

# UNITED STATES PATENT OFFICE.

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## PROCESS OF COMBINING TITANIUM WITH IRON.

SPECIFICATION forming part of Letters Patent No. 713,802, dated November 18, 1902.

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*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 of Combining Titanium with Iron, of which the following is a specification.

The object of my invention is to produce processes by the assistance of which predetermined and moderate amounts of pure metallic titanium may be introduced into iron for industrial purposes with uniformity, ease, and the certainty of securing a homogeneous product, and to enable these ends to be obtained, so far as the iron-founder is concerned, without disturbing the usual conditions or procedures with which the average workman is now familiar—*i. e.*, without any special or novel preparation or manipulation by him of ingredients or treatment thereof with any more than the present ordinary skill of his calling involved in, say, the familiar mixing of different grades of pig-iron in the cupola or in operations involving the production of steel in the converter or open hearth.

I have discovered that the alloys of titanium and iron, for which United States Letters Patent No. 609,466 were granted to me August 23, 1898, and which alloys are characterized as containing not less than five per cent. of titanium, are practically infusible *per se* under the ordinary temperatures sufficient to melt iron or steel, and that in those cases in which my said last-mentioned alloys are employed for seasoning iron with titanium the latter becomes incorporated in the resulting product by dissolving at the temperatures mentioned in the bath of molten iron or in contact with it analogously to the way in which platinum, likewise infusible *per se* at such temperatures, is known to dissolve in a bath of molten iron or to fuse in contact with it, whereby may be produced an iron containing platinum. Therefore in order to insure success in the seasoning or titanizing of iron by the use of my said alloys containing above five per cent. of titanium, and likewise the desired homogeneity of the product, these alloys must preliminarily be reduced to the powdered state, or at least broken

into small fragments, thus involving undesirable expense, owing to their hard and refractory character, or, again, in certain cases—as, for instance, when operating in the ladle stirring of the mixture becomes likewise requisite, all of which necessitates special preparation and treatment by the workmen, involving some inconvenience, the exercise of perhaps more than his usual skill and attention, and thus more or less undesirable expense, and this is especially the case when the operations involved are conducted upon moderate amounts of metal in the smaller foundries and plants. One of the causes contributing to render these manipulations necessary under the circumstances mentioned consists, doubtless, in the comparatively low specific gravity of the alloy of iron and titanium when high in percentage of titanium, the specific gravity of titanium being for instance 4.90, of an alloy containing ten per cent. of titanium 5.60, of cast-iron 7, and of iron 7.78.

My present processes comprise as an essential preliminary the production of an alloy of iron and titanium containing the latter in what my recent experiments and discoveries have demonstrated to be the required proportions for the purposes mentioned—that is to say, in excess of two and not to exceed five per centum of titanium. This new alloy I designate my “special titanic pig.” This I produce by supporting a mixture of ingredients containing such proportions of titanic acid, of iron, or of iron oxids and of carbon as to result in a pig-iron having the requisite proportion of titanium and subjecting the mixture until the requisite reactions have been thereby accomplished to the intense heat produced by the electric current—say a heat of 3,500° Fahrenheit.

It will be understood that the titanic acid in the mixture above referred to may be introduced in any convenient form—as, for instance, as contained in titaniferous iron ore or as rutile.

Any form of electrical device capable of producing the results described may be employed—as, for instance, a Siemens furnace of the type described as early 1879—or I have, for instance, used successfully for this pur-



pose a graphite furnace or crucible properly insulated by an outside lining of refractory materials, such as magnesia bricks or the like, properly secured by means of tie-rods, buckstuffs, and the like, the crucible being properly connected with one of the poles of the current by means of bus-bars, cables, or the like. Into this crucible was charged the mixture referred to, a more specific illustration of which will be given presently. Over this charge I lowered a large carbon or bunch of carbons constituting the anode properly connected with the positive pole of the current, the said anode being so disposed as to admit of its being gradually raised or lowered by any convenient mechanism for accomplishing that purpose. The current being turned on was passed through the materials to be reduced. The anode being of smaller diameter than the aperture of the crucible additions could be made to the charge from time to time as the reduction proceeded and the level of the mixture sank in the crucible. After the charge, with its additions, has been sufficiently exposed to the action of the current and the resulting high temperatures my special titanic pig is cast into proper molds through a suitable opening provided at the bottom of the crucible and the slag run out thereafter analogously to the similar practice involved in the production of pig-iron in cupolas and blast-furnaces, after which the crucible or furnace is charged again as before and the operation repeated indefinitely.

I have successfully produced my special titanic pig by using as one of the ingredients of the charge titaniferous iron ore containing on an average titanic acid, fifteen; oxid of iron, eighty; silica, 2.50; alumina, 1.50; magnesia, one; lime, trace; phosphorous, 0.017; sulfur, 0.045. As this ore, however, contained nine per cent. titanium, being too large a percentage for the purpose of producing the special titanic pig, I added in this instance a proper proportion of another non-titaniferous iron ore containing silica, 5.90; oxid of iron, eighty-five; alumina, 3.50; lime, 3.60; magnesia, 1.90; sulfur, 0.052; phosphorous, 0.07. As the titaniferous ore in this instance contained fifty-six per cent. iron and nine per cent. titanium and the non-titaniferous ore contained fifty-six per cent. iron and no titanium, I mixed one hundred pounds of the former and two hundred and thirty pounds of the latter with the expectation of securing by the process above described and after allowing for losses unavoidable in all metallurgical operations a resulting pig containing some four per cent. of titanium—say theoretically 4.30 per cent.—including carbon and silicon in the pig metal. The said ores properly pulverized were mixed with the requisite quantity of carbon, preferably in the form of charcoal powder, necessary to secure the reduction of the oxids of iron and titanium, or, say, about sixty-five pounds of charcoal, and were charged into

the crucible or electric furnace, as above described, with about ten pounds of caustic lime added to the mixture, in lieu of which I might have added its equivalent in limestone.

The resulting pig metal produced by the treatment of the aforesaid mixture by the process above described contained silicon, 1.90; titanium, 4.09; carbon, 5.26; iron by diff., 88.75; total, one hundred.

It will be observed that instead of using in admixture with the titaniferous ore an ore free from titanium, as above described, for the purpose of reducing the percentage of titanium in the mixture to the proper figure for the purposes of my special titanic pig there might with equally beneficial results be substituted for the non-titaniferous ores and in the required proportions ordinary pig-iron free from titanium.

As further illustrating the entire practicability of regulating with certainty and exactness the percentage of titanium in the resulting pig by proportioning the respective ingredients of the charge upon a theoretic basis, I will add that in similar operations I have found that a mixture which would theoretically yield a titanic pig containing 3.80 per cent. titanium treated as above described resulted in a pig-iron containing 3.68 per cent. titanium; also, that on similarly treating another mixture which would theoretically yield a titanic pig containing 4.38 per cent. titanium I obtained 4.28 per cent. titanium in the pig, and from another mixture which should have yielded 2.80 per cent. titanium I obtained 2.65 per cent. in the resulting pig-iron.

My special titanic pig having been produced as aforesaid, the next step in my present process consists in utilizing it for the seasoning or titanizing of iron containing no titanium. It is manifest that should my special titanic pig contain, say, less than two per cent. of titanium it would prove scarcely available for the purposes in hand on any industrially economical basis, since, among other reasons, too great a bulk thereof would be required to produce the requisite seasoning, and I am therefore of the opinion that it must prove necessarily preferable in all instances that the percentage of titanium should be not less than four per cent. The seasoning required is accomplished by introducing into the metal to be seasoned, while either cold in the charge or in molten condition, the requisite proportion of my special titaniferous pig, which, among other things, owing to its percentage of titanium, as before explained, will under the ordinary and usual temperatures required for the melting of iron employed in foundry-work and without preliminary treatment or other manipulation melt with the same facility as the ordinary non-titaniferous pig-iron with which it is associated.

To secure the required titanizing of iron, for instance, my special titanic pig is charged solid into the cupola and in its original and unbroken form as pigs or ingots, (the same



as any other pig-iron not titanic,) together with the pig-iron to be seasoned with titanium, care being taken to regulate the quantity of the titanic pig according to its percentage of titanium and the total mass of the mixture, so as to secure in the product the required percentage of titanium. The cupola is then run in all respects in the ordinary manner, including the employment of the same temperatures as in the case of ordinary iron, tapped, as usual, the resulting product flowing out into the ordinary receiving-ladle, from which the pouring-ladles are filled for any purpose of casting whatsoever, or if steel is to be made the contents of the cupola—i. e., the molten titanized pig-iron—are run directly into the converter. Again, if my special titanic pig is desired to be used in the open-hearth furnace it is in the required quantity charged into that also, together with the non-titanic pig-iron, and the mixture treated in all respects according to the usual practices of such furnaces the same as though no titanic pig were being smelted.

I am aware that pig-iron produced from titaniferous ores under such temperatures as are attainable in the blast-furnace has been claimed to contain sometimes some titanium; but the proportion of the latter thus attainable has been invariably very small, hardly reaching a few hundredths of one percentum, more exceptionally a few tenths, and whenever, and abnormally and fortuitously only, such quantities as one or even one and one-half percent. of titanium in iron have been observed in the products of experimental work or even of the blast-furnace the presence thereof has been due not to the direct reducing action of the carbon on the titanic oxid, since carbon cannot at the temperatures of the blast-furnace secure such reduction, which requires the intense heat of a powerful current, but to indirect causes, irregular, unreliable, dependent upon special or accidental conditions, such as are uncontrollable by the founder and which may often interfere with the proper and regular running of the furnace. So far as I am informed all efforts to introduce pure metallic titanium into iron in greater proportion than two per centum by the treatment of the ores in crucibles without the use of the electric current have failed. In fact, in many cases no trace of titanium has been discoverable by analysis in the so-called "titanic" product, and even when such a percentage has been supposed to have been observed the analysis has shown that the titanium was not present as pure metallic titanium, as in the case of my special titanic pig, but in combination with carbon and nitrogen, as nitride or cyano nitride of titanium, being products which have long previously been mistaken for titanium and the presence of which tends further to demonstrate that the reduction of the oxid of titanium at temperatures attainable in the crucible by a fire or even in the blast-furnace is not due to a direct action

of carbon on the titanic oxid, but to some special accidental, uncontrollable, uncertain, and unreliable conditions, it being notable that the percentage of titanium in the ore thus treated has no influence whatever upon the amount of titanium thus fortuitously found in the resulting pig. I do not wish, therefore, to be understood as claiming any part of my present invention either the production or use of iron containing titanium derived by processes other than that hereinafter specifically claimed.

What I claim as new, and desire to secure by Letters Patent, is the following, viz:

1. The process of combining homogeneously with iron a certain predetermined percentage of titanium which consists first in bringing together a mixture of titanic acid and iron, with carbon sufficient to deoxidize the titanic oxids, the proportion of titanic acid to the entire mixture being regulated so as to secure in the product derived from the next step not less than two percentum nor as much as five percentum of titanium, next supporting the said ingredients, and while so supported subjecting them to an intense heat say not less than 3,500° Fahrenheit, until the iron and the titanium resulting from the deoxidization of the titanic acid, are thoroughly molten and homogeneously commingled, next withdrawing the said molten mixture, from the said temperature, and allowing it to solidify; next, reducing to molten state, while suitably supported and by application of the temperatures usually required to melt the iron, both the iron to be titanized and the quantity of said solidified mixture required to impart to the resulting final product the required percentage of titanium, and continuing the said support subject to said last-mentioned temperature until the constituent titanium has been melted and homogeneously distributed throughout the mass and finally withdrawing the said product from the said temperature and allowing it to solidify substantially as and for the purposes described.

2. The process of combining homogeneously with iron a certain predetermined percentage of titanium which consists first in bringing together titaniferous iron ore, with carbon sufficient to deoxidize the iron and titanic oxids, the proportion of titanic acid to the entire mixture being regulated so as to secure in the product derived from the next step not less than two per centum nor as much as five per centum of titanium, next supporting the said ingredients, and while so supported subjecting them to an intense heat say not less than 3,500° Fahrenheit, until the iron, resulting from the deoxidization of the oxid of iron, and the titanium, from the deoxidization of the titanic acid, are thoroughly molten and homogeneously commingled, next withdrawing the said molten mixture from the said temperature and allowing it to solidify, next reducing to molten state, while suitably supported and by application of the tempera-



tures usually required, both the iron to be  
titanized and the quantity of said solidified  
mixture required to impart to the resulting  
final product the required percentage of ti-  
5 tanium, and continuing the said support sub-  
ject to said last-mentioned temperature un-  
til the constituent titanium has been melted  
and homogeneously distributed throughout  
the mass and finally withdrawing the said  
10 product from the said temperature and allow-  
ing it to solidify substantially as and for the  
purposes described.

3. The process of titanizing iron which con-  
sists first in producing a pig-iron character-  
15 ized as containing a percentage of titanium  
sufficiently low to insure the melting of the  
titanium at the melting temperature of the  
iron and sufficiently high to effect the re-  
quired titanizing by the use of an industrially  
20 economic minimum of said pig, that is to say  
a pig containing not less than two per cent.  
nor more than five per cent. of titanium;  
next, combining the iron to be titanized with  
the said titaniferous pig in the proportions  
25 necessary to produce the required percentage

of titanium in the resulting product and melt-  
ing the iron and the titaniferous pig together,  
substantially as and for the purposes de-  
scribed.

4. The process of producing a pig-iron con- 30  
taining a predetermined proportion of ti-  
tanium fusible under the ordinary melting  
temperatures of pig-iron, that is to say not to  
exceed five per cent. of titanium which con- 35  
sists in supporting titaniferous iron ore hav-  
ing the requisite proportion of titanic acid in  
the presence of carbon and subjecting the  
mixture to an intense heat say not less than  
3,500° Fahrenheit until a bath of molten iron 40  
is produced by the dioxidization of the iron  
oxid and until the titanium has been re-  
duced and melted and a homogeneous molten  
mixture of iron and pure metallic titanium  
secured substantially as and for the purposes  
described.

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Witnesses:

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