UNITED STATES PATENT OFFICE.

EDWARD GOODRICH ACHESON, OF BUFFALO, NEW YORK.

METALLURGICAL PROCESS.

SPECIFICATION forming part of Letters Patent No. 693,482, dated February 18, 1902.

Application filed October 14, 1899. Serial No. 733,635. (No specimens.)

To all whom it may concern:

Be it known that I, EDWARD GOODRICH ACHESON, a citizen of the United States, residing at Buffalo, in the county of Erie, State of New York, have invented certain new and useful Improvements in Metallurgical Processes, of which the following is a specification.

My invention relates to metallurgical processes, and more particularly those for the production of pure metals, carbids, and other chemical compounds that are produced in an electric furnace in which carbon is used as a conducting, reducing, or combining agent; and it has for its object to provide a process or method of cheaply and practically producing pure metals, carbids, alloys, and other chemical compounds and products, and it may be said to consist, generally stated, in the use of graphite as a conducting, reducing, or combining agent when associated with the metallic ore to be reduced or other substances or compounds to be operated upon.

Efforts heretofore made for the practical direct production of pure products in an electric furnace—as, for example, metals like silicon—or for the production of pure compounds—as, for example, carbids of calcium—have proved failures, owing, among other things, to difficulty in effecting the reduction and also to the introduction into the finished product of impurities contained in the carbon used as a conducting, reducing, or combining agent, or to carbon itself, resulting in the formation of impure products. I have discovered that the above difficulties attending the reducing action in the electric furnace can be overcome and greatly improved results obtained by using as conducting, combining, and reducing agent graphite, and preferably graphite which has been produced by the electric furnace. In this process I utilize not only the purity and high electrical and thermal conductivity of graphite, but I have discovered that its softness and divisibility give it a peculiar property of being brought to an extremely effective distribution with the ore or other substance to be operated upon. Its high electrical conductivity facilitates the passage of sufficient current to produce the desired reactions. Its high thermal conductivity facilitates the conduction of heat throughout the mass of ore or other substances with which it is mixed, and its softness and divisibility facilitate its distribution throughout the mass of ore or other substance with which it is mixed and permit the fullest utilization of the graphite as an electrical and thermal conductor and reducing agent.

I have found that when an ore such as granulated silica and amorphous carbon in an amount sufficient for the reduction of such ore are mixed in the manner and to the extent ordinarily practiced in making a mixture of said materials the mixture is practically non-conductive for currents as ordinarily used for metallurgical work, and even after a prolonged or thorough mixture its electrical conductivity is not sufficiently high for practical operation in the process of electrical reduction of the ore. When, however, graphite is substituted for the amorphous carbon, owing to its high conductivity, softness, and divisibility it can be so mixed with the ore or other material as to furnish the proper and desired degree of electric conductivity, and owing to the softness and divisibility of the graphite during the process of mixing it with the granulated ore the particles of ore become wholly or partially coated with the graphite, and good electrical conductivity is established throughout the mass with all the particles thereof by reason of this surface coating of the particles of the ore. In order to get this partial or complete coating of the particles of ore, it is necessary to thoroughly mix the particles of ore and graphite, and this is preferably accomplished by a tumbling or rubbing process, and the increase of conductivity and successful resultant reducing operation depend largely upon the thoroughness with which the graphite is coated or spread upon the surface of the particles of ore. Thus even when the particles of ore are mixed with graphite in the manner and to the extent ordinarily practiced in making a mixture I have found that the electrical resistance is excessively high, so as to practically inhibit the use of the electric current in reducing the ore, but on continuing the mixing and rubbing process, so that the particles of ore become partially or wholly coated with the graphite, the electrical resistance of the mass is reduced until its conductivity is so far increased as to permit of the successful operation of the reduction with electric currents as ordinarily used in electrometallurgical processes. For the application of my invention I reduce the ore and graphite to particles, preferably to small particles or powder, placing the desired amount of each in a suitable tumbling barrel or mixer and mixing the same, thus to a greater or less extent coating or varnishing the ore particles with the soft graphite. The mixture of ore and graphite is then introduced into an electric furnace, where it is subjected to a temperature sufficient to cause the desired reactions—as, for instance, the reduction of the ore and the liberation of the metal. While any kind of suitable furnace may be used, a direct-acting furnace, wherein the charge to be operated upon is placed or caused to pass directly between the electrodes and in which the current passes through the charge, is more desirable.

Graphite made in the electric furnace has the advantage of great purity, sometimes containing as low as 0.033 per centum of ash, having the capacity, owing to its soft nature and divisibility, of being brought into very intimate contact with the ore and also possessing extremely high electric and thermal conductivity, having usually more than twice the conductivity of the best conducting amorphous carbon. High electrical conductivity is of first importance—for example, when the metal on being freed from the ore combines with any free carbon or other element present to form carbids, alloys, or impure metal and it is desired to obtain a pure metal—for instance, in reducing silica for the production of pure silicon. In making pure elementary substances—such, for example, as silicon—by direct reduction it is essential that the contents of the furnace consist solely of the pure ore or silica and carbon in an amount not greater than necessary for the reduction. Such a mixture of silica and carbon in the amorphous condition is an extremely poor conductor of electricity, and for this reason it is impracticable to make pure silicon when the amorphous carbon is used. When graphite, however, is used for this purpose and is mixed with the silica in a like amount and in the manner above indicated, I have discovered that a good conductivity is obtained and a successful and practicable operation can be performed.

A good illustration of the value and application of my invention is in the reduction of those ores, such as silica and alumina, which are difficult of reduction and from which it has not been heretofore practicable to extract the metal directly in a pure state.

All four of the qualities belonging to graphite and above mentioned—purity, electrical conductivity, thermal conductivity, and capacity of coating or being spread over the surface of the particles with which it is brought in contact—are of great value. Purity is essential, as it is well known that the presence of a very small percentage of other elements will frequently make metals worthless. Capacity for intimate mixture and coating of the ore to be reduced is of great importance, as is also high electrical conductivity of the reducing-carbon, for on it must be placed the burden of conducting the electric current, and usually an excess of this carbon over that necessary for the reduction is not permissible, as a carbid of the metal would be produced or the excess of carbon would enter as an impurity in the metal. High thermal conductivity is desirable, as it facilitates the distribution of the heat throughout the mass of the charge where there is a tendency to its local generation, as would occur where the current found a path of least resistance.

As one example of the application of my invention I will describe the reduction of silica and extraction of pure silicon. I take electrically-made graphite and pure silica both in a state of fine powder and mix them in the proportion of graphite fifty-five parts and silica one hundred and fifty parts. The theoretical proportion would be graphite fifty-five parts and silica 137.5 parts; but I prefer to have a little less than the theoretical amount of carbon. The mixture is thoroughly rubbed together in a mortar, tumbling-barrel, or other suitable device. The amount and perfection of the intermingling and rubbing has much to do with the efficiency of the operation and with the electromotive force necessary to employ for passing the electric current through the mass when in the electric furnace, and it should be well done. The material thus prepared is placed in an electric furnace in such manner that the current passing from electrode to electrode will pass through it, and around the charge on the bottom, side, and top is placed fine silica as a support and covering to the mixture and lining to the furnace, which may be made of brick or other suitable material. The mixture having been placed in this manner in its envelop of silica and in direct contact with the opposing electrodes the current is caused to pass in sufficient volume and for sufficient time to produce the reduction of the silica and liberation of the silicon. Thus in an operation which I have conducted with the electrodes separated to a distance of three inches and each having an exposed end surface of four square inches, the intervening space measuring three inches by two inches by two inches, with its floor and sides of silica, was filled with the mixture of graphite and silica above described, and over all was placed a layer of silica. The current was turned on with one hundred volts. At the beginning the current registered three amperes, but quickly rose and after seven minutes registered eighteen amperes. The current was turned off after twenty-five minutes, the maximum voltage having been one hundred with twenty amperes. Carbon-monoxid gas escaped freely and burned on the surface. On opening the furnace a part of the silicon resulting from the silica of the charge is found as globules of pure silicon mingled with other portions of the charge not wholly reduced. These globules may be separated from the unreduced mass by floating in water or other desirable method.

It is evident that various forms of furnaces may be used in carrying out my invention and that the product may be removed in many ways. Thus it may be removed intermittently either in a fluid or solid condition, as is the practice in making carborundum, carbid of calcium, and aluminium continuously either in a solid, liquid, or gaseous state. When the product of the operation is a volatile metal and can be removed from the furnace in a gaseous condition, as in the reduction of zinc and other metals, suitable condensing-chambers are provided for its reception and retention.

When alloys are to be made of two or more metals, one or more of the metals may be mixed in the powdered metallic state with the ore to be reduced, or the ores of the several metals may be mixed with the appropriate amount of graphite to produce their united reduction, and the metals when liberated will alloy together. Thus oxid of iron and silica in the proportions necessary for the production of the desired silicid of iron, when mixed with the necessary graphite and raised to the proper temperature, will be reduced to a silicid of iron. When carbids are to be made, the amount of graphite employed in the charge is increased over and above that necessary for the reduction to at least the extent of the amount necessary to combine with the metal and form the desired carbid.

I claim—

1. The method herein described of reducing ores, which consists in mixing pulverized ore with graphite as the sole conducting and reducing agent, both in a dry state, coating the particles of ore with graphite, and passing an electric current through the mass of such coated particles, thereby heating it to a point sufficient to effect reduction of the ore.

2. The method herein described of reducing ores, which consists in mixing pulverized ore with sufficient graphitic carbon as the sole conducting and reducing agent, to reduce the ore, both in a dry state, coating the particles of ore with the graphitic carbon and passing an electric current through the mass of such coated particles, thereby heating it to a point sufficient to effect reduction of the ore.

3. The method herein described of reducing ores, which consists in mixing pulverized ore and graphitic carbon in the theoretical proportions necessary for the reduction of the ore, coating or partially coating the particles of ore with the graphitic carbon, and passing an electric current through the mass of such coated particles, thereby heating it to a point sufficient to effect reduction of the ore.

In testimony whereof I have hereunto set my hand.

EDWARD GOODRICH ACHESON.

Witnesses:

GEO. B. BLEMMING,

C. C. BITTNER.