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REACTOR FURNACES

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Fig. 1.

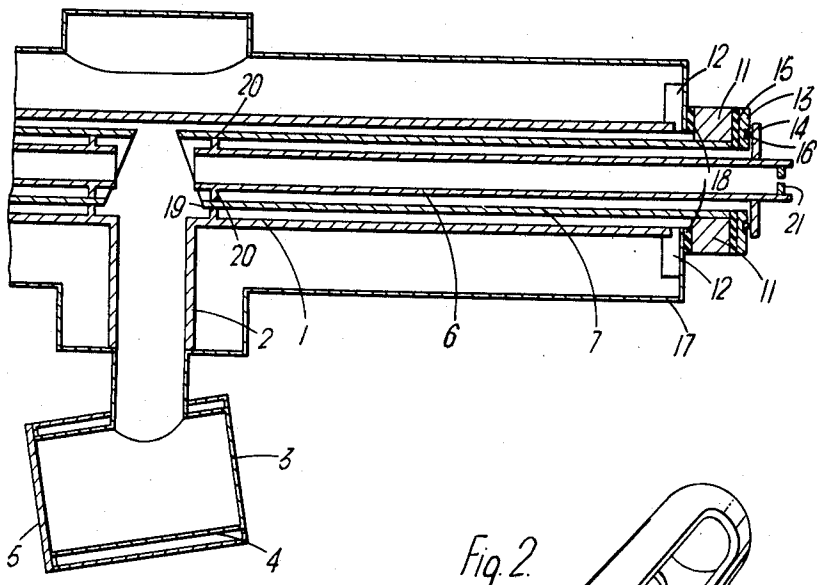
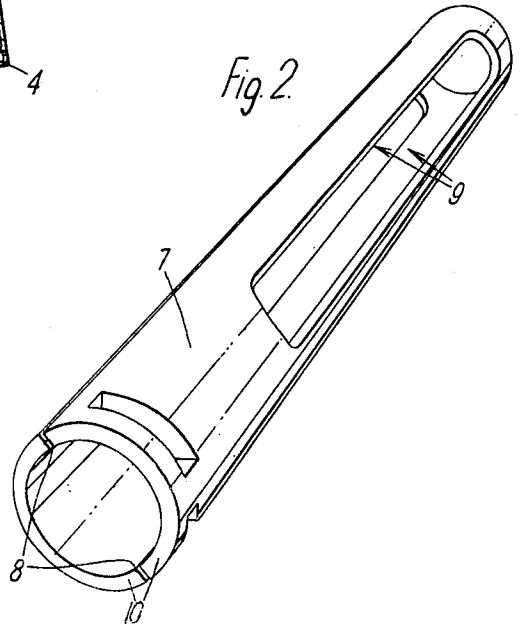


Fig. 2.



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REACTOR FURNACES

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3 Claims. (Cl. 23—277)

This invention relates to furnaces, and especially to furnaces having special utility in the production of titanium diboride.

Titanium diboride can be produced by the reaction at high temperatures of titanium dioxide, boric oxide and carbon according to the equation:

$$TiO_2+B_2O_3+5C \rightleftharpoons TiB_2+5CO$$

In carrying out the reaction it has been found highly desirable to heat an intimate mixture of the reactants in the form of a self supporting cylindrical or other slug in an atmosphere of carbon monoxide at a temperature above 1550° C. As the back reaction is important at temperatures between 900° C. and 1550° C., it is highly advantageous to quench the titanium diboride as rapidly as possible from a temperature of 1550° C. or above to one below 900° C., and it is the object of this invention to provide a form of furnace which is particularly suitable for carrying out this reaction and for rapidly quenching the product.

The furnace of the invention comprises at least one substantially horizontal heating chamber of generally tubular shape provided at or towards one end with a feed opening and at or towards the other with a discharge opening, a quenching chamber at a level lower than the discharge opening, a discharge conduit extending, preferably perpendicularly, from the discharge opening of the heating chamber to the quenching chamber, an electrical resistance heating element outside the heating chamber and extending along it substantially up to or past the discharge opening. It is to be understood that when the expressions "substantially horizontal" and "at a lower level" are used in the specification and claims, the apparatus is considered as being set up for operation. The expression "extending substantially up to the discharge opening" means that the heating element extends sufficiently close to the opening to prevent the temperature of the reaction chamber falling off significantly between the end of the heating element and the discharge opening.

Advantageously the furnace comprises two or more such heating chambers, each with associated heating element extending up to or past the discharge opening, leading into a single discharge conduit so that while one of the chambers is being charged another or others can be in operation thus providing the continuous presence of carbon monoxide gas within the system. In the preferred form of furnace, two heating chambers and the discharge conduit are disposed in the shape of a "T." When the furnace is to be used in the production of titanium diboride as described above, it is highly desirable that the heating chamber be made of graphite, and the discharge conduit may also advantageously be made of this material. The electrical resistance heating element or elements for the heating chamber or chambers preferably are generally tubular, surround while being spaced from the heating chamber, and are provided with two longitudinal slits extending from one end almost to the other end so providing a there-and-return path for an electric current. Preferably the element comprises a cylindrical graphite tube in which the two slits are diametrically opposed and which is provided with a contact terminal on each of the halves of the tube near the slit end.

Such "slit tube" heating elements are similar in principle to the "hair-pin" type of element known in the resistance furnace industry and are not in themselves new; and in their simplest form do not provide a completely uniform temperature along the whole length of a heating chamber within them. In the past this defect has been overcome by suitably contouring the element, i.e. reducing its thickness over certain parts to varying degrees; the details of the contouring required are determined empirically for each particular application. This has the disadvantage that the element is considerably weakened by the local reductions in thickness. We have found, and this forms another aspect of the present invention, that uniform heating can be obtained without this disadvantage by completely cutting away part of the tubular element, while maintaining the rest at its original thickness. The precise size and shape of the cut-away portions which will give the most uniform heating effect can readily be determined by experiment, as has been done with contoured elements, and it has been found that cutting at least one longitudinal opening in each of the wall portions of the heating element separated by the slits gives good results. The complete cutting away of some parts of the tube has a much smaller weakening effect, if indeed any, from a practical standpoint, than the mere thinning of selected areas; moreover if any oxidation takes place in these areas it has a much smaller effect on the local resistance of the heater.

When the furnace is to be used for the production of titanium diboride as described above, provision should be made for removal of effluent gases produced in the reaction. Preferably these gases are allowed to escape through an exit port or ports such that they do not pass through the quenching chamber. Where the furnace has a single heating chamber the gases may be led out through the feed opening. Alternatively, especially with the preferred form of furnace having two heating chambers and being T-shaped, the gas may be led away from the furnace through a port near the discharge opening; this arrangement can be used to ensure that the gases are not cooled sufficiently before leaving the furnace for relatively non-volatile materials to be deposited.

One form of furnace according to the invention is shown in the accompanying drawing in which FIGURE 1 is a sectional side view of the furnace, and FIGURE 2 is a perspective view of the heating element of the furnace.

Referring to the figures, the furnace comprises a horizontal graphite tube **1** (hereinafter called the outer graphite tube) into which at its middle opens a discharge conduit **2** which leads to a quenching chamber **3** provided with a surrounding water cooling jacket **4** and a discharge opening having a gas tight door **5**. The two limbs of the outer graphite tube (of which only one is shown in full in the drawing) both comprise a horizontal tubular graphite heating chamber **6** disposed inside and coaxial with the outer graphite tube. A gas exit port (not shown) may be provided in the outer graphite tube above the discharge conduit. Between the heating chamber and the outer tube there is a graphite electrical resistance heating element **7**, the top of which at its inner end projects further than the bottom. This element (which is shown in detail in FIGURE 2) comprises a graphite tube having two diametrically opposed longitudinal slits **8** extending from the outer end of the element almost to the inner end; the element is of uniform thickness but is provided with a longitudinal opening **9** in each half **10** of the element so that in operation the heating effect does not vary substantially along the length of the element. Each half **10** of the slit end of the element is provided with an

aluminium contact block **11** for connection with a source of electrical supply. A water cooling ring **12** is provided for supporting one end of the outer graphite tube and the heating chamber is supported, cooled and sealed by water-cooled plates **13** and **14**, insulating washer **15** and rubber O-ring **16**. A tubular casing **17** encloses the outer graphite tube and also the shaft almost to its bottom end. The free space within the casing is packed with carbon black (not shown) for heat-insulating purposes. The contact blocks **11** are separated from the casing **17** by an insulating washer **18**. At its inner end the element rests on a pair of graphite studs **19** which project inwards from the outer tube. The inner end of the heating chamber is provided with four studs **20** (of which only two are shown) which are equally spaced around the circumference of the heating chamber. The studs **20** serve to space the heating chamber from the element while allowing the heating chamber to be periodically rotated so as to even out corrosion. When the heating chamber eventually requires replacement it can readily be removed from the furnace and a new chamber inserted even while the furnace is still hot. A removable apertured plate **21** is provided at the feed inlet of the heating chamber **6**.

The furnace is particularly suitable for the production of titanium diboride by the reaction described above and its method of use will be described with particular reference to this reaction.

Self supporting slugs comprising an intimate mixture of boric oxide, carbon and titanium dioxide, such as described by the copending applications Timms Serial No. 194,216 and Wood et al. Serial No. 194,215, both of which were filed May 10, 1962, are introduced into the heating chamber **6** and are heated therein at a temperature above 1550° C. until reaction is complete, the inner end of the heating chamber **6** being maintained at a temperature of at least 1550° C. The heated slugs, which now consist of titanium diboride, are pushed along the heating chamber and fall down the discharge conduit **2** into the water-cooled quenching chamber **3**. The titanium diboride can be removed from the quenching chamber on opening the gas tight door **5**, in the form of broken masses which can readily be pulverised or crumbled.

While the furnace is highly suitable for use in the production of titanium diboride, it can also be used with advantage for other reactions, including the production of similar metal borides, boron carbide, and boron nitride.

We claim:

1. A furnace comprising at least two substantially horizontal heating chambers of generally tubular shape, each consisting of end portions and an intermediate portion, one of said end portions of each heating chamber being provided with a feed opening and the other being provided with a discharge opening, a discharge conduit extending downwards from said heating chambers and into which the discharge opening of each of the heating chambers leads, a quenching chamber at the bottom of the discharge conduit and, outside each of the heating chambers, a graphite electrical resistance heating element of generally tubular shape and of uniform thickness extending along the chamber at least substantially up to the discharge opening, said graphite electrical resistance heating elements being provided with two diametrically opposed

longitudinal slits extending from the feed opening end towards but not reaching the discharge opening end of the heating element, the wall of said heating element having at least one longitudinal opening cut between each longitudinal slit whereby in operation of the furnace the temperature along the length of the heating chamber is rendered more uniform, and each half of the slit end of each heating element being provided with an electrical contact.

2. A furnace comprising at least one substantially horizontal heating chamber of generally tubular shape and consisting of end portions and an intermediate portion, one of said end portions being provided with a feed opening and the other end portion being provided with a discharge opening, a quenching chamber at a level lower than the discharge opening, a discharge conduit extending from the discharge opening to the quenching chamber, and a graphite electrical resistance heating element of generally tubular shape surrounding and spaced from the heating chamber, said heating element being provided with two diametrically opposed longitudinal slits extending from one end towards but not reaching the other end of the heating element, each half of the slit end being provided with an electrical contact.

3. A furnace comprising at least one substantially horizontal heating chamber of generally tubular shape and consisting of end portions and an intermediate portion, one of said end portions being provided with a feed opening and the other end portion being provided with a discharge opening, a quenching chamber at a level lower than the discharge opening, a discharge conduit extending from the discharge opening to the quenching chamber, and a graphite electrical resistance heating element of generally tubular shape and of uniform thickness surrounding and spaced from the heating chamber, said heating element being provided with two diametrically opposed longitudinal slits extending from one end towards but not reaching the other end of the heating element, the wall of said heating element having at least one longitudinal opening cut between each longitudinal slit whereby in operation of the furnace the temperature along the length of the heating chamber is rendered more uniform, and each half of the slit end of said heating element being provided with an electrical contact.

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