an object of my invention to produce from silicon tetrachloride and suitable organic compounds completely dry gaseous HCl which can be used with advantage in the production of a metal chloride-hydrogen mixture needed in carrying out my invention.

Other objects and the advantages of the invention will be apparent from the appended claims and the following specification, which illustrate by way of example some embodiments of my invention.

It has been previously suggested to apply certain compounds of aluminum, particularly AlF, to the surface of iron in order to obtain a corrosion resistant surface coating comprising aluminum and iron. However, it was not hitherto possible to isolate a compound of the formula AlF and it is doubtful whether such a compound can be prepared at all.

It has also been suggested to place iron articles to be treated in a drum or the like with a mixture of powdered aluminum, aluminum oxide and ammonium chloride, to rotate the drum and heat it in a hydrogen atmosphere in order to provide the articles with a corrosion resistant surface. It has been found that this process does not give satisfactory results.

I have now found that the application of metallic aluminum to iron can be carried out in a satisfactory manner and yields products having a surface of high resistance to corrosion particularly at elevated temperatures by proceeding in the manner described hereinafter.

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According to my present invention, the iron surface to be protected is treated with a gaseous mixture which contains vaporized $AlCl_3$ and hydrogen and has been obtained by reacting metallic aluminum with completely dry HCl gas in the manner described further below. In carrying out this procedure, the $AlCl_3$ is used in dry condition and is applied to the iron in the absence of water or moisture in an atmosphere of hydrogen. Both conditions must be observed, as the absence of water, steam or moisture as well as the presence of a hydrogen atmosphere are essential.

In carrying out the process of my invention, dry gaseous HCl gas is caused to pass at a temperature between 300° and 600° C. over a mixture of aluminum, which may be present for example in the form of shavings or grit, with aluminum oxide (Al_2O_3) and small coal. The aluminum oxide may be used in an amount of 10% by weight and the coal likewise in an amount of 10% by weight based on the amount of aluminum. The aluminum is subjected to the action of HCl gas in mixture with aluminum oxide and coal in order to prevent sticking together of the metal particles. Moreover, the coal prevents oxidation and combines with water vapor if the latter is present. No water is formed by a reaction between Al_2O_3 and HCl in the above mentioned temperature range of 300° to 600° C., as such reaction does not set in below 800° C. The HCl gas reacts with the metallic aluminum according to the equation

$$2Al+6HCl=2AlCl_3+3H_2$$

and this reaction provides for the presence of the necessary hydrogen in my process. As the aluminum is present in great excess in the reaction, it is possible that sub-chlorides of the formula AlCl and/or $AlCl_2$ are also formed to some extent.

I have found that the anhydrous gaseous mixture obtained in the above described manner and consisting of an anhydrous intimate mixture of vaporized aluminum chloride and hydrogen, can be used with excellent results in the production of a protective layer on iron. In order to obtain such layer, said gaseous mixture is passed over iron heated to at least 700° C. and preferably 900°–1000° C. or higher, up to 1200° C. Upon bringing the gaseous mixture in contact with the heated iron, the aluminum chloride is split into Al and Cl, the aluminum forming an alloy with the surface layer of the iron, while the Cl is converted into iron chloride. The thickness of the

alloy layer formed depends upon the temperature and duration of the treatment of the iron with the gaseous stream of aluminum chloride and hydrogen.

I have also found that the protective effect of my process can be increased by using the aluminum in combination with copper. This can be done for example by using a mixture of aluminum shavings and copper shavings or by using an alloy of aluminum and copper in the reaction with dry gaseous HCl. In this case a gaseous mixture containing hydrogen, aluminum chloride and copper chloride is formed and in the treatment of the heated iron with such mixture in the above described manner, in addition to aluminum, copper is also deposited in the iron and increases the resistance of the iron to corrosion. I use for example a gaseous mixture containing 2% by weight of copper based on the weight of Al in the gaseous mixture, and found that admixtures of copper to Al varying between 1 and 5% give good results.

The duration and temperature of the treatment according to my invention depends on the requirements of resistance of the iron to corrosion. I have found that treatments with the gaseous $AlCl_3$-containing mixture at 900° to 1000° C. during 10 to 60 minutes give satisfactory results. In order to decrease the brittleness of protective layers in iron treated according to the process of my invention, the latter may be subjected to a thermal after-treatment at about 1000° C. in an atmosphere of hydrogen or air. Such treatment causes the aluminum to diffuse deeper into the iron so that concentration of aluminum in the top layer of iron decreases and said layer becomes softer.

I have found that a completely dry stream of HCl gas can be obtained with advantage by reacting silicon tetrachloride with an alkanol, for example ethyl alcohol, methyl alcohol, propyl alcohol, butyl alcohol, whereby a completely dry stream of HCl gas and a compound for example of the formula $Si(OC_2H_5)_4$ is obtained which can be used in a manner known by itself for the preparation of silicone resins. It will be understood, however, that HCl gas prepared in any other suitable manner may also be used in carrying out my invention, provided that it is in a substantially anhydrous condition.

It is essential in carrying out my invention that the iron is treated with a gaseous mixture obtained by the reaction of metallic aluminum and dry HCl gas.

It will be understood by those skilled in the art that my invention is not limited to the specific steps, conditions and materials described above and may be carried out with various modifications. For example, instead of reacting the aluminum with HCl in the form of shavings or grit, it can be used in the form of powder or other suitable form and in combination with admixtures other than aluminum oxide and/or coal. The temperature and duration of the treatment of iron with the mixture of vaporized $AlCl_3$ and hydrogen may be varied and the amount of copper used in combination with aluminum may be varied within the limits stated above. These and other modifications may be made without departing from the scope of the invention as defined in the appended claims.

What is claimed is:

1. A process for the protective treatment of iron, comprising causing iron to react at 700° to 1000° C. with an anhydrous gaseous medium consisting of hydrogen and aluminum chloride and obtained by the reaction, at 300° to 600° C., of anhydrous HCl gas and a mixture of aluminum, aluminum oxide, and coal, until a coating of aluminum is deposited on the surface of the iron treated.

2. A process for the protective treatment of iron, comprising causing the iron to react at 700° to 1000° C. with an anhydrous gaseous medium consisting of hydrogen and aluminum chloride and obtained by the reaction, at 300° to 600° C., of anhydrous HCl gas and a mixture of metallic aluminum and copper, aluminum oxide, and coal until a coating is deposited on the surface of the iron treated.

3. A process for the protective treatment of iron, comprising causing iron to react at 700° to 1000° C. with an anhydrous gaseous medium consisting of the reaction product, formed at 300° to 600° C. of anhydrous HCl gas and aluminum and copper, in mixture with aluminum oxide and coal, until a coating is deposited on the surface of the iron treated.

4. A process for the protective treatment of iron, in which the product obtained according to claim 1, is subsequently subjected to heating at about 1000° C.

5. A process for the protective treatment of iron, as claimed in claim 1, in which anhydrous HCl gas obtained by reacting $SiCl_4$ with an alcohol, is used.

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