PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION

Improvements in and relating to the Recovery of Tantalum and 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 Société Generale Metallurgique de Hoboken, a Belgian Company, of Hoboken-lez-Anvers, Belgium), to be as follows:—

This invention relates to an improved process for the treatment of primary materials, ores or metallurgical by-products containing tantalum and niobium and which may contain other elements, such as titanium, and in which the various metals may be present in an oxidized form.

According to one characteristic feature of the present invention, an alloy niobium-tantalum is separated from the primary material by a reducing operation, in which alloy the ratio Nb/Ta is higher than that present in the primary material.

According to another characteristic feature of the invention, the reducing operation is carried out in the presence of calcium or calcium and aluminium, or of an alloy of tantalum or/and niobium with calcium or calcium and aluminium.

The addition of reducing agent allows of obtaining an alloy Nb—Ta containing a higher proportion of niobium relatively to tantalum than that contained in the charge, or of separating the major part of the niobium contained in the charge, whilst the major part of the tantalum remains in the slag. The latter may be subjected to a separate treatment with calcium or aluminium, or to any other known treatment, for the recovery of the tantalum.

The amount of reducing agent which should be added for the selective separation of niobium from the charge is to be determined in each particular case according to the nature of the charge which is treated and to the nature of the reducing agent (Ca or Ca and Al or/and their alloys). It also varies according to the contents in Nb of the raw material treated.

For a given raw material and a given reducing agent, there is an optimum amount of reducing agent to be added for obtaining the highest ratio Nb/Ta.

If the said amount is increased or decreased, the ratio Nb/Ta in the final product is diminished.

The reducing operation may be effected in any suitable apparatus, for instance in an electric furnace such as an induction furnace, arc furnace, radiating furnace, or other electric furnace, or in a crucible furnace, or in a flame furnace, etc. The furnaces used may be of the stationary type, of a tilting or rotary or oscillating type.

According to another characteristic feature of the invention, a metal or metals is or are added to the charge, so as to obtain an alloy of niobium and tantalum with this other metal or metals.

For instance iron may be added in an oxide form either before or during the reducing operation. Iron may also be added in the metal form at any stage of the treatment (for instance after the reducing operation) for producing an iron alloy with niobium, tantalum, etc. The reducing operation may also be carried out on a bath of molten metal (for instance iron) or alloy. The tantalum, niobium, and also titanium if present, may thus be obtained in a concentrated form as an iron alloy.

The slag rich in tantalum may be subjected to a concentrating treatment with a reducing metal or alloy, preferably with calcium or/and aluminium, or with an alloy of tantalum with calcium or/and aluminium.

Dated this 13th day of September, 1935.

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COMPLETE SPECIFICATION

Improvements in and relating to the Recovery of Tantalum and 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 Société Generale Metallurgique de Hoboken, a Belgian 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 treatment of primary materials, ores or metallurgical by-products containing tantalum and niobium in oxide form and which may contain other elements, such as titanium in oxide form.

According to the present invention, the primary material is reduced with an amount of calcium which is insufficient completely to reduce both the niobium and the tantalum 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.

The reducing operation may be carried out by means of calcium and aluminium conjointly. The reducing operation may also be carried out by means of an alloy of tantalum or/and niobium with calcium, or with calcium and aluminium.

The alloy rich in niobium which is obtained according to the present invention may be treated separately to eliminate the Ca and/or Al which have been drawn into the alloy during the reducing operation.

The slag may be subjected to a separate treatment with calcium and/or aluminium, or to any other known treatment, for the recovery of the tantalum and of any niobium.

The amount of reducing agent which should be added for the selective separation of niobium from the charge is to be determined in each particular case according to the nature of the charge which is treated and to the nature of the reducing agent (Ca or Ca and Al or/and their alloys). It also varies according to the contents in Nb of the raw material treated. The said amount of reducing agent is preferably at least equal to the amount which is theoretically necessary for fixing all the oxygen of the niobium oxide. The excess to be added for obtaining a suitable reduction depends on several factors:

a) the nature of the furnace which is employed and the atmosphere of which may be more or less reducing, may cause losses of the reducing agent by the oxidation of gases;

b) the temperature of the bath. The selective reduction depends not only on the difference of affinity of the various metals for oxygen, but also on the temperature at which the reduction is being carried out.

c) the melting point of the alloy produced. If the latter is in the molten state the yield of the reducing agent may be better than if the alloy produced is a semi-fused state, owing to secondary reactions which may take place.

The reducing operation may be effected with external heat, in a crucible or in any suitable apparatus, for instance in an electric furnace such as an induction furnace, arc furnace, or radiating furnace, or in a crucible furnace, or in a flame furnace. The furnaces used may be of the stationary type, or of tilting or rotary or oscillating type.

A metal or metals may be added to the charge, so as to obtain an alloy of niobium and tantalum with this other metal or metals.

For instance iron may be added in the form of oxide either before or during the reducing operation. Iron may also be added in the metallic form at any stage of the treatment (for instance after the reducing operation) for producing an iron alloy with niobium and tantalum. The reducing operation may also be carried out in a bath of molten metal (for instance iron) or alloy. The tantalum, niobium, and also titanium if present, may thus be obtained in a concentrated form as an iron alloy.

The slag rich in tantalum obtained by the reducing operation may be subjected to a concentrating treatment with a reducing metal or alloy, preferably with calcium or/and aluminium, or with an alloy of tantalum with calcium or/and aluminium.

It has previously been proposed to obtain powders of metals, the oxides of which cannot be reduced to metal by means of hydrogen, for example the oxides of tantalum, chromium, titanium, or any other similarly difficultly reducible oxide, by well known metallurgical processes, one of which involved the reduction of the oxides by means of calcium.

EXAMPLES OF CARRYING THE INVENTION INTO EFFECT.

I. A concentrate was treated having the following composition:

Al_2O_3	4.36%
MgO	traces
CaO	traces
SiO_2	3.25
Fe	9.04
Mn	7.50
TiO_2	3.50
Ta_2O_5	29.78 (Ta 24.45)
Nb_2O_5	35.42 (Nb 24.80)

The reducing operation was carried out in an electric furnace of the Heroult type, the charge being as follows:

Concentrate	1000 kilos
Calcium	382 kilos
Bauxite	100 kilos
Fluorspar	30 kilos
	1512 kilos

The additions of bauxite and fluorspar were made in order to obtain a more fluid slag.

The bauxite used had the following composition:

Al_2O_3	58.00%
SiO_2	17.65
TiO_2	2.50
Fe_2O_3	2.64
Loss by ignition	19.00

After the reducing operation 390 kilos were obtained of an alloy containing:

Ta	23.16%
Nb	35.75
Fe	21.03
Mn	17.22
Si	0.80

Also 1030 kilos of slag were produced containing:

CaO	45.38%
MgO	7.09
Al_2O_3	8.95
SiO_2	3.73
TiO_2	3.23
Ta_2O_5	16.37
Nb_2O_5	13.84

This slag was retreated for recovering the Ta and Nb, the new charge being as follows:

Slag	1030 kilos
Calcium	250 kilos
Bauxite	500 kilos
Fluorspar	60 kilos
Iron	130 kilos
	1970 kilos

369 kilos of the following alloy were obtained:

Ta	35.13%
Nb	26.02
Fe	32.67
Ti	3.91
Si	2.75
Ca	0.84

Also 1258 kilos of slag were obtained of the following composition:

CaO	61.77%
MgO	7.15
Al_2O_3	30.19
SiO_2	0.24
TiO_2	0.49
Ta_2O_5	0.37
Nb_2O_5	0.25

II. A concentrate was treated of the same composition as in the example I in a Heroult electric furnace with a Ca—Al alloy containing 25% Ca and 75% Al.

The charge contained:

Concentrate	1000 kilos
Ca—Al alloy	196 kilos
	1196 kilos

395 kilos of the following alloy were produced:

Ta	19.23%
Nb	39.76
Fe	20.98
Mn	18.00
Si	0.31
Al	0.24

750 kilos of slag were also produced containing:

Al_2O_3	33.91%
SiO_2	4.00
CaO	7.84
MgO	8.03
Ta_2O_5	25.97
Nb_2O_5	15.26
TiO_2	3.80

The slag was retreated to recover the Ta and Nb, the new charge being as follows:

Slag	750 kilos
Ca—Al alloy	124 kilos
Iron	150 kilos
Fluorspar	35 kilos
	1059 kilos

635 kilos of slag were produced containing:

Al_2O_3	63.87%
SiO_2	0.38
CaO	14.39
MgO	17.60
Ta_2O_5	0.59
Nb_2O_5	0.24
TiO_2	0.94

383 kilos of the following alloy were produced:

Ta	36.52%
Nb	20.29
Fe	37.35
Si	1.53
Al	1.07

In my co-pending application No. 25433/35 (Serial No. 467,483) I have described a process for the joint recovery of tantalum and niobium from ores containing the same, or from metallurgical by-products in which the said elements are found in the form of oxide compounds, the characteristic feature of said process being that tantalum and niobium contained in the primary material are completely, or substantially completely, reduced by means of calcium and aluminium conjointly, whilst applying external heat.

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 primary materials, ores or metallurgical by-products containing tantalum and niobium in oxide form and which may contain other elements, such as titanium in oxide form, characterised in that the primary material is reduced with an amount of calcium which is insufficient completely to reduce both the niobium and the tantalum 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.

2. A process as claimed in claim 1, in which the reducing operation is effected by means of calcium and aluminium conjointly in quantity insufficient to effect complete reduction.

3. A process as claimed in claim 1 or 2, in which the reducing operation is carried out by means of an alloy of tantalum or/and niobium with calcium, or with calcium and aluminium in quantity insufficient to effect complete reduction.

4. A process as claimed in claim 1, 2 or 3, in which the reducing operation is carried out in the presence of iron, for the purpose set forth.

5. A process as claimed in claim 1, 2, 3 or 4, characterised in that the slag obtained by the reducing operation is subjected to a concentrating treatment with calcium or/and aluminium, or with an alloy of tantalum with calcium or/and aluminium.

6. A process as claimed in any of the previous claims characterised in that the treatment is carried out in an electric furnace.

Dated this 10th day of September, 1936.

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to be performed, I declare that what I claim is:—

1. A process for the treatment of primary materials, ores or metallurgical by-products containing tantalum and niobium in oxide form and which may contain other elements, such as titanium in oxide form, characterised in that the primary material is reduced with an amount of calcium which is insufficient completely to reduce both the niobium and the tantalum 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.

2. A process as claimed in claim 1, in which the reducing operation is effected by means of calcium and aluminium conjointly in quantity insufficient to effect complete reduction.

3. A process as claimed in claim 1 or 2, in which the reducing operation is carried out by means of an alloy of tantalum or/and niobium with calcium, or with calcium and aluminium in quantity insufficient to effect complete reduction.

4. A process as claimed in claim 1, 2 or 3, in which the reducing operation is carried out in the presence of iron, for the purpose set forth.

5. A process as claimed in claim 1, 2, 3 or 4, characterised in that the slag obtained by the reducing operation is subjected to a concentrating treatment with calcium or/and aluminium, or with an alloy of tantalum with calcium or/and aluminium.

6. A process as claimed in any of the previous claims characterised in that the treatment is carried out in an electric furnace.

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