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F. C. WEBER.

PROCESS OF REDUCING METALLIC OXIDS WITH METALLIC ALUMINIUM.

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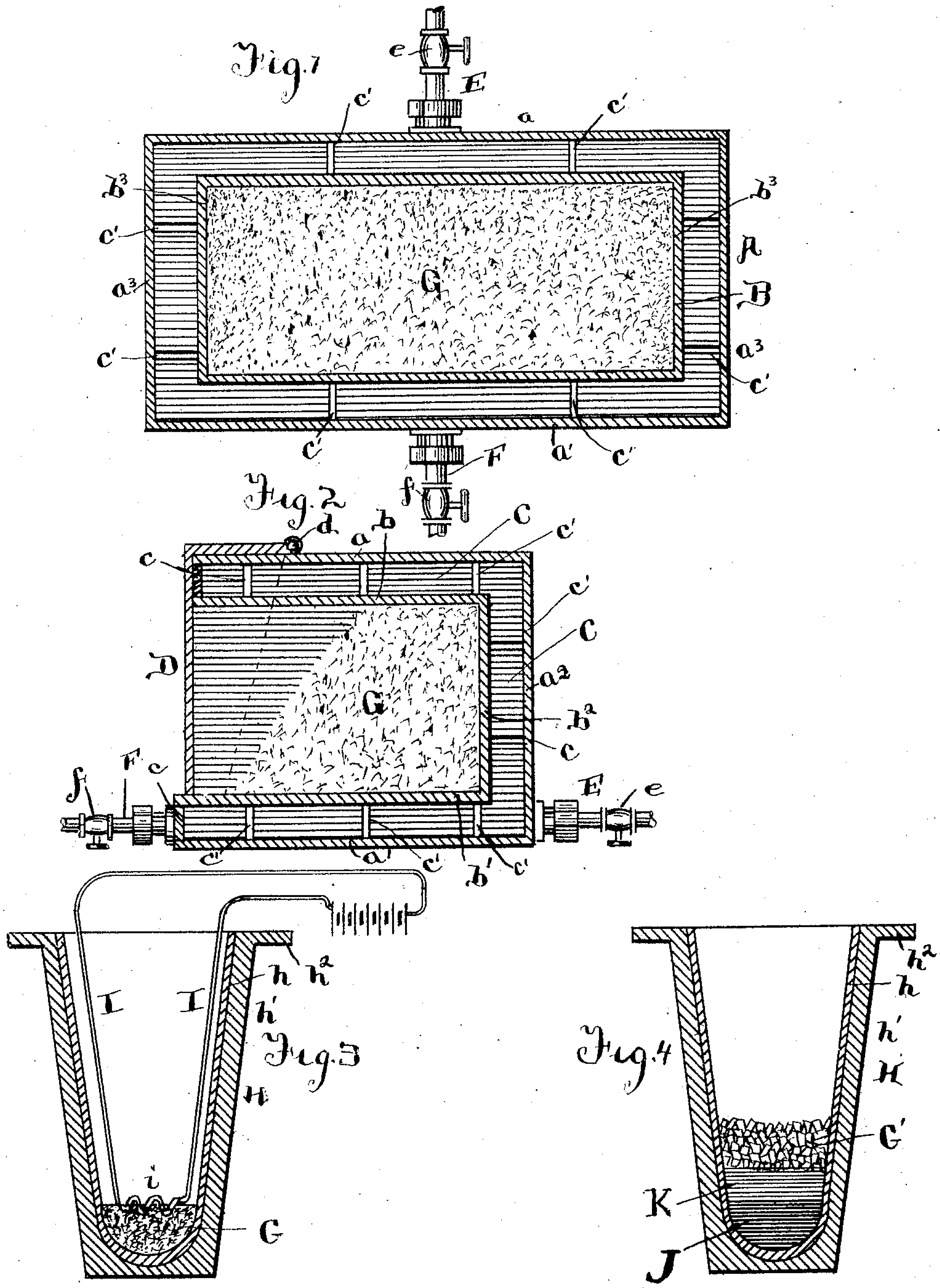

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UNITED STATES PATENT OFFICE.

FREDERICK C. WEBER, OF CHICAGO, ILLINOIS.

PROCESS OF REDUCING METALLIC OXIDS WITH METALLIC ALUMINIUM.

SPECIFICATION forming part of Letters Patent No. 778,345, dated December 27, 1904.

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To all whom it may concern:

Be it known that I, FREDERICK C. WEBER, a citizen of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented a new and useful Improvement in Processes of Reducing Metallic Oxids with Metallic Aluminium, of which the following is a specification.

The product derived from the reduction of metallic oxids and sulfids with metallic aluminium as the agent for reduction must be of such nature that it will be suitable for commercial and mechanical uses and purposes—that is, the carbonless metals, alloys, and borid and silicid compounds derived from the reduction of metallic oxids and sulfids by the employment of metallic aluminium as the reducing agent must be fairly pure and especially free from any appreciable amount of alloyed metallic aluminium. This result of fairly pure and perfect carbonless metals, alloys, and borid and silicid compounds is attained by having the carbonless metals, the metallic alloys, and the borid and silicid compounds homogeneous as well as pure, and in addition the borid and silicid compounds must also be crystalline. It is therefore essential that the aluminium present in the produced compounds, metals, and alloys must be in such fractional percentages only as to give the best results, for which purpose a proportion as low as one-fifth of one per cent. is desirable, especially in the making of low-carbon crucible steel or carbonless metals, for when adding alloys or compounds containing any appreciable amount of free aluminium the aluminium so introduced will act as a nascent atom and be a powerful chemical reagent, attacking the combined carbon of the steel and rendering it graphitic, which action is very objectionable. The aluminium, if present in appreciable amounts, will also combine with some of the free oxygen and the oxygen of oxids reducible by aluminium, which are always present in molten metal, forming corundum, which also becomes a source of contamination that must then be removed when cleansing and purifying molten metal.

The present process pertains more especially to overcoming these difficulties as found at present in the production of alloys, compounds, and metals when using metallic aluminium as the reducing agent, especially as to the alloys, metals, or compounds containing an appreciable amount of free aluminium; and to this end the process of the present invention consists in initially drying the material and then briqueting the dry and hot material for the charge as a preparation treatment before feeding the material of the charge into the crucible for final reduction.

In carrying out the process to obtain the best results the granuled or powdered materials composing the charges are to be pure and should be first carefully analyzed, so that the mixture is made accurate and correct as to the atomic proportions. The granuled or powdered materials of the charge are then thoroughly mixed together and placed in a hot box and subjected to heat until the materials become fully dehydrated, after which the hot dehydrated material of the charge is formed into briquets, using for the purpose any well-known briqueting-machine that will do the work, and when briqueted the materials for the charge are in the best shape possible for a quiet and uniform reaction by which practically all of the metallic aluminium of the charge undergoes oxidation and forms corundum. The pressure employed in briqueting the dehydrated material will insure a compressing of the oxids and aluminium firmly together, thus dispensing with the use of any binder other than the materials. This result is attained by reason of the aluminium particles being softer than the metallic-oxid particles, so that under the pressure employed in forming the materials into briquets the harder particles are forced into the softer ones, thus giving or producing the required binding effect by which the briqueted material will be held together. The briqueting of the dry and hot material of the charge in a dehydrated condition possesses the advantage of producing as a resultant carbonless metals showing only faint traces of free aluminium. Another advantage arising from the use of the material in the shape of briquets is concentrating the space in which the chemical reaction takes place, and thereby confining the heat derived from the reaction to such a degree that crystalline borid compounds are produced, even when using dry briqueted charges which are cold instead of hot, though the best results are obtained by using the dry briqueted charges in a hot condition, which is a great advantage, as crystalline borid compounds can only be produced by using very hot and dry charges if non-briqueted. The briqueting of the charge enables metallic silicids to be formed, thus demonstrating that the sum or volume of caloric produced by the alumino-thermic reaction is transformed as to intensity of a sufficient degree to create metallic silicids, which heretofore have only been created at the temperature of the electric arc, and the temperature of the electric arc is well known to be greater than that of the alumino-thermic reaction obtained by bringing about the reaction in the ordinary way—that is, by dry and either cold or hot material not briqueted. The briqueting of the hot dehydrated charges of the materials forms the material into a compressed shape, so that the charges can be inclosed in air-tight vessels and used elsewhere than near a source of steam-supply or other heating medium, and for inclosing in an air-tight vessel the hot dehydrated material is put in the vessel, which preferably can be an ordinary tin can having a sealing-cover, and after the vessel or can is filled with the material the cover is applied as usual in hermetically-sealing vessels or cans, the vessel or can preferably being warmed before putting therein the hot dehydrated material, thereby insuring dryness, and when inclosed in an air-tight vessel, so as to be out of contact with air, the briqueted charges will not absorb moisture hygroscopically, thus insuring a perfect and entirely quiet reaction free from any explosive violence, even while using the briquets cold. Caloric is one form of energy and is transformable from amount into intensity by diminishing the cubic space within which a given sum of caloric is developed, and such diminution of space is brought about by briqueting the materials. The concentrating of the space of the reaction transforms the sum of caloric, therefore, into intensity, and thereby furnishes a perfect resultant or product, which result is non-attainable in the case of the silicids when using dry and cold or dry and hot charges without briqueting.

An apparatus suitable for dehydrating the material before forming into briquets and for heating the briquets is shown in the drawings, and the manner of initially igniting the charge and adding the briquets in a crucible for reduction is also shown in the drawings, in which—

Figure 1 is a vertical longitudinal section of a hot box suitable for drying the material before briqueting and for heating the briquets, and Fig. 2 a vertical cross-section of the hot box, both Figs. 1 and 2 showing the material being dried before briqueting; Fig. 3, a vertical section of a crucible, showing the initial igniting of a charge for reducing the briquets; and Fig. 4, a vertical section of a crucible, showing the reduction of the material and the forming of the regulus and the corundum.

The hot box consists of an outer casing A, having a top wall a, a bottom wall a', a rear wall a^2, and end walls a^3, made of sheet steel or other suitable material of a strength sufficient to withstand a steam-pressure of from eighty to one hundred and seventy-five pounds. The outer casing surrounds an inner casing B, having a top wall b, a bottom wall b', a rear wall b^2, and end walls b^3, with the front of the casing open to enter the material into the chamber B′ of the inner casing. A heating-space C is formed on the top, bottom, rear, and ends between the two casings, and, as shown, the steam-space at the top and bottom on the front side is closed by a wall c, so as to confine the heat within the space. The inner casing, as shown, is sustained and held in position within the outer casing by cross-braces or tie-rods c' or in any other suitable manner. The open front of the casing is closed by a door D, hinged in the arrangement shown to the top of the outer casing by a suitable hinge d, and the door can be of the form shown or other suitable form, so as to insure the retention of the heat within the chamber B′ of the inner casing. A steam-supply pipe E leads into the heating-space C and is controlled by a valve e, and an exhaust-pipe F leads from the heating-space C and is controlled by a valve f when the heating medium used is steam. The granuled or powdered material G after being mixed is entered into the chamber B′ and steam or other heating medium is supplied to the heating-space C, and the material is subjected to heat until it is completely dehydrated, when it can be withdrawn and formed into briquets. The briquets when used hot can be heated in this same drying-box or in any other suitable manner.

The briqueted material is reduced in a crucible of any suitable construction. The crucible H shown has an inner lining h, of fireclay and magnesite, inclosed in a shell or case h', of iron or other suitable material, and, as shown, the iron shell or case has at its top a flange h^2, which can be used or not, as desired. A small amount of the granuled or powdered charge in its natural condition is placed in the bottom of the crucible, and this charge is ignited in the arrangement shown by means of an aluminum wire I, connected with a source of electric-current supply and having a coil i, which is surrounded by the granuled or powdered charge. The charge after its ignition has an additional amount of the granuled or powdered material supplied to the crucible, and such supply continues until a molten mass J is formed, comprising a regulus of molten metal at the bottom and molten corundum above the regulus of molten metal, and when a sufficient bath of molten corundum and molten regulus is formed in the crucible the briquets of the material are supplied thereto, the briquets sinking into the molten corundum and by chemical reaction continuing to form the regulus and corundum, and such supplying of the briquets can be proceeded with until the crucible is full of molten regulus and corundum. After the briqueted material composing the charge has been fed to the crucible and the reduction thereof therein completed, the crucible, which then contains the molten regulus and the corundum, is allowed to cool, and when cool the product, consisting of the metal alloy or compound and the corundum, will separate from the crucible through cooling and contracting and can be removed, and after removal the corundum can be separated from the resultant in the usual manner.

The preparatory treatment of heating and drying and then briqueting the dehydrated charge before the final reduction in a crucible avoids all danger and liability of explosion and enables a powdered or granuled aluminium and a metallic oxid to be effectually mixed and treated with all the moisture removed and through the preparatory heating, drying, and briqueting of the dehydrated charge before subjecting the charge to reaction enables a more perfect and uniform resultant to be obtained. The preparatory heating, drying, and briqueting to remove all the moisture can be successfully employed with metallic oxids and oxids of metalloids having one metal as a base and also with oxids having two metals as a base, and in both instances the result will be the reduction of the oxids, and this is also true with sulfids, as the chemico-physical law that each metal and chemical molecule has a definite latent heat of stability is effective and acts with metallic oxids and the oxids of metalloids so as to dissociate the component atoms and form new molecules which are true chemical molecules having definite composition and definite crystalline structure.

The heating, drying, and briqueting of the dehydrated charge results in the positive removal of all hygroscopic or ordinarily-present moisture from the charge, enabling an almost endless number of metals, metalloids, alloys, and compounds to be obtained, and it also results in making each charge a definite composition, with the charge in its entirety composed of the ingredients in atomic proportions, thus insuring an accurate and perfect reduction of the oxids, whether of metal or metalloids or whether having a base of one metal or a base of two metals, so that assurance is had that the reduction of the metallic oxids, sulfids, and metalloids will be perfect and under the best possible conditions for the production in a practical commercial way of the desired metals, alloys, and compounds.

What I regard as new, and desire to secure by Letters Patent, is—

1. The method of treating metallic oxids with aluminium in the production of metals and metalloids and alloys and their compounds, which consists in subjecting the to-be-treated charge, comprising aluminium in admixture with a metallic oxid reducible by aluminium, to an initial drying for removal of hygroscopic or ordinarily-present moisture thereby completely and absolutely dehydrating the material and then briqueting the dehydrated dry hot charge in a treatment preparatory to reduction of the charge, substantially as described.

2. The method of treating metallic oxids with aluminium in the production of metals and alloys, which consists in subjecting the to-be-treated charge, comprising aluminium in admixture with a metallic oxid reducible by aluminium, both the aluminium and metallic oxid in the condition of a fine powder or granule, to an initial drying by steam heat for removal of the hygroscopic or ordinarily-present moisture thereby completely and absolutely dehydrating the material and then briqueting the dehydrated dry hot charge as a treatment preparatory to reduction of the charge, substantially as described.

3. The method of treating metallic oxids with aluminium in the production of metals and metalloids, which consists in subjecting the to-be-treated charge, comprising aluminium in admixture with a metallic oxid reducible by aluminium, to a drying by heat for removing the hygroscopic or ordinarily-present moisture thereby completely and absolutely dehydrating the material and dissociating the oxid and sulfid molecules from the to-be-treated charge in one operation, and then briqueting the dehydrated hot dry charge as a treatment preparatory to reduction of the charge, substantially as described.

4. The method of treating metallic oxids with aluminium in the production of metals and metalloids, which consists in subjecting the to-be-treated charge, comprising aluminium in admixture with a metallic oxid reducible by aluminium, to a drying by heat for removing the hygroscopic or ordinarily-present moisture thereby completely and absolutely dehydrating the material and dissociating the oxid and sulfid molecules from the to-be-treated charge in one operation, then briqueting the dehydrated hot dry charge, and then hermetically sealing the briquets as a treatment preparatory to reduction of the charge, substantially as described.

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