

C. A. PFANSTIEHL.
PROCESS OF REDUCING METALLIC OXIDS.
APPLICATION FILED NOV. 16, 1916.

1,283,286.

Patented Oct. 29, 1918.

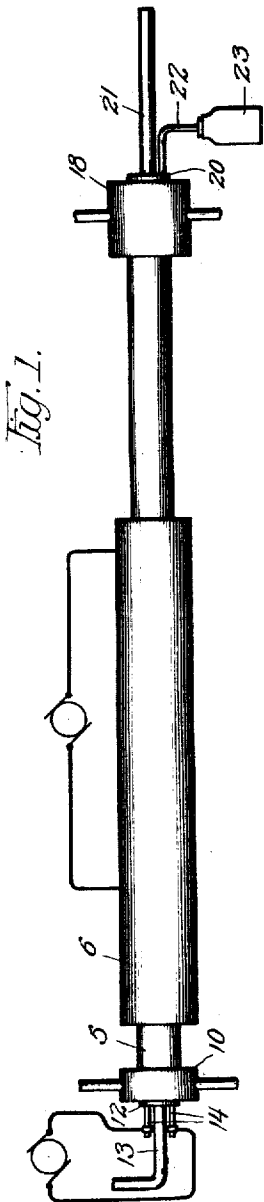


Fig. 1.

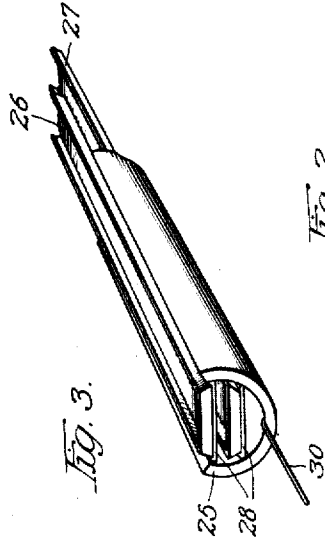


Fig. 3.

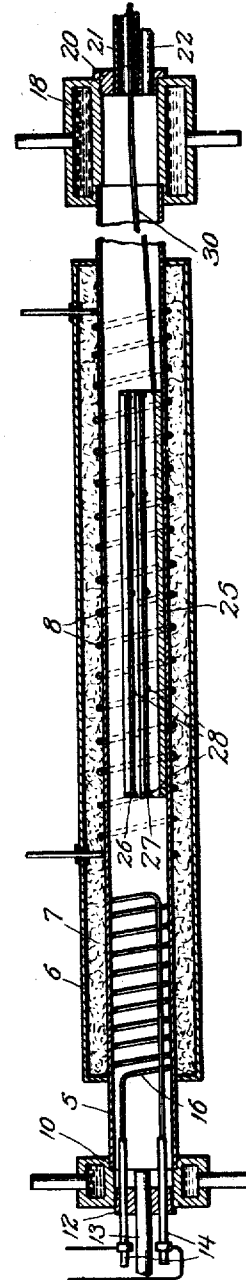


Fig. 2.

Witnesses:
Robert H. Wein
Arthur W. Carlson

Inventor
Caryl A. Pfanstiehl
Williams, Bradbury & Co.
Attys.

UNITED STATES PATENT OFFICE.

CARL A. PFANSTIEHL, OF HIGHLAND PARK, ILLINOIS, ASSIGNOR TO PFANSTIEHL COMPANY, INC., OF NORTH CHICAGO, ILLINOIS, A CORPORATION OF NEW YORK.

PROCESS OF REDUCING METALLIC OXIDS.

1,283,286.

Specification of Letters Patent.

Patented Oct. 29, 1918.

Original application filed October 15, 1915, Serial No. 55,942. Divided and this application filed November 16, 1916. Serial No. 131,633.

To all whom it may concern:

Be it known that I, CARL A. PFANSTIEHL, a citizen of the United States, residing at Highland Park, in the county of Lake and State of Illinois, have invented a certain new and useful Improvement in the Processes of Reducing Metallic Oxids, of which the following is a full, clear, concise, and exact description, reference being had to the accompanying drawings, forming a part of this specification.

My invention relates to the process of reducing metallic oxids and particularly to a process which insures the rapid and complete reduction of the material with practically no reoxidization by the condensation of moisture created by the reduction process.

In order to produce malleable tungsten with little or no mechanical working of the metal after it has been fused into an ingot, it has been found to be important that the tungsten powder from which the tungsten ingots are formed be very fine and absolutely free from tungsten trioxid from which it is ordinarily formed.

In a co-pending application 872,952 I have described a process for the production of tungsten trioxid in a very finely divided state and from which powdered tungsten satisfactory for the production of malleable tungsten may be secured. The present invention relates to a process for reducing the tungsten trioxid to tungsten powder in a manner which experience has shown produces very fine and pure tungsten powder.

This application is a division of my co-pending application Serial No. 55,942, filed October 15, 1915, relating to the apparatus by which the herein described process may be readily carried out.

It has been found important that the hydrogen gas brought into contact with the tungsten trioxid for reducing purposes be kept circulating over the surface of the tungsten trioxid at a high velocity so that the gas will become only slightly laden with water vapor given off during the reducing process. It has also been found that if the hydrogen gas is brought into contact with the heated tungsten trioxid at a temperature substantially equal to the temperature of the trioxid and that if during the passage of the hydrogen over the tungsten trioxid the same temperature is maintained none of the water

vapor picked up by the rapidly moving hydrogen will be again deposited in the tungsten.

Apparatus satisfactory for carrying out this process is illustrated in the accompanying drawings in which—

Figure 1 is an elevation of the furnace in which the external electrical connections are diagrammatically illustrated;

Fig. 2 is a section through Fig. 1 on a somewhat larger scale and with a portion of the refractory tube broken away, and

Fig. 3 is a perspective of the nest of trays in which the tungsten trioxid is supported so as to be exposed to the passing hydrogen gas.

The preferred form of apparatus for carrying out my invention herein illustrated comprises a main tube 5 of refractory material, such as silica or porcelain, this tube being in the commercial device about five feet long and two and one-half inches in diameter. Surrounding a portion of the tube 5 is a second metal tube 6, of larger diameter, the space between the two tubes being packed with magnesia, or other refractory sand, as indicated at 7. Encircling the tube 5 and in contact therewith throughout substantially two-thirds of the portion thereof inclosed by the tube 6 is a heating coil 8 of nickel-chromium or other non-oxidizing resistance wire. The left end of the refractory tube 5 extends several inches beyond the end of the tube 6 and carries the metallic water jacket 10 having an opening therein of substantially the same bore as that of the tube 5. Into the outer end of this bore is fitted a stopper 12 through which a hydrogen intake tube 13 passes and through which also pass conducting bars 14 upon the inner ends of which the hydrogen heating nichrome coil 16 is supported, this coil being adapted to be withdrawn from the end of the tube with the removal of the stopper 12. The external ends of the conducting bars 14 are connected into a suitable electric circuit, as diagrammatically shown in Fig. 1. The tube 5 extends for a distance of substantially two feet beyond the right end of the inclosing tube 6 and is not throughout this space provided with either heating or cooling means. The extreme right end of the tube 5, however, is provided with a metallic water jacket 18 similar in construction to the water jacket

10 at the left end of the tube 5, with the exception that the jacket 18 is provided with more extensive cooling surfaces. A stopper 20 is provided for the exposed end of the jacket 18 and through this the hydrogen exhaust tube 21 and the water drain 22 extend, the drain 22 being connected with a reservoir 23 into which the water formed by the chemical union of the released oxygen and the hydrogen collects, this water being condensed by the contact of the vapor with the cool water-jacketed member 18.

Fig. 3 illustrates the boat 25 carrying a nest of trays 26 and 27 in which the tungsten trioxid is distributed for the reducing operation. These trays and the boat carrying them are preferably constructed of pure nickel, the boat being provided with nickel crossrods 28 for supporting the trays one above the other so that space is provided for the circulation of the hydrogen gas between the top of the tungsten trioxid and the base of the tray next above. Tungsten trioxid is also placed in the lower part of the boat 25 to a height somewhat less than the level of the base of the lower tray.

In operating my reducing furnace, the boat 25 and its accompanying trays are filled to the proper level with tungsten trioxid and the stopper 12 having been removed and the nickel or tungsten wire 30 having been attached to the boat 25 and threaded through the hydrogen exhaust 21, the boat with its contents is drawn into the position shown in Fig. 2. The stopper 12 is then replaced and both electric circuits are completed and the supply of hydrogen gas turned into the tube 13. The heating coils 16 and 8 gradually bring the temperature of the tube and the hydrogen contained therein up to that degree at which hydrogen attacks the tungsten trioxid, reducing it to pure tungsten. The hydrogen gas is kept in rapid circulation across the surface of the tungsten trioxid until all of the trioxid has been reduced to pure tungsten. During this operation a part of the water produced by the chemical union of the released oxygen and the hydrogen passes out through the tube 21 in the form of vapor, while the greater part thereof is condensed and conducted into the reservoir

23. The entire furnace is preferably mounted with the right end slightly lower than the left end so that moisture will naturally drain to the right end of the furnace. After the reducing operation is continued for that length of time necessary for complete reduction of the tungsten trioxid, the boat with its contents is drawn into that portion of the tube 5 which is not surrounded by the heat-retaining material 7 and tube 6 and is there allowed to stand until its temperature is reduced sufficiently that it may be exposed to the atmosphere without oxidation. During this period both electric circuits are interrupted and cool, dry hydrogen is permitted to pass over the tungsten to aid in quickly reducing its temperature. When the temperature has been finally reduced sufficiently, the plug 20 is removed and the boat 25 and its contents are withdrawn and refilled with tungsten trioxid for a second operation.

Although it is obvious that the process herein described may be carried out by means of many different types of apparatus, the apparatus herein illustrated is the most satisfactory of any of which I am at present aware.

What I claim as new and wish to secure by Letters Patent of the United States is:

1. The method of reducing finely powdered metallic oxids which consists in heating the oxid in an atmosphere of rapidly moving hydrogen which has been preheated to a temperature equal to that of the metallic oxid.

2. The method of reducing finely powdered tungsten trioxid which consists in passing a blast of highly heated hydrogen over the surface of the tungsten trioxid and at the same time heating the tungsten trioxid just sufficiently to overcome radiation, and to supply the necessary heat for the vaporization of the moisture created by the reduction of the oxid.

In witness whereof, I hereunto subscribe my name this 8th day of November, A. D. 1916.

CARL A. PFANSTIEHL.

Witnesses:

CLARENCE W. BALKE,
MARGARET GRÖBLEIN.