United States Patent Office

2,995,439

Patented Aug. 8, 1961

1

2,995,439

PREPARATION OF HIGH PURITY CHROMIUM AND OTHER METALS

Lawrence M. Litz, Lakewood, Ohio, assignor to Union Carbide Corporation, a corporation of New York

No Drawing. Filed Feb. 2, 1959, Ser. No. 790,390

11 Claims. (Cl. 75—84)

This invention relates to the purification of certain metals, and more particularly relates to a process of purifying metals utilizing a novel scavenging compound, thereby decreasing the impurity content and subsequently improving the metallurgical properties of such metals.

The hardness and ductility of metals are generally dependent upon their impurity content in addition to the inherent properties of the metals themselves. It would be greatly advantageous to purify certain metals, such as chromium and vanadium, thereby rendering them softer and occasionally more ductile than they are as presently available. Such improved ductility and softness will facilitate drawing, extruding, machining, and molding of such metals into desired shapes.

It is therefore the primary object of this invention to produce purer metals of the above-disclosed group than have been available in the past. It is another object of this invention to provide a novel process for the purification of such metals thereby rendering them softer and occasionally more ductile than has been formerly possible.

In accord with and fulfilling the above-mentioned objects, the invention comprises either heating a metal chosen from the group consisting of chromium and vanadium to an elevated temperature in the presence of cerium monosulfide, or heating the metal in question to an elevated temperature and then placing cerium monosulfide in its presence. Broadly, the process of this invention may be carried out by heating such metal to above its melting point in contact with this compound. Constant agitation at this elevated temperature may be beneficial in order to allow all of the metal to come into contact with the compound, thereby providing an interface where purification occurs.

More particularly, the invention may be carried out by heating a metal from the above-disclosed group in a container made of cerium monosulfide to effect the escape of gaseous impurities from the metal without substantial reaction occurring between the metal and the container. A related method of purification encompasses adding the cerium monosulfide directly to a pool of the metal to be purified in the molten state. The mixture is desirably agitated for a given length of time, depending upon the impurity content of the metal and the temperature involved, and the purified metal is then recovered. It is important in the practice of any of the above-noted methods that the purification be carried out in a non-oxidizing inert atmosphere. Such an atmosphere may be provided by evacuating the system or by blanketing it with an inert gas such as helium or argon. Heating of the metal may be accomplished by induction heating through the compound if such is used as the container for the metal to be purified, or by any other convenient means which minimizes contamination of the metal by the heating element.

It is to be understood that the above discussion of chromium and vanadium applies equally as well to chromium and vanadium base alloys.

As specific examples of the practice of this invention, chromium has been purified by heating it in a cerium monosulfide crucible to various temperatures between 50° C. and 150° C. above its melting point for periods of from 5 to 32 minutes, under an argon atmosphere. The results of these test runs appear in summary form in Table A below.

2

TABLE A

Chromium melted in CeS

	Run I	Run II	Run III
Superheat, °C., above melting point.	50	75	140.
Duration, Minutes	30	32	5.
Helium Pressure, mm. Hg	380	380	100.
Surface Appearance	Silvery	Silver-Yellow	Gold.

Chemical Analysis		Run I	Run II	Run III
Impurity	Before Purification			
O_2 wt. percent	0.28	nil	0.079	0.04
H_2 wt percent	0.04	0.017	0.004	0.007
N_2 wt. percent	0.05	0.039	nil	0.012
Ce wt. percent	[1] N.T.	0.047	N.T.	0.76
S wt. percent	0.036	0.013	N.T.	0.05
Hardness: Diamond Pyramid	637	174	N.T.	205

[1] N.T.—Not tested.

A careful consideration of the above table shows that by purification with cerium monosulfide, the hardness of the various samples tested was considerably reduced, in some cases to less than half of what it had been before purification. It is also to be noted that the impurity content was generally reduced. In similar tests run on vanadium using cerium monosulfide to purify the metal, a marked reduction in hardness was also observed, as shown below in Table B.

TABLE B

Metal	° C. Superheat	Pressure, mm. Hg	Hardness	
			Standard	Purified
			DPH [1]	DPH [1]
Vanadium	50	1×10^{-5}	259	190

[1] Diamond Pyramid hardness.

Metallographic examination of vanadium showed a very significant reduction in impurity occlusions.

What is claimed is:

1. A process for purifying a material consisting of a metal selected from the group consisting of vanadium, chromium, vanadium base alloys and chromium base alloys which comprises heating said material in the presence of independently produced cerium monosulfide to a temperature above the melting point of said material, at such elevated temperature for a sufficient time to render said metal relatively ductile, and cooling said metal.

2. A process for purifying vanadium metal which comprises heating said material in the presence of independently produced cerium monosulfide to a temperature above the melting point of said metal, agitating said metal at such elevated temperature for a sufficient time to render said metal relatively ductile, and cooling said metal.

3. A process for purifying chromium metal which comprises heating said material in the presence of independently produced cerium monosulfide to a temperature above the melting point of said metal, agitating said metal at such elevated temperature for a sufficient time to render said metal relatively ductile, and cooling said metal.

4. A process for purifying chromium base alloys which comprises heating said alloy in the presence of independently produced cerium monosulfide to a temperature above the melting point of said alloy, agitating said alloy

at such elevated temperature for a sufficient time to render said alloy relatively ductile, and cooling said alloy.

5. A process for purifying vanadium base alloys which comprises heating said alloy in the presence of independently produced cerium monosulfide to a temperature above the melting point of said alloy, agitating said alloy at such elevated temperature for a sufficient time to render said alloy relatively ductile, and cooling said alloy.

6. A process for purifying a material consisting of a metal selected from the group consisting of vanadium, chromium, vanadium base alloys and chromium base alloys which comprises melting said material in a cerium monosulfide crucible under an inert atmosphere, agitating said molten material, superheating such during said agitation and cooling said purified material.

7. A process for purifying vanadium metal which comprises melting said metal in a cerium monosulfide crucible under an inert atmosphere, agitating said molten metal, superheating such during said agitation, and cooling said metal.

8. A process for purifying chromium metal which comprises melting said metal in a cerium monosulfide crucible under an inert atmosphere, agitating said molten metal, superheating such during said agitation, and cooling said metal.

9. A process for purifying chromium base alloys which comprises melting said alloy in a cerium monosulfide crucible under an inert atmosphere, agitating said molten alloy, superheating such during said agitation, and cooling said alloy.

10. A process for purifying vanadium base alloys which comprises melting said alloy in a cerium monosulfide crucible under an inert atmosphere, agitating said molten alloy, superheating such during said agitation, and cooling said alloy.

11. A process for purifying chromium which comprises melting said chromium in a cerium monosulfide crucible under an inert atmosphere, agitating said molten chromium for from 5 to 32 minutes, superheating said molten chromium between 50° C. and 150° C. above the melting point thereof during said agitation, and cooling said chromium.

References Cited in the file of this patent

UNITED STATES PATENTS

1,869,496	Osborg	Aug. 2, 1932
1,869,979	Osborg	Aug. 2, 1932
2,101,919	Schichtel	Dec. 14, 1937
2,509,189	Jordan	May 23, 1950
2,624,667	Tour	Jan. 6, 1953
2,670,284	Zvanut	Feb. 23, 1954
2,759,723	Crespi	Aug. 21, 1956
2,766,110	Meister	Oct. 9, 1956
2,801,099	Larson	Jan. 30, 1957

FOREIGN PATENTS

1312/36	Australia	Nov. 30, 1936