PATENT SPECIFICATION

Application Date: March 22, 1937. No. 8332/37. **492,606**
" " Nov. 8, 1937. No. 30564/37.
One Complete Specification Left: Feb. 2, 1938.
(Under Section 16 of the Patents and Designs Acts, 1907 to 1932).
Specification Accepted: Sept. 22, 1938.

PROVISIONAL SPECIFICATION
No. 8332 A.D. 1937.

Improvements in and relating to the Treatment of Materials containing Tantalum and/or Niobium

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, of 57 & 58, Lincoln's Inn Fields, London, W.C.2, a British subject, do hereby declare the nature of this invention (a communication to me from abroad by Societe Generale Metallurgique de Hoboken, a Belgian Limited Company, of Hoboken-lez-Anvers, Belgium), to be as follows:—

This invention relates to the treatment of materials, such as ores or metallurgical by-products, containing tantalum and/or niobium.

According to the present invention the primary material is subjected to a reducing operation in the presence of carbon, to reduce the impurities accompanying the tantalum and/or the niobium in the primary material, so as to leave:

1°) a slag rich in tantalum and/or niobium, practically free from the strange metals present as impurities in the primary material, and

2°) a ferro-alloy in which these impurities are being concentrated together with some tantalum and/or niobium.

The reducing operation may be carried out in any suitable metallurgical apparatus, e.g. in an electric furnace, with addition of carbon in such quantity as to reduce the impurities accompanying the tantalum and/or niobium in the primary material. Those impurities are being concentrated together with some tantalum and/or niobium reduced in an impure ferro-tantalum and/or ferro-niobium. If this alloy is not sufficiently carburized, it may be smelted in the presence of carbon in order to carburize as much as possible of the tantalum and/or the niobium contained.

The impure ferro-alloy obtained either directly or after the carburizing treatment just mentioned may be treated by chemical processes followed by mechanical separation in order to obtain the tantalum and/or niobium carbides free from impurities, according to the British Application No. 34,190/36 (Serial No. 485,021).

The slag rich in tantalum and/or niobium practically free from impurities, obtained by the original reducing operation, may be treated by any suitable known process for obtaining a commercial ferro-tantalum and/or ferro-niobium.

Dated the 22nd day of March, 1937.
MARKS & CLERK.

PROVISIONAL SPECIFICATION
No. 30564 A.D 1937

Improvements in and relating to the Treatment of Materials containing Tantalum and/or Niobium

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, of 57 & 58, Lincoln's Inn Fields, London, W.C.2, a British subject, do hereby declare the nature of this invention (a communication to me from abroad by Societe Generale Metallurgique de Hoboken, a Belgian Limited Company, of Hoboken-lez-Anvers, Belgium) to be as follows:—

This invention relates to the treatment of primary materials, such as ores or metallurgical by-products, containing tantalum and/or niobium, and is an improvement of the process described in Patent application No. 8332 of 22—3—1937.

According to the latter specification, the primary material is subjected to a reducing operation in the presence of carbon, to reduce the impurities accompanying the tantalum and/or niobium in the primary material so as to leave a slag rich in tantalum and/or niobium, and a ferro-alloy in which the impurities are

concentrated together with some tantalum and/or niobium.

It has now been ascertained that the said reducing operation may be advantageously carried out in the presence of carbon together with a carbide or carbides such as calcium or/and iron carbide.

If the raw material does not contain iron or does not contain sufficient iron for collecting the impurities during the reducing operation, iron or any other metal, such as cobalt, nickel, etc., should be added to the raw material before or during the treatment. These metals are being often used in special steels containing tantalum and/or niobium.

The impure ferro-alloy and the slag obtained by the present process may be treated as described in the main Patent application.

The reducing operation according to the present invention may be carried out in any suitable metallurgical apparatus, preferably in an electric furnace.

Dated this 8th day of November, 1937.
MARKS & CLERK.

COMPLETE SPECIFICATION

Improvements in and relating to the Treatment of Materials containing Tantalum and/or Niobium

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, of 57 & 58, Lincoln's Inn Fields, London, W.C.2, a British subject, do hereby declare the nature of this invention (a communication to me from abroad by Societe Generale Metallurgique de Hoboken, a Belgian Limited Company, of Hoboken-lez-Anvers, Belgium,) do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the treatment of primary materials, such as ores or metallurgical by-products, containing tantalum and/or niobium

The object of the invention is to separate the impurities from the tantalum and/or niobium in a comparatively simple and economical way.

According to the present invention the primary material is subjected to a reducing operation in the presence of slag-forming agents, of another metal such as iron or a compound thereof, and of an amount of free carbon which is sufficient to reduce the impurities accompanying the tantalum and/or niobium in the primary material, including the iron or other metal if in the form of a compound, but insufficient to appreciably reduce the tantalum and niobium oxides, so as to leave:

1—a slag rich in tantalum and/or niobium, practically free from the foreign metals present as impurities in the primary material, and

2—an alloy in which the impurities are concentrated with the said metal such as iron, and which may also contain some tantalum and/or niobium.

The reducing operation may be carried out in the presence of carbon or carbon with carbide or carbides, such as calcium or/and iron carbide.

The reducing operation may be carried out in any suitable metallurgical apparatus e.g., in an electric furnace with addition of carbon or carbon with carbide or carbides in such quantity as to reduce the impurities and the iron, or other metal, if present in the form of a compound, accompanying the tantalum and/or niobium in the primary material.

The impure alloy obtained either directly or after the carburizing treatment just mentioned may be treated by chemical processes followed by mechanical separation in order to obtain the tantalum and/or niobium carbides free from impurities.

The slag rich in tantalum and/or niobium practically free from impurities, obtained by the original reducing operation, may be treated by any suitable known process for obtaining a commercial ferro-tantalum and/or ferro-niobium.

If the raw material does not contain iron or does not contain sufficient iron for collecting the impurities, iron or another metal, such as a metal of the iron group (cobalt, nickel) should be added to the raw material before or during the treatment.

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concentrated together with some tantalum and/or niobium.

It has now been ascertained that the said reducing operation may be advantageously carried out in the presence of carbon together with a carbide or carbides such as calcium or/and iron carbide.

If the raw material does not contain iron or does not contain sufficient iron for collecting the impurities during the reducing operation, iron or any other metal, such as cobalt, nickel, etc., should be added to the raw material before or during the treatment. These metals are being often used in special steels containing tantalum and/or niobium.

The impure ferro-alloy and the slag obtained by the present process may be treated as described in the main Patent application.

The reducing operation according to the present invention may be carried out in any suitable metallurgical apparatus, preferably in an electric furnace.

Dated this 8th day of November, 1937.
MARKS & CLERK.

COMPLETE SPECIFICATION

Improvements in and relating to the Treatment of Materials containing Tantalum and/or Niobium

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, of 57 & 58, Lincoln's Inn Fields, London, W.C.2, a British subject, do hereby declare the nature of this invention (a communication to me from abroad by Societe Generale Metallurgique de Hoboken, a Belgian Limited Company, of Hoboken-lez-Anvers, Belgium,) do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the treatment of primary materials, such as ores or metallurgical by-products, containing tantalum and/or niobium

The object of the invention is to separate the impurities from the tantalum and/or niobium in a comparatively simple and economical way.

According to the present invention the primary material is subjected to a reducing operation in the presence of slag-forming agents, of another metal such as iron or a compound thereof, and of an amount of free carbon which is sufficient to reduce the impurities accompanying the tantalum and/or niobium in the primary material, including the iron or other metal if in the form of a compound, but insufficient to appreciably reduce the tantalum and niobium oxides, so as to leave:

1—a slag rich in tantalum and/or niobium, practically free from the foreign metals present as impurities in the primary material, and

2—an alloy in which the impurities are concentrated with the said metal such as iron, and which may also contain some tantalum and/or niobium.

The reducing operation may be carried out in the presence of carbon or carbon with carbide or carbides, such as calcium or/and iron carbide.

The reducing operation may be carried out in any suitable metallurgical apparatus e.g., in an electric furnace with addition of carbon or carbon with carbide or carbides in such quantity as to reduce the impurities and the iron, or other metal, if present in the form of a compound, accompanying the tantalum and/or niobium in the primary material.

The impure alloy obtained either directly or after the carburizing treatment just mentioned may be treated by chemical processes followed by mechanical separation in order to obtain the tantalum and/or niobium carbides free from impurities.

The slag rich in tantalum and/or niobium practically free from impurities, obtained by the original reducing operation, may be treated by any suitable known process for obtaining a commercial ferro-tantalum and/or ferro-nobium.

If the raw material does not contain iron or does not contain sufficient iron for collecting the impurities, iron or another metal, such as a metal of the iron group (cobalt, nickel) should be added to the raw material before or during the treatment.

EXAMPLE OF CARRYING THE INVENTION INTO EFFECT.

As a starting material a slag was used having the following composition:

$(Ta+Nb)_2O_5$ %	18,35
TiO_2	2,40
FeO	23,60
MnO	2,56
SnO_2	1,78
SiO_2	8,05
Al_2O_3	8,75
CaO	19,28
MgO	8,15

The charge consisted of:

The above slag	1000 kilos
Coke	79 kilos
	1079 kilos

The charge was treated in an open Heroult furnace.

653 kilos of refined slag were obtained containing:

$(Ta+Nb)_2O_5$ %	25,70
TiO_2	3,24
FeO	0,75
MnO	1,20
SnO_2	0.05
SiO_2	12,17
Al_2O_3	12,83
CaO	29,46
MgO	12,32

It has previously been proposed in a process of treatment of materials containing tantalum and/or niobium and another metal or metals such as iron, to subject the raw material to a reducing operation at a sufficiently high temperature to melt the mass and to reduce the reducible oxides of metals present in the raw material including those of the tantalum and/or niobium, with formation of a slag containing the major part of the silica and lime present in the raw material and of an alloy of tantalum and/or niobium with another metal or metals present in the raw material, and separating the slag thus formed from the said alloy, which latter is then subjected to a chemical treatment with a view to recovering the tantalum and/or niobium, preferably in oxide form.

It has also been proposed in a process of treatment of materials containing tantalum and/or niobium in the presence of at least one of the following metals: tin, manganese, iron, to remove those of the elements tin, manganese, iron, which are present in the material treated, prior to the extraction of tantalum and/or niobium, by a reducing operation in the presence of charcoal, which reducing operation is so carried out as to avoid a melting of the charge, the reduction being followed by an acid lixiviation.

It has further been proposed in a process for the treatment of materials containing tantalum and/or niobium oxides, to subject the primary material to a reducing operation in the presence of carbide of calcium in an electric furnace and in the presence of iron, in order to reduce the tantalum and niobium oxides and to obtain a ferro-alloy of Ta/Nb which may contain carbon.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for the treatment of materials containing tantalum and/or niobium, characterised in that the primary material is subjected to a reducing operation in the presence of slag-forming agents, of a metal such as iron or a compound thereof, and of an amount of free carbon which is sufficient to reduce the impurities accompanying the tantalum and/or the niobium in the primary material, including the iron or other metal if in the form of a compound, but is insufficient to appreciably reduce the tantalum and niobium oxides, so as to leave a slag rich in tantalum and/or niobium, practically free from the foreign metals present as impurities in the primary material, and an alloy in which these impurities are concentrated together with some tantalum and/or niobium and with the said metal, such as iron.

2. A process as claimed in claim 1, characterised in that the reducing operation is carried out in the presence of free carbon and calcium carbide, the amount of reducing material being such as to reduce the impurities alone.

3. A process as claimed in claim 1 or 2, characterised in that the reducing operation is carried out in the presence of free carbon and iron carbide, the amount of reducing material being such as to reduce the impurities alone.

4. A process as claimed in any of the preceding claims, characterised in that the reducing operation is carried out in an electric furnace.

5. A process as claimed in any of the preceding claims, characterised in that iron or another metal of the iron group is added to the raw material before or during the treatment.

6. The improved process for the treatment of materials containing tantalum and/or niobium, substantially as described.

Dated this 31st day of January, 1938.

MARKS & CLERK.

Leamington Spa: Printed for His Majesty's Stationery Office, by the Courier Press.—1938.