No. 745,122. PATENTED NOV. 24, 1903.

F. J. TONE.

REDUCTION OF METALS AND ALLOYS.

APPLICATION FILED JAN. 22, 1902.

NO MODEL. 2 SHEETS—SHEET 1.

Fig. 1.

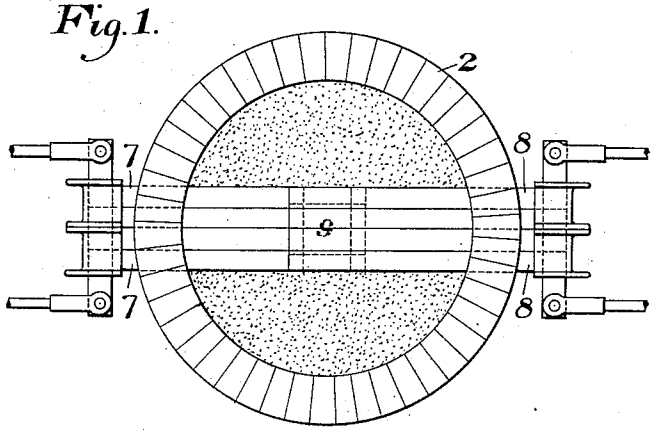
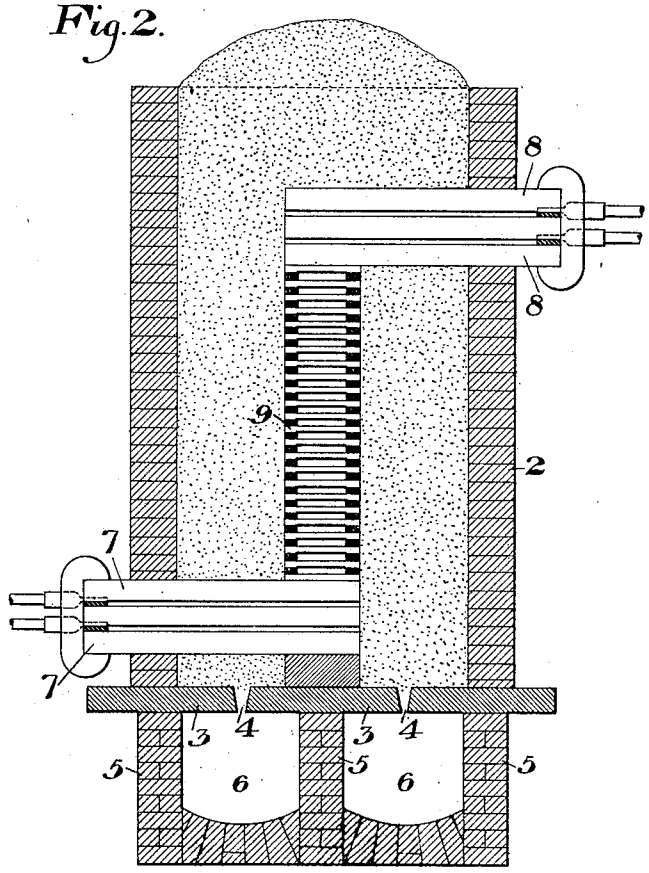


Fig. 2.



WITNESSES

Warren W. Swartz

A. M. Corrie

INVENTOR

F. J. Tone

by Barnwell & Byrnes

his Attorneys

THE NORRIS PETERS CO., PHOTO-LITHO., WASHINGTON, D. C.

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APPLICATION FILED JAN. 22, 1902.

NO MODEL. 2 SHEETS—SHEET 2.

Fig. 3.

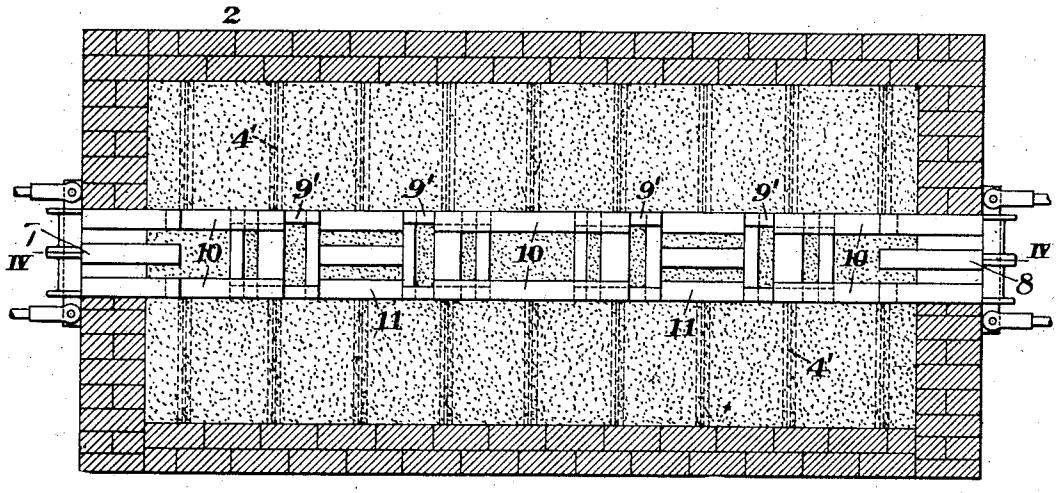
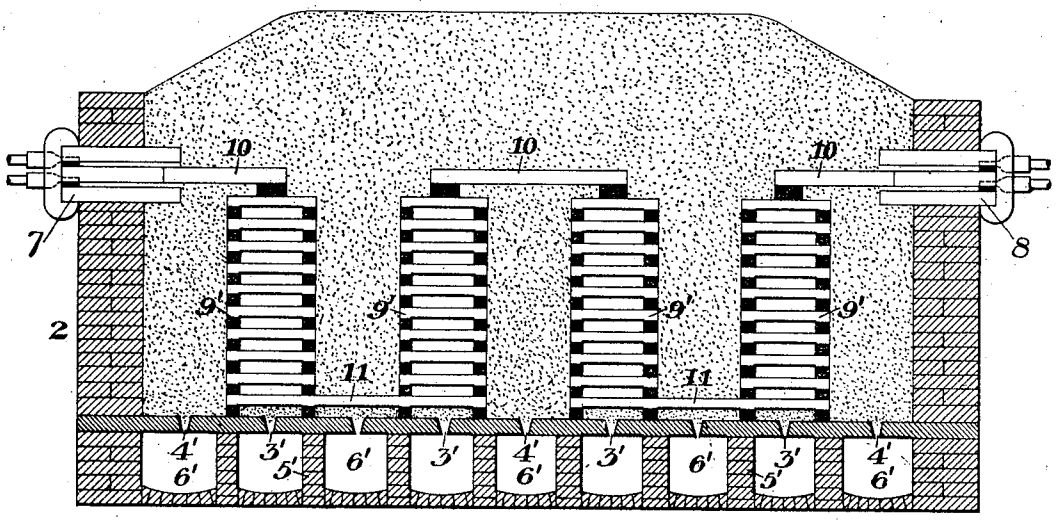


Fig. 4.



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A. M. Corwin

INVENTOR

F. J. Tone

by Burdett & Byrnes

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UNITED STATES PATENT OFFICE.

FRANK J. TONE, OF NIAGARA FALLS, NEW YORK.

REDUCTION OF METALS AND ALLOYS.

SPECIFICATION forming part of Letters Patent No. 745,122, dated November 24, 1903.

Application filed January 22, 1902. Serial No. 90,793. (No model.)

To all whom it may concern:

Be it known that I, FRANK J. TONE, of Niagara Falls, in the county of Niagara and State of New York, have invented a new and useful Improvement in the Reduction of Metals and Alloys, of which the following is a full, clear, and exact description.

In the attempts heretofore made to reduce from their compounds certain metals whose temperatures of reduction and volatilization are nearly the same, such as silicon and aluminium, and to produce silicides of certain elements other than carbon difficulty has been experienced in accomplishing the reduction by means of carbon because of the seeming tendency of such elements to form carbids. It has been frequently stated by experimenters that a mixture of the oxids of such metals with sufficient carbon to combine with the oxygen does not when the mixture is heated to the temperature of reduction yield the metal as the main resulting product, but yields the carbid, for the reason that the metal becomes volatile as soon as it is formed and combines with uncombined carbon present in the mixture. I have discovered that this difficulty can be avoided by proper conditions of temperature, proper distribution of heat, and proper arrangement of the charge to be treated in the furnace, as hereinafter set forth.

My invention is illustrated in the accompanying drawings, in which—

Figure 1 is a plan view of a furnace adapted to the practice of my invention. Fig. 2 is a vertical central section of the same, showing a furnace charged with material to be treated. Fig. 3 is a horizontal section of a modified construction of the furnace, and Fig. 4 is a longitudinal section on the line IV IV of Fig. 3.

I will illustrate my invention with reference to its operation in the reduction of the metal silicon or aluminium, in the reduction of which it has been found difficult to prevent the formation of carbids, as stated above, and also to prevent immediate volatilization of the metallic products, owing to the fact that their temperatures of reduction and volatilization are nearly the same. For example, in a mixture of silica and carbon which is brought to the temperature necessary for the reduction to silicon the following reactions occur:

(1.) $SiO_2+2C=Si+2CO$.

(2.) $SiO_2+3C=SiC+2CO$.

(3.) $2SiC+SiO_2=3Si+2CO$.

(4.) $Si+C=SiC$.

Equation 1 represents a mixture of the theoretical proportions required for the reduction of all the silica to silicon without the formation of any carbid, and if it be conceived that the materials could be so thoroughly mixed and in so fine a state of subdivision that every molecule of SiO_2 is in proximity to two atoms of carbon, and certain other molecules of SiO_2 will be in proximity to two atoms of carbon, and certain other molecules of SiO_2 not in proximity to carbon there will be three products of the reaction—viz., Si, SiC, and SiO_2, as indicated by the equation:

$$4SiO_2+8C=Si+2SiC+SiO_2+6CO.$$

When the mixture indicated by equation 1 is finely subdivided and well incorporated, I have found that the reaction under proper conditions gives a large proportion of Si and only a small proportion of SiC and SiO_2. Under these conditions there is no evidence that the silicon volatilizes as soon as it is formed, and owing to the peculiar construction of my furnace, as explained below, it agglomerates into molten masses and seeks by gravity the lower portions of the furnace, leaving the products SiC and SiO_2. These being brought together, the secondary reaction $2SiC+SiO_2=3Si+2CO$ occurs, and the reduction is complete. This reaction should not be regarded as primary, as in the state of Si in SiC is merely transitory.

For the economical production of silicon the mixture of silica and carbon should be thorough and the constituents finely subdivided in order that the major portion of the charge mixture may be at once converted into silicon, leaving only a minor portion for the secondary reaction. The distribution of heat should be over a wide zone (as opposed to localized heat) in order that the progress of the reaction may be relatively slow and give the best conditions for the agglomera-

tion of particles. The most even temperature possible should be maintained throughout the zone of reaction, so that the silicon shall not be volatilized as soon as it is formed, and, lastly, the charge should be so arranged in the furnace as to allow the globules of Si to drop by gravity out of the zone of reaction to a lower portion of the furnace.

The chemistry of aluminium is so well known that I deem it unnecessary to state it in detail; but it is evident that the above principles not only apply to this metal, but to all others whose temperatures of reduction and volatilization are nearly the same.

In carrying the above principles into practice I employ an incandescent furnace as distinguished from an arc-furnace. In furnaces of the latter type a large amount of energy is liberated in a relatively small space, causing the reaction to proceed very rapidly. In an incandescent furnace the energy is radiated from a relatively large surface, and while the same temperatures may be attained it requires longer time and the reactions proceed more slowly. This slowness of reaction and absence of violence is important in order to allow the reduced particles to gather together into globules, when they are less subject to secondary reaction. Also to prevent immediate volatilization of said globules I provide a space 6 beneath a resistance-column the major portion of whose radiating-surface is disposed in a substantially vertical direction, whereby said globules gravitate out of the zone of maximum temperature as soon as they are formed, and new portions of the mixture are thus continuously exposed for reduction.

For the purpose just stated I dispose the resistance material in an approximately vertical direction or form it so that the major portion of its radiating-surface will be in approximately vertical planes, and, although preferable, it is not essential that the current-path shall be in a vertical direction, it being possible to use any suitable form of resistance-bar so arranged as to give a zone of reaction and reduction disposed along substantially vertical planes.

I prefer to use the apparatus shown in the accompanying drawings, in Figs. 1 and 2 of which 2 is an electrical furnace whose walls are preferably made of refractory brick and whose floor 3 is composed of a thin layer of refractory brick or tile laid so as to afford spaces 4, through which the reduced and molten material may drop. These flooring-tiles may be supported on a substructure of brick piers 5, between which are spaces 6 for the collection of the reduced metal. The charge of silica and carbon is placed within the furnace on the floor 3 and is preferably charged into the furnace from the top. 7 7 are carbon terminals or conductors at the lower part of the charge. 8 8 are the upper terminals, which enter the furnace at or near its upper portion, and between them is a vertical column 9, made of carbon blocks preferably piled together with intervening spaces, so as to afford a conductor which connects the terminals and is of sufficient resistance to cause the generation of the required heat.

The current is passed through the resistance-column 9 from one terminal to the other, and the heat generated therein is communicated to the surrounding column of charged mixture. The distribution of heat from this column is even and gradual, and as the silicon is reduced therefrom it sinks down below the column into the cooler portion of the furnace and finally into the receptacles 6, where it solidifies as a metallic block.

The resisting column may be constructed otherwise than by carbon blocks, though I find this a very convenient arrangement, and the lower portion of the furnace may be constructed otherwise than as shown, it being only essential that there shall be a portion of the furnace below the zone of heat in which the reduced metal can collect.

In Fig. 3 I show a modification of the apparatus in which the furnace-receptacle is rectangular in form and the terminals 7 8 are at the ends of the furnace in the same horizontal plane. The resistance material or path for the current instead of being a single column, as in Fig. 1, is composed of two or more columns 9′ set vertically on the furnace-floor with spaces between them, and these columns are connected in series by bridging their upper and lower ends alternately by carbon conducting-bars 10 11. In this figure I also show the furnace formed with a floor of tiles with intervening spaces 4′, through which the metal may drop, and with receptacles 6′ below these spaces for the reception of the reduced metal. The charge of the furnace fills the spaces between the conducting-columns 9′, and as the current passes through the latter and heats them the heat is communicated to the charge and the silicon is reduced and collects at the bottom of the furnace.

The apparatus may be varied in other ways by the skilled electrician; but I regard the construction shown as preferable, for the reason, among others, that the conducting resistance material is self-sustained, and not being carried by the charged mixture is not apt to be displaced by settling of the latter and also because the greater portion of the effective radiating-surface is disposed in vertical planes.

In the use of both these forms of apparatus there is a wide zone of reaction and a slowly-developed temperature is employed whose limits are high enough for the reduction of the metal, but not so high when the metal is immediately removed as to cause its waste by volatilization and conversion into carbids.

I claim—

1. The method herein described of reducing from their oxygen compounds, metals whose temperatures of reduction and volatilization are nearly the same, which consists

in subjecting such oxygen compounds admixed with carbon, to heat in an incandescent furnace, keeping the temperature of the charge below the limits at which volatilization occurs, and removing the reduced metal from the zone of maximum temperature as fast as it is formed; substantially as described.

2. The method herein described of reducing from their oxygen compounds, metals whose temperatures of reduction and volatilization are nearly the same, which consists in subjecting such oxygen compounds admixed with carbon, to heat in an incandescent furnace, the conducting resistance material of which is disposed with the major portion of its effective radiating-surface in an approximately vertical direction, keeping the temperature of the charge below the limits at which volatilization occurs, and removing the reduced metal from the zone of maximum temperature as fast as it is formed; substantially as described.

3. The method herein described of reducing from their oxygen compounds, metals whose temperatures of reduction and volatilization are nearly the same, which consists in subjecting such oxygen compounds admixed with carbon, to heat in an incandescent furnace, the conducting resistance material of which is disposed with the major portion of its effective radiating-surface in an approximately vertical direction, keeping the temperature of the charge below the limits at which volatilization occurs, providing a space below the resistance material in which the reduced material may collect, and removing the reduced metal from the zone of maximum temperature as fast as it is formed; substantially as described.

4. The method herein described of reducing from their oxygen compounds, metals whose temperatures of reduction and volatilization are nearly the same, which consists in subjecting such oxygen compounds admixed with carbon, to heat in an incandescent furnace, whose zone of reaction and of maximum temperature is disposed in substantially vertical direction, keeping the temperature of the charge below the limits at which volatilization occurs, and removing the reduced metal from the zone of maximum temperature as fast as it is formed; substantially as described.

5. The herein-described method of producing metallic silicon, which consists in reducing a silicon compound and concurrently fusing the product into a dense coherent pig or mass, substantially as described.

6. The herein-described product, consisting essentially of metallic silicon in the form of a dense coherent pig or mass, produced by reduction of a silicon compound and concurrent fusion of the product, substantially as described.

In testimony whereof I have hereunto set my hand.

FRANK J. TONE.

Witnesses:
T. S. MANLEY,
R. S. MARVIN.