PATENT SPECIFICATION

Application Date: Dec. 12, 1936. No. 34190/36. **485,021**
" " March 19, 1937. No. 8103/37.
One Complete Specification Left: Sept. 22, 1937.
(Under Section 16 of the Patents and Designs Acts, 1907 to 1932).
Specification Accepted: May 13, 1938.

PROVISIONAL SPECIFICATION
No. 34190 A.D. 1936.

Improvements in or relating to the Production of Tantalum and/or Niobium Carbides

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, 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 Société Générale Metallurgique de Hoboken, a Belgian Company, of Hoboken-lez-Anvers, Belgium) to be as follows:—

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

According to the present invention the tantalum and/or niobium are separated from the raw material in the form of carbides.

The raw material is subjected to a smelting operation under reducing conditions, in the presence of a sufficient amount of iron and of an excess of carbon (coke, charcoal, etc.) for obtaining ferro-alloys in which the whole or the major part of the tantalum and/or niobium is concentrated in the form of carbides.

The smelting operation may be carried out in any suitable furnace, for instance in an electric arc furnace.

The ferro-alloy obtained is subjected to a chemical purification. After being finely subdivided by any suitable treatment, such as crushing or granulation, it is subjected to an attack by acids, such as sulphuric acid. The residue is separated from the liquor and is subjected to a mechanical treatment with a view to separating the heaviest particles therefrom. These particles are the carbide of tantalum and/or niobium.

Dated the 12th day of December, 1936.
MARKS & CLERK.

PROVISIONAL SPECIFICATION
No. 8103 A.D. 1937.

Improvements in or relating to the Production of Tantalum and/or Niobium Carbides

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 from abroad by Société Générale 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 tantalum and/or niobium are separated from the raw material in the form of carbides, by reducing the primary material without the addition of carbon, inside an electric furnace, an arc furnace for instance, the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft parts, are made of carbon bricks or are tamped with carbon.

If necessary, iron may be added in metal or oxide form to the charge so as to obtain by the reducing operation a ferro-alloy in which the whole or the major part of Ta and/or Nb is concentrated in the form of carbide.

If the raw material consists of a ferro-alloy, which does not contain carbon or contains only an insufficient amount of carbon, it is treated inside an electric furnace, an arc furnace for instance, in which the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft part of

which, are made of carbon bricks or are tamped with carbon.

According to a modification of the process, a raw material which consists of a ferro-alloy, which does not contain carbon or contains only an insufficient amount of carbon, is melted without addition of carbon inside a furnace of any type and is cast afterwards into ~~ladle~~ a ladle or other receptacle which is lined with carbon (bricks or tamped carbon).

The ferro-tantalum and/or ferro-niobium obtained as above, may be subsequently treated by crushing, leaching, and mechanical separation, as described for instance in provisional specification No. 34190 of 1936, for obtaining commercial carbide of tantalum and/or niobium.

Dated this 19th day of March, 1937.
MARKS & CLERK.

COMPLETE SPECIFICATION

Improvements in or relating to the Production of Tantalum and/or Niobium Carbides

I, WILLIAM WARREN TRIGGS, of the firm of Marks & Clerk, 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 Société Générale Metallurgique de Hoboken, a Belgian Limited Company, of Hoboken-lez-Anvers, Belgium) 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 production of carbides of tantalum and/or niobium.

According to the present invention an ore of Ta and/or Nb or a metallurgical by-product containing one or both of these elements is subjected to a reducing operation in the presence of a sufficient amount of iron and of an excess of carbon (coke, charcoal, or the like) to obtain an impure alloy containing iron and the whole or the major part of the tantalum and/or niobium concentrated in the form of carbides, and the impure alloy thus obtained is subjected to a chemical treatment with a view to separating the carbides of Ta and/or Nb therefrom.

The smelting operation may be carried out in any suitable furnace, for instance in an electric arc furnace.

The impure alloy (containing Si, Mn, Sn, and the like) obtained as said above may be finely subdivided by any suitable treatment, such as crushing or granulation, and subjected to an attack by a suitable acid, such as sulphuric acid, the residue being separated from the liquor and subjected to a mechanical treatment with a view to separating the heaviest particles therefrom. These particles are the carbide of tantalum and/or niobium.

According to another feature of the invention, the primary material is subjected to a reducing operation in the presence of iron, without the addition of carbon, inside an electric furnace, an arc furnace for instance, the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft parts, are made of carbon bricks or are tamped with carbon, so as to obtain by the reducing operation a ferro-alloy in which the whole or the major part of Ta and/or Nb is concentrated in the form of carbide, and which is treated for obtaining carbides of Ta and/or Nb. Alternatively, if the raw material consists of a ferro-alloy, which does not contain carbon or contains only an insufficient amount of carbon, it is treated inside an electric furnace, an arc furnace for instance, in which the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft part of which, are made of carbon bricks or are tamped with carbon.

According to a modification of the process, a raw material which consists of a ferro-alloy, which does not contain carbon or contains only an insufficient amount of carbon, is melted without addition of carbon inside a furnace of any type and is cast afterwards into a ladle or other receptacle which is lined with carbon (bricks or tamped carbon).

For the reducing operation, metal reducing agents, such as aluminium, magnesium, calcium, may be added to the charge.

The ferro-tantalum and/or ferro-niobium obtained as above, may be subsequently treated by crushing, leaching and mechanical separation, as described above, for obtaining commercial carbide of tantalum and/or niobium.

Example of treatment of a slag containing tantalum and niobium.

The slag treated had the following composition:

$(Ta + Nb)_2O_5$	20.05%
SiO_2	13.68
Al_2O_3	7.50
CaO	18.62
MgO	9.42
Fe	18.00
Mn	2.05
Sn	1.92

The charge had the following composition:

Slag	2.500 Kilos
Coke	325 ,,
Charcoal	100 ,,
	2.925 Kilos.

The charge was introduced by increments into an electric furnace of the Heroult type and treated at about 1600° C., thus obtaining on the bottom of the furnace 910 kilos of an impure alloy containing the iron and practically the whole of the Ta and Nb in suspension in the alloy in the form of carbides. 1146 kilos of a spent slag containing less than 1% of (Ta and $Nb)_2O_5$ were also obtained which slag was cast at intervals from the furnace.

The impure alloy was crushed, and treated with sulphuric acid at 18° Bé.

The residue was subjected to a mechanical separation for separating the heaviest particles thereof, consisting of carbides of Ta and/or Nb.

In a process of producing an alloy steel it has previously been proposed in British Patent Specification No. 457,760 to add tantalum or niobium to a high carbon iron containing at least 2% carbon whereby a suspension of the carbide of the added element is formed by reaction thereof with the carbon in the iron, and to separate the carbides thus produced by a chemical process such as an attack by hydrochloric acid.

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 production of carbides of tantalum and/or niobium characterized in that an ore of Ta and/or Nb or a metallurgical by-product containing one or both of these elements is subjected to a reducing operation in the presence of a sufficient amount of iron and of an excess of carbon (coke, charcoal, or the like) to obtain an impure alloy containing iron and the whole or the major part of the tantalum and/or niobium concentrated in the form of carbides, and the impure alloy thus obtained is subjected to a chemical treatment with a view to separating the carbides of Ta and/or Nb therefrom.

2. A process as claimed in claim 1, in which the impure alloy obtained is subjected to a chemical purification by means of an acid, such as sulphuric acid, the residue being subjected to a mechanical treatment with a view to separating the heaviest particles therefrom.

3. A modification of the process as claimed in claim 1 or 2, in which the primary material is reduced in the presence of iron, without the addition of carbon, inside an electric furnace, an arc furnace for instance, the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft parts, are made of carbon bricks or are tamped with carbon so as to obtain by the reducing operation an alloy in which the whole or the major part of Ta and/or Nb is concentrated in the form of carbide, and which is further treated for obtaining carbides of Ta and/or Nb.

4. A modification of the process as claimed in claim 1 or 2, in which the primary material which consists of a ferro-alloy, is melted without addition of carbon inside a furnace of any type and is cast afterwards into a ladle or other receptacle which is lined with carbon (bricks or tamped carbon).

Dated this 22nd day of September, 1937.

MARKS & CLERK.

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

$(Ta + Nb)_2O_5$	20.05%
SiO_2	13.68
Al_2O_3	7.50
CaO	18.62
MgO	9.42
Fe	18.00
Mn	2.05
Sn	1.92

The charge had the following composition:

Slag	2.500 Kilos
Coke	325 ,,
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The charge was introduced by increments into an electric furnace of the Heroult type and treated at about 1600° C., thus obtaining on the bottom of the furnace 910 kilos of an impure alloy containing the iron and practically the whole of the Ta and Nb in suspension in the alloy in the form of carbides. 1146 kilos of a spent slag containing less than 1% of $(Ta \text{ and } Nb)_2O_5$ were also obtained which slag was cast at intervals from the furnace.

The impure alloy was crushed, and treated with sulphuric acid at 18° Bé.

The residue was subjected to a mechanical separation for separating the heaviest particles thereof, consisting of carbides of Ta and/or Nb.

In a process of producing an alloy steel it has previously been proposed in British Patent Specification No. 457,760 to add tantalum or niobium to a high carbon iron containing at least 2% carbon whereby a suspension of the carbide of the added element is formed by reaction thereof with the carbon in the iron, and to separate the carbides thus produced by a chemical process such as an attack by hydrochloric acid.

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 production of carbides of tantalum and/or niobium characterized in that an ore of Ta and/or Nb or a metallurgical by-product containing one or both of these elements is subjected to a reducing operation in the presence of a sufficient amount of iron and of an excess of carbon (coke, charcoal, or the like) to obtain an impure alloy containing iron and the whole or the major part of the tantalum and/or niobium concentrated in the form of carbides, and the impure alloy thus obtained is subjected to a chemical treatment with a view to separating the carbides of Ta and/or Nb therefrom.

2. A process as claimed in claim 1, in which the impure alloy obtained is subjected to a chemical purification by means of an acid, such as sulphuric acid, the residue being subjected to a mechanical treatment with a view to separating the heaviest particles therefrom.

3. A modification of the process as claimed in claim 1 or 2, in which the primary material is reduced in the presence of iron, without the addition of carbon, inside an electric furnace, an arc furnace for instance, the crucible part and/or the shaft part of which, or in which only portions of the crucible and/or shaft parts, are made of carbon bricks or are tamped with carbon so as to obtain by the reducing operation an alloy in which the whole or the major part of Ta and/or Nb is concentrated in the form of carbide, and which is further treated for obtaining carbides of Ta and/or Nb.

4. A modification of the process as claimed in claim 1 or 2, in which the primary material which consists of a ferro-alloy, is melted without addition of carbon inside a furnace of any type and is cast afterwards into a ladle or other receptacle which is lined with carbon (bricks or tamped carbon).

Dated this 22nd day of September, 1937.

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