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EXAMINER
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UNITED STATES PATENT OFFICE.

FREDERICK M. BECKET, OF NIAGARA FALLS, NEW YORK, ASSIGNOR TO ELECTRO METALLURGICAL COMPANY, A CORPORATION OF WEST VIRGINIA.

PROCESS OF PRODUCING MOLYBDENUM AND ITS ALLOYS.

No. 835,052. Specification of Letters Patent. Patented Nov. 6, 1906.

Application filed February 1, 1906. Serial No. 298,945.

To all whom it may concern:

Be it known that I, FREDERICK M. BECKET, a subject of the King of Great Britain, residing at Niagara Falls, in the county of Niagara and State of New York, have invented certain new and useful Improvements in Processes of Producing Molybdenum and Its Alloys, of which the following is a specification.

This invention relates to the production of fused molybdenum, low in carbon, directly from its sulfid ore, molybdenite, in a single operation.

The usual commercial method of producing molybdenum comprises two steps, molybdenite (MoS_2) being first roasted in an oxidizing atmosphere to an oxid or mixture of oxids of molybdenum, which is then subjected to the reducing action of carbon in an electric or other furnace. In order to produce a low-carbon metal by this method, it is necessary to use an amount of the reducing agent somewhat less than that theoretically requisite and to maintain the temperature below the fusion-point of the reduced molybdenum. The molybdenum thus obtained is in the undesirable form of powder and contains several per cent. of the oxids of molybdenum, chiefly the lower oxids—such as Mo_3O_8. When the metal is produced in the fused condition, the oxids are generally eliminated; but the molybdenum contains an objectionable proportion of carbon. The yield of molybdenum by this method is also low.

Another commercial method consists in reduction of molybdenum oxid by aluminium. A fused metal of high purity is thereby obtained; but the expense of the reducing agent is excessive.

I have found that substantially pure molybdenum may be produced in the fused condition by smelting in an electric furnace a mixture of molybdenite, carbon, and an oxid or other compound of an alkali or alkaline-earth metal, or by similarly treating a mixture of molybdenite and a carbid of an alkaline-earth metal, such as calcium carbid.

As typical of the process, the chief reaction which takes place when the mixture consists of molybdenum sulfid, lime, and carbon may be expressed by the following equation:

$$2MoS_2+2CaO+3C=2Mo+2CaS+CS_2+2CO. \quad (1)$$

and when calcium carbonate is used:

$$2MoS_2+2CaCO_3+5C=2Mo+2CaS+CS_2+6CO. \quad (2)$$

When calcium carbid is the reducing agent the reaction is as follows:

$$5MoS_2+2CaC_2=5Mo+2CaS+4CS_2. \quad (3)$$

It is not necessary to adhere strictly to the molecular proportions above represented, because the reaction

$$MoS_2+2CaO+2C=Mo+2CaS+2CO \quad (4)$$

may be made to take place to a limited extent, and the reaction

$$MoS_2+CaO+4C=Mo+CaC_2+CS_2+CO \quad (5)$$

to an extent proportional to the additional quantities of lime and carbon employed, provided the temperature is sufficient for the formation of calcium carbid; but I have found that, irrespective of the proportions of lime and carbon used, Equation 1 takes place, and for this reason any calcium carbid formed is invariably contaminated with calcium sulfid. The intermediate formation of calcium or other carbid is not essential to the operation of this process, and, in fact, I have produced thoroughly-fused molybdenum and obtained a high-percentage recovery of the metal according to the following reaction:

$$2MoS_2+2MgO+3C=2Mo+2MgS+CS_2+2CO. \quad (6)$$

Furthermore, when the charge treated is so proportioned as to obtain molybdenum metal and a carbid simultaneously the carbon content of the metal produced is greater than is the case when the charge is made in accordance with Equation 1. It is therefore preferable to proportion the mixture practically as represented in the typical equations, using the alkali or alkaline-earth oxids or carbonates, and especially the carbon, in but slight excess to insure a high yield of molybdenum.

The materials constituting the charge are mixed and then brought to such a temperature in an electric furnace as to not only cause the reaction to take place but to thoroughly fuse the reduced molybdenum and allow of its complete separation from the slag, which consists largely of an alkali or alkaline-earth metal sulfid. I prefer to use an electric furnace provided with two superposed tap-holes in order that metal or slag may be withdrawn as desired.

The carbon bisulfid liberated from the furnace may be collected as such or utilized as sulfur dioxid after oxidation by air.

This process possesses the important advantage that a fused molybdenum of low carbon content (not more than two-tenths of one per cent.) and free from molybdenum oxids may be produced from molybdenite in a single operation by the use of very cheap raw materials as reducing agents.

In the production of ferromolybdenum, molybdenum nickel, or other alloy of molybdenum I prefer to incorporate metallic iron, nickel, or other metal with the charge prepared for the reduction of the molybdenum, or to introduce the required proportions of these metals into the furnace at any time during the operation; but the metals—iron and nickel, for example—may be reduced from their sulfids or oxids simultaneously with the reduction of the molybdenum without interfering with the process.

The mixture containing an alkali or alkaline-earth metal and carbon, specified in certain claims, is to be understood as one in which the metal may be present either as an oxid, carbonate, or other salt, or as a carbid, the carbon being either free or combined as carbid.

I claim—

1. The process of reducing molybdenum, which consists in smelting a mixture of molybdenum sulfid and a desulfurizing agent, as set forth.

2. The process of reducing molybdenum, which consists in smelting a mixture containing molybdenum sulfid and a plurality of elements capable of uniting with sulfur, as set forth.

3. The process of reducing molybdenum, which consists in smelting a mixture containing molybdenum sulfid, an alkali or alkaline-earth metal and carbon, as set forth.

4. The process of reducing molybdenum, which consists in smelting a mixture containing molybdenum sulfid, an alkali or alkaline-earth-metal compound and carbon, as set forth.

5. The process of reducing molybdenum, which consists in smelting a mixture of molybdenum sulfid, an oyxgen compound of a metal having a relatively high affinity for sulfur and carbon, as set forth.

6. The process of reducing molybdenum, which consists in smelting a mixture of molybdenum sulfid, a calcium compound and carbon, as set forth.

7. The process of reducing molybdenum, which consists in smelting a mixture of molybdenum sulfid, calcium oxid and carbon, as set forth.

8. The process of producing molybdenum alloys, which consists in smelting a mixture of molybdenum sulfid, a desulfurizing agent and a source of the alloying metal, as set forth.

9. The process of producing molybdenum alloys, which consists in smelting a mixture containing molybdenum sulfid, a plurality of elements capable of uniting with sulfur, and a source of the alloying metal, as set forth.

10. The process of producing molybdenum alloys, which consists in smelting a mixture containing molybdenum sulfid, an alkali or alkaline-earth metal, carbon and a source of the alloying metal, as set forth.

11. The process of producing molybdenum alloys, which consists in smelting a mixture of molybdenum sulfid, an oxygen compound of a metal having a relatively high affinity for sulfur, carbon and a source of the alloying metal, as set forth.

12. The process of producing molybdenum alloys, which consists in smelting a mixture of molybdenum sulfid, calcium oxid, carbon and a source of the alloying metal, as set forth.

In testimony whereof I affix my signature in presence of two witnesses.

FREDERICK M. BECKET.

Witnesses:

R. L. MacDonald,

Chas. M. Saxe.