

UNITED STATES PATENT OFFICE.

AUGUSTE J. ROSSI AND WILLIAM F. MEREDITH, OF NIAGARA FALLS, NEW YORK,
ASSIGNORS TO THE TITANIUM ALLOY MANUFACTURING COMPANY, OF NEW YORK,
N. Y., A CORPORATION OF MAINE.

COMPOUND OR COMPOSITION OF MATTER AND METHOD OF PRODUCING SAME.

1,039,672.

Specification of Letters Patent.

Patented Sept. 24, 1912.

No Drawing.

Application filed April 17, 1912. Serial No. 691,515.

To all whom it may concern:

Be it known that we, AUGUSTE J. ROSSI and WILLIAM F. MEREDITH, both citizens of the United States, residing at Niagara Falls, in the county of Niagara and State of New York, have jointly invented certain new and useful Compounds or Compositions of Matter and Methods of Producing Same, of which the following is a specification.

Our present inventions relate to the production of essentially metallic substances or compounds particularly useful in the purification or cleansing of metals from therein occluded undesired elements and their compounds including slags, or in imparting other superior qualities to metals.

The objects of our inventions comprise production of more efficient such compounds, and by methods more rapid, certain and economical than heretofore, and we attain these objects by our novel method of production and novel resulting compounds as hereinafter disclosed and claimed.

The nature, novelty and scope of our present inventions will be the better understood by reference to the prior art relating to compounds useful for the improving or purifying purposes referred to. It is believed that these are best exemplified in their highest development, and as now applied on extensive industrial scales, by numerous Letters Patent of the United States heretofore issued to Auguste J. Rossi, of which, for example, No. 609,466, dated August 23, 1898, discloses the production, for the said purposes, of an alloy of iron and titanium containing also some carbon. Inspection of the said Rossi patents and of the prior art will, it is believed, disclose that hitherto it has been supposed that the aforesaid improvements of other metals by aid of titanium as the effective agent could be successfully accomplished only by addition to such other metals, while molten, of an alloy or compound containing titanium in its elemental or metallic state. The previous industrial practice of such improvements on extended scales has apparently proceeded exclusively on this theory, the methods practised of producing such alloys or compounds, for instance those of iron and titanium, as evidenced by the Letters Patent referred to, involving pro-

cedures carefully guarded to secure a final product devoid, as far as possible, of the chemical compounds of titanium with other elements, and particularly it has not, prior to our present invention, been known or suggested, so far as we are aware, that compounds of titanium with carbon, *i. e.* carbids of titanium, could as additions to molten metal operate successfully, or at all, to accomplish its said purification or other improvement. We are now however satisfied that this is an error, and that, on the contrary, carbid of titanium, provided it be very intimately mixed and homogeneously conglomerated in very minute sub-divisions with other metal, is, if the compound so constituted be added to molten metal to purify or improve it, ultimately as efficacious, or more so, than metallic titanium added as such, and this at considerably less cost to the manufacturer. This novel compound, so containing the carbid of titanium, as substitute for metallic titanium, possesses, we have found, characteristics rendering it superior, for industrial uses and purposes, to said alloys of elemental titanium with other metals, as for instance the ferro-titanium product of the aforesaid Letters Patent, such superiorities comprising its comparatively economical production, as per the method hereinafter described, and also its comparative stability, or capacity to delay momentarily liberation of the metallic titanium, thus enabling less of latter to produce more complete purification owing to its more complete permeation and distribution throughout the bath during disintegration of the carbid therein before actual purification by liberated metallic titanium begins.

It will be understood that, prior to our present invention, the practised methods of producing alloys of titanium by carbon reduction, as for example per the process described in said Letters Patent No. 609,466, involved application of temperature, *i. e.* energy, to the charge sufficiently great to insure presence, in the final product, of the most possible of metallic titanium; also the addition of substantially no more carbon than calculated to be sufficient to reduce the oxids of said charge, and leave in the final product not to exceed a comparatively small

percentage of uncombined carbon, *i. e.* carbon in graphitic state, thereby insuring the acquisition and retention in the alloy of the greatest amount of metallic titanium derivable from the charge. To this end, in producing said alloys, the additions of carbon were regulated accordingly, and, the temperature of the bath raised and maintained, at corresponding expense of energy, to insure not only the reduction of the oxides but also the prevention of carbids in the final product. This procedure resulted in two considerable items of expense, which our method saves to the manufacturer, *viz*: the expense of maintaining the temperature sufficiently high and long to attain the desired metallic titanium result, and also the expensive drafts meanwhile made upon the constituents of the carbon electrodes employed and also upon the graphitic walls of the furnace, and aggravated by the aforesaid stinting of the carbon additions to the charge to insure the previously desired constitution of the final alloy product. Our novel product, or composition, is thus not only more effective and economical than the said alloys of titanium for purifying, or other improvement of metals, but is also easier and cheaper to produce, and therefore correspondingly more desirable and useful, particularly in view of the present magnitude of operations in metals being practised by aid of metallic titanium.

We are satisfied that additions of carbid of titanium in isolated form to baths of such metals must prove ineffective to improve them, owing not only to the magnitude of the sub-divisions of such carbid, however finely pulverized by any economically practicable mechanical means, but also to the comparative indissolubility, in the bath, of carbid thus added by itself alone. We have found, however, that the carbid, if added in association with other metal, and in the condition of much smaller sub-division and homogeneous conglomeration with such metal resulting from our method of production, not only dissolves freely, rapidly, and homogeneously, throughout the bath of metal treated, thus permeating it thoroughly, but also that its content of combined carbon soon thereafter, owing to the existing temperature and other conditions, separates from its content of titanium, thus releasing within the metal treated, and when and where most needed, a multitude of comparatively homogeneously distributed very minute particles of free metallic titanium, which then combine with the undesired elements and compounds present to form new compounds which, as is now well understood, accomplishes the desired purification, the surplus of titanium, if any desired, imparting to the final product other improved characteristics for special uses.

It will be observed that the addition of the titanic agent in the form of its carbid thus serves, in a measure, as it were, to postpone the action of the titanium as a purifier until better opportunity accorded for its extended and intimate co-mixture with the ingredients of the bath, as compared to the hitherto additions of the titanium in metallic state when it immediately commences its reactions at the points where introduced and without similar opportunity accorded for preliminary, more uniform, distribution. It may be that this increases momentarily the time of treatment required, *i. e.* the interval in the operation after addition of the purifying agent, but this, requiring only a few minutes, we regard as more than counterbalanced by the greater opportunity for more evenly distributed action and purification accorded by the carbid character in which the titanium is introduced as above explained, the result including a saving in the total amount of metallic titanium otherwise required to produce the desired final result.

Our said novel purifying compound, possessing the required characteristics above referred to, may be produced as follows:—In a furnace capable of developing and maintaining the required temperatures, as for example preferably an electric furnace of ordinary type and construction, such for instance as shown and described in U. S. Letters Patent to Auguste J. Rossi, No. 802,941, dated October 24, 1905, or No. 822,305, dated June 5, 1906, is charged, in such proportions as may be desired, metal, for example iron, also titanium oxid and also carbon, the latter preferably in such proportions as not only to satisfy the affinity therefor of the oxygen of said oxid, but also enough to fully satisfy the affinity therefor of the therefrom liberated titanium, and also enough carbon besides to insure to the resulting product such uncombined carbon content as may be desired, these, more liberal additions of carbon than heretofore, resulting also in desirably diminishing the former expensive losses of carbon from the electrodes and the graphite linings of the hearth or crucible.

It will be understood that instead of the metal or iron, mentioned, oxides thereof might be charged, care being, in that case, taken to increase the proportion of carbon so as to insure their reduction also. The temperature of the charge is then raised, as by turning on the current, but no higher than sufficient to insure the reduction of the oxid of titanium. It will be understood that the application of such temperature will result in the formation of a bath of molten metal other than titanium, and this whether introduced in elemental or in oxid form, provided in the latter case sufficient carbon be added. The said temperature is then

maintained only sufficiently long, and high, to insure, as thoroughly as possible, not only the said reduction of oxids, but also formation, throughout the bath, of the most possible of carbid of titanium. When this stage has been reached, the temperature must be lowered as rapidly as is industrially possible, lest continuation, or even slow diminution, thereof result in robbing the carbid of its combined carbon, as by latter's passing into the graphitic state, or making new combinations as with oxids present. This required speedy lowering of the temperature may be accomplished by tapping the charge as soon as it has reached the stage mentioned, and promptly casting the molten product sufficiently distributed in graphite, or other heat conductive receptacles. After it has thus, or otherwise, been rapidly cooled, our carbid-containing final product is ready for use as a purifying, or otherwise improving, addition to baths of molten metals, to which it may be added in manner substantially as indicated by prevailing practise when ferro-titanium, for example, is used as a purifier.

It will be understood that our compounds of titanium carbid with other metals than iron are producible by the method above specifically described, the procedure in each case being substantially the same except that such other desired metal, or its oxid, is substituted for the iron. Thus compounds of carbid of titanium with copper, tin, lead, zinc, manganese, antimony, etc., may be produced for use in the purification of such metals. Our said compounds so obtained will be found to comprise the metal of the charge, having therewith intimately mixed and homogeneously conglomerated minute particles or sub-divisions of carbid of titanium, its said minute sub-division and association with metal being as aforesaid, we believe, particularly important, if not indispensable, to the successful action thereafter of the titanium constituent of the car-

bid as a purifying or otherwise improving agent.

It seems unnecessary to further describe the details of operation required to produce our said compound, since the necessarily ever-varying character of subordinate details will be readily understood by those skilled in the metallurgical art. For instance, the proportions and temperatures of each charge will, of course, be varied to suit requirements of each particular case and of the particular type and quality of the constituents employed; but we prefer, as more universally useful, to regulate the proportions of our said compounds so that their content of carbid of titanium is from 10 to 20 per cent. by weight of the mass, but such compounds containing as low as 5 per cent. of said carbid may be profitably employed, and also as high as 80 per cent. It will be understood that a low content of the carbid may prove undesirable owing to the proportionally greater bulk of the compound required for purifying. On the other hand it seems inadvisable to exceed 80% of carbid in the compound because of inversely proportional influence of the therewith associated constituent metal of the compound when the latter is added as a purifier to a bath of molten metal.

Having now described our invention, what we claim as new and desire to secure by Letters Patent is:—

1. As a new article metal containing therewith intimately mixed not less than 5 per cent. and not more than 80 per cent. of carbid of titanium.

2. As a new article iron containing therewith intimately mixed not less than 5 per cent. and not more than 80 per cent. of carbid of titanium.

AUGUSTE J. ROSSI.

WILLIAM F. MEREDITH.

Witnesses:

WALTER D. EDMONDS,
PHILIP C. PECK.