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PRODUCING COLUMBIUM ALLOYS

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The invention is a process for producing alloys of columbium from materials which contain oxides of columbium.

The important ores of columbium, for example columbite ores, frequently contain substantial amounts of tantalum, titanium, and other metals. The separation and purification of columbium from its ores by known chemical procedures is difficult and expensive. This is especially true when a separation of the columbium from tantalum or titanium, or both, is required.

It is an object of the invention to provide a simple and efficient process for producing low carbon alloys of columbium from ores and oxydic compounds of columbium.

It is a further object of the invention to provide a process for reducing ores and oxydic compounds of columbium selectively in respect to tantalum and titanium, so that the ratio of columbium to tantalum and titanium is materially higher in the alloy product than in the raw material.

The invention comprises, broadly, reducing oxydic ores or compounds of columbium with silicon or silicon alloys, the silicon preferably being so proportioned to the charge that a selective reduction of columbium compounds in respect to compounds of tantalum and titanium occurs.

I have found that in order to obtain an optimum reduction and recovery of columbium consistent with a strongly selective reduction of columbium compounds in preference to tantalum and titanium, it is necessary so to proportion the silicon in the charge that there is enough silicon to yield an alloy containing a columbium to silicon ratio between about 6 to 1 and about 12 to 1. Preferably, this ratio is between 7 to 1 and 9 to 1.

If the optimum proportion of silicon is used, it is possible to recover as metal upwards of 75% of the columbium originally present in the charge, and also to produce an alloy in which the ratios of columbium to tantalum and of columbium to titanium are considerably higher than in the ore or compound smelted. If greater proportions of silicon than the optimum are used, the resulting alloy will contain greater proportions of tantalum and titanium; and if smaller proportions of silicon are used, the recovery of columbium will be poorer.

The process of the invention is suitably effected in an electric furnace. Basic slags are preferably used; but neutral or acidic slags may be used with some success under certain conditions.

The silicon reducing agent is suitably either silicon metal or a silicon alloy. Ferrosilicon, chromium-silicon, ferrochromium-silicon, or columbium-silicon, and, in general, any alloy of silicon with metals desired or unobjectionable in the final product, may be used for the reducing agent.

The quantity of silicon required for the reduction of the ore or compound of columbium may be estimated roughly from the theoretical requirements for the reduction of the metal oxides and oxydic compounds contained therein. An allowance should be made for the amount of silicon oxidized by the oxygen contained in the furnace atmosphere, and of course, for the silicon desired in the final alloy. The precise proportions to be used under any given set of conditions may be determined by trial.

Iron, or nickel, or chromium, or any other metal, or any mixture of metals, may be added to the product of reduction, to produce an alloy of the desired composition, either by effecting the reduction in the presence of a metal bath, or by adding one or more of said metals during or after the reduction.

Example No. 1

As a specific example of the process: A charge containing

	Pounds
Columbite	1,000
Silicon metal (97% Si)	243
Lime	1,625
Quartzite	25
Ferrocolumbium scrap	375
	3,268

was smelted in an electric arc furnace. The columbite used contained by analysis:

	Percent
Cb_2O_5	55.66
Ta_2O_5	5.77
TiO_2	5.32
Fe_3O_4	23.27
MnO	2.10

Cb=38.96%
Ta=4.73%
Ti=3.19%

Ratio Cb:Ta=8:1
Ratio Cb:Ti=12:1

The ferrocolumbium scrap was a mixture of metal and slag containing approximately 13.9% Cb.

The smelting yielded 461½ pounds of ferrocolumbium analyzing:

	Per cent
Cb	55.57
Ta	1.76
Fe	32.61
Si	6.68
Ti	0.15
Mn	1.82
C	0.32
	98.91

Ratio Cb:Ta=32:1
Ratio Cb:Ti=370:1
Ratio Cb:Si=8.3:1

The separation of clean metal from slag also produced 772 pounds of rather lean scrap containing approximately 10.5% Cb. Of the columbium fed in the charge 76.4% was recovered in the clean alloy and scrap.

The slag analyzed 5.20% Cb_2O_5, 0.90% Ta_2O_5, and 2.46% TiO_2, or a ratio of columbium to tantalum of 5 and of columbium to titanium of 2.5.

Example No. 2

In another example a larger proportion of silicon was employed and a poorer selectivity of reduction of columbium over titanium and tantalum was obtained. The following charge was smelted in an electric arc furnace:

	Per cent
Columbite	1,234
Silicon metal (97% Si)	527
Lime	2,380
Silica sand	15
Ferrocolumbium scrap	94
	4,250

The columbite contained by analysis:

	Per cent
Cb_2O_5	54.80
Ta_2O_5	5.36
TiO_2	6.90
FeO	3.61
Fe_3O_4	18.09
MnO	3.04

Cb=38.36%
Ta=4.39%
Ti=4.14%

Ratio Cb:Ta=9:1
Ratio Cb:Ti=9:1

The ferrocolumbium scrap contained approximately 54% columbium.

The smelting yielded 749 pounds of alloy analyzing:

	Per cent
Cb	54.87
Ta	3.11
Ti	0.75
Si	11.65
C	0.62

Balance largely iron

Ratio Cb:Ta=18:1
Ratio Cb:Ti=73:1
Ratio Cb:Si=4.7:1

The separation of clean metal from slag also produced 153 pounds of metallic scrap containing approximately 39% columbium. Of the columbium fed in the charge 90.2% was recovered in the metallic products.

The slag analyzed 1.32% Cb_2O_5, 0.26% Ta_2O_5, and 1.68% TiO_2, or a ratio of columbium to tantalum of 4 and of columbium to titanium of less than 1.

It will be observed that in this example, where an alloy with a ratio of columbium to silicon of 4.7 was produced, the alloy contains much larger proportions of both tantalum and titanium than was the case in Example No. 1 where an alloy of lower silicon content having a ratio of columbium to silicon of 8.3 was made.

Example No. 3

Below is given an example of the production of ferrocolumbium, in which a columbium-silicon alloy was used as reducing agent. The following charge was smelted in an electric arc furnace:

	Pounds
Columbite Lot #1	300
Columbite Lot #2	600
Columbium-Silicon	725
Lime	750

Below are given analyses of these materials:

	Columbite lot No. 1	Columbite lot No. 2
	Percent	*Percent*
Cb_2O_5	60.36	24.97
Ta_2O_5	6.43	1.75
Fe_3O_4	18.84	33.30
TiO_2	5.17	29.84

Columbium-silicon analysis	Percent
Cb	34.59
Ta	16.58
Ti	13.20
Fe	5.14
Si	21.00

The run yielded 647.5 pounds of ferrocolumbium alloy of the following analysis:

	Percent
Columbium	50.53
Tantalum	5.36
Titanium	0.75
Silicon	5.67
Carbon	0.87

Ratio Cb:Ta=approximately 10:1
Ratio Cb:Ti=approximately 73:1
Ratio Cb:Si=approximately 9.7:1

This corresponds to a recovery of columbium of 70.5% in the clean alloy, and 2.9% additional was obtained in the small amount of scrap resulting from the run. In addition, 1,988 pounds of slag were obtained which contained columbium equal to 26.2% of that originally carried in the raw materials.

The slags obtained from the above-described process step usually contain substantial amounts of columbium, tantalum, and titanium. It will sometimes be desired to recover these metals from the slags, and this may be done by smelting the slags with sufficient silicon to yield a high-silicon columbium-silicon alloy, which product may then be used as the reducing agent to produce the lower-silicon alloy as described above.

The process is generally applicable to the smelting of columbium ores and compounds containing widely varying ratios of columbium to tantalum and to titanium, and while the recovery of columbium will be in some degree dependent on the richness of the ore or compound in columbium it will be found that maximum recovery of

columbium, accompanied by a high selectivity of reduction of columbium over tantalum and titanium, will be obtained when the silicon-containing reducing agent is proportioned to produce an alloy containing an approximate ratio of columbium to silicon between about 7 and 9 to 1.

All proportions mentioned herein and in the appended claims are proportions by weight.

I claim:

1. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of selectively reducing the said columbium oxide in preference to the tantalum oxide by reacting said material with silicon.

2. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of selectively reducing the said columbium oxide in preference to the tantalum oxide by reacting said material with a silicon alloy.

3. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of selectively reducing the said columbium oxide in preference to the tantalum oxide by reacting said material with a columbium-silicon alloy.

4. Process for producing alloys of columbium from material containing oxides of columbium, tantalum, and titanium, which comprises the step of reacting said material with silicon, whereby the columbium oxide is selectively reduced in preference to the oxides of tantalum and titanium.

5. Process for producing alloys of columbium from material containing oxides of columbium, tantalum, and titanium, which comprises the step of reacting said material with a silicon alloy, whereby the columbium oxide is selectively reduced in preference to the oxides of tantalum and titanium.

6. Process for producing alloys of columbium from material containing oxides of columbium, tantalum, and titanium, which comprises the step of reacting said material with a columbium-silicon alloy, whereby the columbium oxide is selectively reduced in preference to the oxides of tantalum and titanium.

7. Process for partially separating columbium from tantalum which comprises the step of selectively reducing with silicon a mixture of oxides of columbium and tantalum.

8. Process for partially separating columbium from tantalum and titanium which comprises the step of selectively reducing with silicon a mixture of oxides of columbium, tantalum, and titanium.

9. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of reacting said material with silicon sufficient to reduce a preponderant proportion of the columbium oxide but insufficient to reduce as great a proportion of the tantalum oxide as of the columbium oxide.

10. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of reacting said material with silicon in the form of a silicon alloy sufficient to reduce a preponderant proportion of the columbium oxide but insufficient to reduce as great a proportion of the tantalum oxide as of the columbium oxide.

11. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of reacting said material with silicon in the form of a columbium-silicon alloy sufficient to reduce a preponderant proportion of the columbium oxide but insufficient to reduce as great a proportion of the tantalum oxide as of the columbium oxide.

12. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of reacting said material with sufficient silicon to yield an alloy in which the ratio of columbium to silicon is between about 6 to 1 and about 12 to 1, whereby a preferential reduction of columbium oxide is obtained.

13. Process for producing alloys of columbium from material containing oxides of columbium, titanium, and tantalum, which comprises the step of reacting said material with sufficient silicon in the form of a columbium-silicon alloy to yield an alloy in which the ratio of columbium to silicon is between about 6 to 1 and about 12 to 1, whereby a preferential reduction of columbium oxide is obtained.

14. Process for producing alloys of columbium from material containing oxides of columbium which comprises reacting said material with sufficient silicon to yield an alloy in which the ratio of columbium to silicon is between about 6 to 1 and about 12 to 1, whereby a slag containing columbium oxide is obtained; reacting said slag with sufficient silicon to yield a high-silicon columbium alloy in which the ratio of columbium to silicon is less than about 6 to 1; and reducing a second lot of columbium oxide containing material with this high-silicon columbium alloy.

15. Process for producing alloys of columbium which comprises reacting columbite ore in the presence of a basic slag with sufficient silicon in the form of a silicon alloy to yield an alloy in which the ratio of columbium to silicon is between about 7 to 1 and about 9 to 1.

16. Process for producing alloys of columbium from material containing oxides of columbium and tantalum, which comprises the step of reacting said material with sufficient silicon to yield an alloy in which the ratio of columbium to silicon is between about 7 to 1 and about 9 to 1, whereby a preferential reduction of columbium oxide is obtained.

17. Process for producing ferrocolumbium which comprises reacting columbite ore in the presence of a basic slag with sufficient silicon to yield an alloy in which the ratio of columbium to silicon is between about 6 to 1 and about 12 to 1.

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