

# UNITED STATES PATENT OFFICE.

AUGUSTE J. ROSSI, OF NEW YORK, N. Y., ASSIGNOR OF ONE-HALF TO JAMES MACNAUGHTON, OF SAME PLACE.

## PROCESS OF PRODUCING ALLOYS OF IRON AND TITANIUM.

SPECIFICATION forming part of Letters Patent No. 648,439, dated May 1, 1900.

Application filed June 20, 1899. Serial No. 721,207. (No specimens.)

*To all whom it may concern:*

Be it known that I, AUGUSTE J. ROSSI, a citizen of the United States, residing in the borough of Manhattan, city, county, and State of New York, have invented certain new and useful Improvements in Processes for Producing Alloys of Iron and Titanium, of which the following is a specification.

The object of my invention is to produce such an alloy containing very much less carbon than heretofore and also by a process more rapid and economical than those now used.

My invention, among other things, consists in utilizing in the production of said alloy a bath consisting in whole or in part of a molten metal whose heat of formation of its own oxid is greater than that of the oxid of titanium at like temperatures and in the other features of novelty, which will be hereinafter described.

Letters Patent of the United States No. 609,466 were granted to me on the 23d day of August, 1898, for an alloy of iron and titanium and process of producing it. In the practice of that process I utilized carbon subjected to an intense heat—say not less than 3,500° Fahrenheit—as the principal agent for reducing the oxygen of the titanic acid while in the presence of and supported by a bath of molten iron. While that process has proved eminently useful and satisfactory, occasions have arisen in which it is desirable to produce the aforesaid alloy of iron and titanium freer from carbon and also at an expenditure of heat smaller than was in many cases possible by the use of my previous process. My present invention subserves the aforesaid special requirements and depends in a measure upon the fact that the heat of a given metal's combination with oxygen varies at different temperatures, diminishing generally as the latter rise, and that while the heats of such combination of formation in the case of two given metals may be identical at a certain very high temperature such heats vary absolutely and particularly relatively to each other as the temperatures vary, so that at certain lower temperatures said heat of oxid formation of the one metal used in my process as a reducing agent may be enormously greater than the

corresponding heat at the same temperature of another similarly-used metal or substance—as, for instance, assuming that the temperatures of oxid formation of carbon and aluminium per one atomic weight of oxygen are substantially identical at the temperature of 2,000° centigrade, or thereabout, at the reduced temperature of, say, 0° centigrade to 500° centigrade the heat of formation of alumina is substantially one hundred and thirty thousand calories and that of carbon only twenty-eight thousand calories, while at temperatures above 2,000° the heat of formation of carbon monoxid becomes greater than that of alumina, and carbon begins to decompose alumina. From this it follows that certain metals may be employed with great advantage, under conditions hereinafter prescribed, as reducing agents for such a refractory oxid as that of titanium, and my experiments and tests have demonstrated such to be the fact. I am aware that aluminium is a very powerful reducing agent and has long been known as such and that it has been heretofore used to a limited extent for the reduction of metallic oxids; but according to my information the aluminium and oxids involved have in such cases been preliminarily reduced to a state of impalpable powder and intimately mixed or subjected to the action of additional reagents—as, for instance, fluorids of the alkaline metals and aluminium. In this manner under ordinary temperature one point of the mixture has been raised to a temperature sufficiently high to commence the desired reaction, whereupon the latter is then continued exothermally until complete—that is, without the assistance of any external heat; but the expense of the aforesaid previous preparation of the aluminium has proved prohibitory of industrial utilization of such reaction at least in many fields, among them conspicuously that of ferrotitanium production.

The process of producing ferrotitanium involved in my present invention is as follows: I provide in any convenient way for the support of the titanic acid and iron involved a bath consisting in whole or in part of a liquefied metal, the heat of the formation of the oxid of which is at a certain high temperature, presumably greater than that of the formation of

oxid of titanium—for instance, preferably aluminium—that is to say, in a crucible or other convenient supporting vessel I prepare a bath, preferably, so far as my present observation extends, of molten aluminium by utilizing in any convenient manner temperatures sufficient to melt the aluminium. I then further introduce into the crucible such proportion of iron as will be required to form an alloy of the composition desired, and this iron I also melt, so that it shall become a component of my bath. I next introduce into the bath titanic acid—as, for instance, rutile or iron ore containing titanic acid or a mixture of both—and I then raise the temperature to a point sufficiently high to insure the commencement of the requisite reaction, in which the refractory titanic acid parts with its oxygen and the latter combines with the aluminium to form alumina, and I continue the operation until the reaction is complete, whereby will be produced in the crucible a button or other mass of alloyed iron and titanium—ferrotitanium.

As will be readily appreciated by those skilled in metallurgical operations, the preparation of the ingredients charged into the crucible will vary according to the circumstances. For instance, if titaniferous iron ore is used it is possible that this will contain in itself a sufficient supply of iron oxid to furnish the proportion of iron required in the resultant ferrotitanium, or the ore may be relied upon to furnish a part of such iron and the requisite remainder be charged into the crucible in the metallic state. My experience, however, leads me to the conclusion that in all cases it is preferable that a part at least of the supporting-bath of molten metal should consist of molten metallic iron, as this facilitates the reaction. As carbon is not relied upon as a reducing agent in this process, it will be observed that it may be substantially eliminated from the charge and therefore from the resultant desired product. It will also be remarked that in the case of aluminium owing to the great heat of formation of alumina the external heat applied for the purpose of liquefying the metals and setting up the reaction may be largely diminished, if not wholly discontinued, after the exothermic action has commenced.

As regards temperature, the requirements are the application of an external initial heat sufficiently high to set up the required reaction. While the exothermic action is occurring, and consequently high temperatures generated thereby, the amount of external heat may, if preferable, be diminished. Upon the termination of the exothermic action external heat should be again raised to such a temperature as to insure the fluidity and homogeneity of the resulting alloy. To this end I have found it convenient and economical to use in the application of my said process an electric furnace or electricity as the source of heat, since by this means the heat may be

readily controlled during the various stages of the operation aforesaid, and it will be observed that the amperage required for the application of my present process will fall materially below that requisite in my former process, in which carbon exclusively was used as the reducing agent. In the following instances of the application of my process I obtained the required heat by the use of an electric current of moderate amperage and intensity. The ingredients used were supported in a graphite crucible constituting one of the electrodes, the other electrode being brought into operative relation thereto, so as to insure the formation of an arc between them. It will be observed from the results of the following applications of my process that an alloy comparatively low in titanium was secured owing to the fact that the current available at the time was of such low amperage as to be insufficient to secure the homogeneity required in the alloy, and I therefore approximately increased the proportion of molten iron in the bath for the purpose of facilitating the fusibility of the alloy at a consequently lower temperature. It will also be observed that other metals, such as zinc, the heat of formation of whose oxides proves at certain temperatures to be greater than that of titanic acid may be substituted in whole or in part for the aluminium, though I prefer the latter. Furthermore, in cases where the exclusion of carbon from the resulting alloy is not important carbon may be used with advantage as an element of the charge and as contributing to the reduction of the iron oxides in the ores employed.

It will be observed that I do not confine myself to any particular formula or preparation of ingredients or otherwise in the application of my invention. Such details will be furnished by the experience of those skilled in metallurgical processes and will vary according to the requirements of the various ores treated and the required proportions of the various metals in the resulting alloys. I will now, however, for illustration, mention the details of a few actual examples of my application of my said process.

I. On this occasion I proportioned the materials of my charge as follows: titaniferous mixture, two hundred and seventy-five parts; aluminium, one hundred parts, (reducing agent;) iron-bath, fifty parts. Here, as in II and III following, the titaniferous mixture contained about fifty-six per cent.  $\text{TiO}_2$  and forty-four per cent. of oxid of iron, with but a little silicon and earthy bases. The aluminium was charged into the crucible and melted and then the iron added and also melted. The bath being well liquefied and the heat well up, the powdered mixture, agglomerated to the shape of bricks, cubes, or cylinders, was then added. It melted and gradually disappeared in the bath with incandescence. The intensity of the current was then somewhat moderated until all the mixture

had melted in the bath. The temperature was then raised for a short time sufficiently to render the alloy fluid and homogeneous. The ferrotitanium contained 40.62 per cent. of titanium, with but traces of carbon and a few hundredths of silicon. In this instance no carbon was added and aluminium was exclusively used as a reducing agent.

II. In this case, operating in the same manner as above, I used carbon as a part of the reducing agent for the oxid of iron of the mixture. The proportions were: titaniferous mixture, (same as above in I,) two hundred and seventy-five parts; reducing agent, (carbon thirty-five to thirty-seven per cent. or aluminium sixty to sixty-three per cent.,) ninety-five parts; iron-bath, one hundred and twenty-five parts. The alloy contained 29.46 per cent. titanium and traces of carbon. The carbon in the condition of fine powder was mixed and agglomerated with the powdered titaniferous mixture.

III. In this instance, operating as in I and II, the amount of carbon was increased to a quantity just sufficient to reduce all the oxid of iron of the mixture, the aluminium being depended upon only to reduce the titanic acid. The proportions were as follows: titaniferous mixture, (same as in I and II,) two hundred and seventy-five parts; reducing agent, (carbon fifty-eight to seventy per cent. or aluminium twenty-five to thirty per cent.,) eighty-three parts; iron-bath, two hundred and fifty parts. The alloy contained 19.82 per cent. titanium and a few tenths of one per cent. of carbon.

IV. In this case, operating always as above, a part of the titaniferous mixture was a highly-titaniferous slag, containing some sixty per cent. to sixty-two per cent. of titanic acid and practically no iron, (0.90 oxid of iron.) The titaniferous mixture was powdered and ag-

glomerated as above. The proportions were: titaniferous slag, sixty to sixty-two per cent.  $\text{TiO}_2$ , one hundred parts; titaniferous ore, about eighteen per cent.  $\text{TiO}_2$ , one hundred and seventy-five parts; iron-bath, two hundred parts; reducing agent, exclusively aluminium, ninety parts. The alloy contained 14.08 per cent. titanium.

Having now described my invention, what I claim as new, and desire to secure by Letters Patent, is the following, viz:

1. The process of producing a compound or alloy of iron and titanium which consists in providing a bath of molten reducing metal the heat of the formation of whose oxid is at a given temperature greater than that of titanic acid; next supporting in said bath in the presence of molten iron titanic acid, and developing in the charge a temperature sufficiently high to insure the requisite reaction between said reducing metal and the oxygen of the titanic acid leaving the liquefied metallic titanium to become alloyed with the liquefied iron, substantially as and for the purposes described.

2. The process of producing a compound or alloy of iron and titanium which consists in providing a bath of molten aluminium; next supporting in said bath in the presence of molten iron, titanic acid, and developing in the charge a temperature sufficiently high to insure the requisite reaction between said aluminium and the oxygen of the titanic acid leaving the liquefied metallic titanium to become alloyed with the liquefied iron, substantially as and for the purposes described.

AUGUSTE J. ROSSI.

Witnesses:

WALTER D. EDMONDS,  
PHILIP C. PECK,  
GEO. G. MEASURES.