PATENT SPECIFICATION

Application Date: March 19, 1937. No. 8105/37. **486,693**

Complete Specification Left: Aug. 28, 1937.

Complete Specification Accepted: June 9, 1938.

PROVISIONAL 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) to be as follows:—

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

The object of the invention is to obtain the Ta and/or Nb in the form of alloys, for instance ferro-alloys, in a comparatively simple, economical and efficient way.

According to the present invention the primary material is subjected to a reducing operation by means of one or several of the metals aluminium, calcium, magnesium, and the slag obtained from the said reducing operation is subjected to a reducing operation in an electric furnace so as to obtain on the one hand a tantalum and/or niobium alloy and on the other hand a slag poor in tantalum and niobium.

The first reducing operation in the presence of one or more of the metals Al, Ca, Mg may be carried out without other external heat than that required for starting the reaction. The said first reducing operation is carried out in the presence of the metal with which it is desired to alloy the tantalum and/or niobium, for instance with iron. If the said metal is not present in a sufficient amount in the primary material, it is added in the metal or oxide form, so as to obtain the required tantalum and/or niobium alloy, for instance ferro-tantalum and/or ferro-niobium.

The second reducing operation, of the slag in an electric furnace, may be carried out in the presence of reducing agents, such as Ca, Al, Mg used either separately, or together, or by two.

The reducing metals, Ca, Al, Mg used in the first reducing operation or in the operation for the reduction of the slag in the electric furnace may be used in liquid or molten form.

Dated this 19th day of March, 1937.

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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, 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) 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 an improved process for the recovery of tantalum and/or niobium in metal or alloy form from their ores, or from metallurgical by-products such as those in which the said elements may be found in the form of oxide compounds, free or combined.

The invention consists in a process for the treatment of materials containing tantalum and/or niobium with a view to obtaining the tantalum and/or niobium in metal or alloy form by means of a reducing operation with Ca and/or Al and/or Mg, and is characterized in that the reducing operation is carried out in two phases, the first phase being carried out without external heat other than that required for starting the reaction so as to

obtain a slag comparatively rich in Ta and/or Nb, and the second phase being carried out in an electric furnace with the said slag comparatively rich in Ta and/or Nb and the reducing agents to obtain a slag poor in Ta and/or Nb.

In the first phase of the treatment a selective reduction of Nb relatively to Ta may be effected.

The reducing agents used during the second phase of the treatment may be alloys of Nb and/or Ta with Al and/or Ca and/or Mg. In this way it is possible to concentrate the Ta in a product comparatively poor in Nb; this tantaliferous product may also serve as the primary material for the manufacture of commercial tantaliferous products.

A metal such as iron may be present in or added to the charge if it is desired to obtain an alloy of the tantalum or niobium with such a metal.

Example of carrying the invention into effect:

A concentrate is treated having the following analysis:

Fe %	11.12		
Mn	7.17		
Sn	0.15		
TiO_2	4.13		
Ta_2O_5	37.12	or Ta %	30.48
Nb_2O_5	36.27	or Nb %	25.39
Al_2O_5	2.22		
CaO + MgO	0.09		
SiO_2	2.31		

$$\text{Ratio } \frac{Nb}{Ta} = 0.83$$

Composition of the charge:

Concentrate	100 kilos
Aluminium	26 ,,
Iron	13 ,,
	139 kilos.

The charge is treated without other external heat than that required for starting the reaction. The products obtained are:

Alloy	68 K. 750
Slag	77 K. 600

Analysis of the alloy:

Fe % 36.12, Mn 4.02, Sn 0.25, Al 0.51, Si 0.83, Ti 0.13, Ta 21.74, Nb 34.53, C 0.04.

$$\text{Ratio } \frac{Nb}{Ta} = 1.58.$$

Analysis of the slag:

Ta_2O_5 % 21.98, (or Ta % 18.05), Nb_2O_5 2.96 (or Nb % 2.07), TiO_2 3.27, Al_2O_3 63.38, CaO + MgO 0.23, SiO_2 1.06, MnO 5.87, FeO 0.34.

$$\text{Ratio } \frac{Nb}{Ta} = 0.11.$$

The above slag is treated in an electric furnace, with application of external heat during the entire reaction:

Composition of the charge:

Slag	1000 kilos
Aluminium	102 ,,
Lime	170 ,,
Fluorspar	50 ,,
Iron	100 ,,
	1422 kilos.

The products obtained are: Alloy 340 kilos and slag 928 kilos.

Analysis of the alloy

Ta % 49.32, Nb 4.85, Fe 28.35, Mn 11.65, Ti 2.82, Al 1.45, Si 0.67, C 0.14.

$$\text{Ratio } \frac{Nb}{Ta} = 0.1.$$

Analysis of the slag:

Ta_2O_5 % 0.72, Nb_2O_5 0.30, TiO_2 1.68, SiO_2 0.53, CaO 15.27, MgO 14.91, Al_2O_3 65.40.

In our Patent No. 467,484 we have proposed broadly to treat a primary material containing tantalum and niobium with an amount of Ca and/or Al or an alloy of tantalum and/or niobium with Ca and Al, which is insufficient completely to reduce both the Nb and Ta, whereby an alloy is obtained having a higher niobium/tantalum ratio than the primary material and a slag having a higher tantalum/niobium ratio than the primary material, which slag may be subjected to a separate treatment in an electric furnace with Ca and/or Al for the recovery of Ta and Nb.

According to the present invention, a slag comparatively rich in Ta and/or Nb is obtained in a first phase which is carried out without external heat other than that required for starting the reaction, the said slag thus obtained being treated in an electric furnace with the reducing agents for obtaining a slag poor in Ta and/or Nb.

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 with a view to obtaining the tantalum and/or niobium in metal or alloy form by means of a reducing operation with Ca and/or Al and/or Mg, characterized in that the reducing operation is carried out in two phases, the first phase being carried out without external heat other than that required for starting the reaction so as to obtain a slag comparatively rich in Ta and/or Nb, and the second phase being carried out in an

obtain a slag comparatively rich in Ta and/or Nb, and the second phase being carried out in an electric furnace with the said slag comparatively rich in Ta and/or Nb and the reducing agents to obtain a slag poor in Ta and/or Nb.

In the first phase of the treatment a selective reduction of Nb relatively to Ta may be effected.

The reducing agents used during the second phase of the treatment may be alloys of Nb and/or Ta with Al and/or Ca and/or Mg. In this way it is possible to concentrate the Ta in a product comparatively poor in Nb; this tantaliferous product may also serve as the primary material for the manufacture of commercial tantaliferous products.

A metal such as iron may be present in or added to the charge if it is desired to obtain an alloy of the tantalum or niobium with such a metal.

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A concentrate is treated having the following analysis:

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Analysis of the slag:

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The above slag is treated in an electric furnace, with application of external heat during the entire reaction:

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	1422 kilos.

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According to the present invention, a slag comparatively rich in Ta and/or Nb is obtained in a first phase which is carried out without external heat other than that required for starting the reaction, the said slag thus obtained being treated in an electric furnace with the reducing agents for obtaining a slag poor in Ta and/or Nb.

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 with a view to obtaining the tantalum and/or niobium in metal or alloy form by means of a reducing operation with Ca and/or Al and/or Mg, characterized in that the reducing operation is carried out in two phases, the first phase being carried out without external heat other than that required for starting the reaction so as to obtain a slag comparatively rich in Ta and/or Nb, and the second phase being carried out in an

electric furnace with the said slag comparatively rich in Ta and/or Nb and the reducing agents to obtain a slag poor in Ta and/or Nb.

2. A process as claimed in claim 1, in which the reducing agents in the second phase are alloys of Nb and/or Ta with Al and/or Ca and/or Mg.

3. A process as claimed in claim 1, in which during the first phase of the treatment, a selective reduction of the Nb relatively to Ta is effected.

4. The improved process for treating materials containing tantalum and/or niobium substantially as hereinbefore described.

Dated this 28th day of August, 1937.

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

