

Double-walled graphite funnel

The present invention relates to a double-walled graphite funnel which is filled with a pourable filling material. The present invention further relates to a kit for manufacturing the double-walled graphite funnel.

Graphite funnels, also known as graphite pans or graphite cones, are used in processes carried out at high temperatures. The manufacture of monocrystals by the Czochralski process is associated with high-temperature methods of this type.

In this process, the material to be formed into the monocrystal is melted in a crucible at high temperatures and the monocrystal which forms on a seed crystal is drawn out of the melt. This process is described for example in "Industrielle Anorganische Chemie" ("Industrial Inorganic Chemistry"), M. Bertau et al., WILEY-VCH Verlag GmbH & Co. KGaA, edition 4, 24 July 2013, pages 402-403.

It is known to be advantageous to shield the forming monocrystal from the influences present in the system such as the heat of the melt crucible and any gas streams which may be present in the systems, so as to maintain optimum crystallisation conditions. Graphite funnels are used for this purpose in the art, and are arranged above the melt crucible in the system.

The graphite funnel is arranged in the Czochralski system in such a way that the lower, smaller opening thereof points towards the melt crucible. The monocrystal, which is drawn out of the melt through the lower opening of the funnel towards the upper opening of the funnel, is enclosed at a distance, and thus shielded, by the funnel walls, which extend between the upper and lower opening of the funnel.

Depending on the construction of the system in which they are used, the graphite funnels used in the Czochralski process substantially have the shape of a frustum outer surface, which can be modified by circumferential extensions or circumferential reductions, in such a way that for example mounting faces or engagement faces can be formed as holding elements for the corresponding system regions or can engage therein.

So as further to improve the shielding properties of the described graphite funnels, they are for example formed double-walled and additionally insulated. For this purpose, the graphite funnel is formed of an inner and an outer funnel, which are formed in such a way that the smaller funnel is fitted into the outer funnel so as to form a cavity. The cavity is subsequently filled with planar insulating material.

In the art, graphite felts for example are used as a planar insulating material. A distinction is made between soft felts and hard felts. Graphite felts have a low thermal conductivity, making them suitable as an insulating material. However, these graphite felts are disadvantageous in terms of the manufacturing costs and the ease of handling when they are fitted into the cavity of the graphite funnel.

Depending on the graphite funnel, the hard graphite felts have to be provided in a precise fit for the corresponding cavity, necessitating individual production. The hard graphite felts are thus generally prepared as a component in accordance with the required adaptation to the graphite funnel. This individual preparation of the precisely fitting hard graphite felts is cost-intensive because of the material costs and the costs of the adaptation.

If soft graphite felts are used, they do not need to be provided in precisely fitting dimensions. However, there are still comparatively high material costs and a technical complexity of use. So as to introduce the flexible material into the graphite funnel, in some cases it is necessary to plug the material into the cavity in a complex manner. This is disadvantageous because sub-regions of the cavity may be extremely poorly accessible, but as uniform an insulation as possible is to be achieved.

Thus, overall, there are comparatively high costs associated with the use of graphite felts. Further, the manufacture and the introduction into the graphite funnel involve considerable technical complexity.

Further, the service life of the graphite felts is usually less than that of the graphite funnel, meaning that the insulating material has to be replaced during the service life of the graphite funnel, and so the procedure has to be repeated.

Therefore, an object of the present invention is to overcome the drawbacks of the prior art and to provide a graphite funnel which can be produced cost-effectively, without great

technical complexity, and at the same time is adaptable to specific requirements in terms of thermal conductivity.

Where the term “thermal conductivity” is used in connection with the present application, unless stated otherwise this refers to the thermal conductivity at room temperature. In the context of the present application, room temperature is understood to mean a temperature of 20 °C.

A further object of the present invention is to provide a graphite funnel which, as a result of the properties thereof, can be used in Czochralski processes.

The graphite funnel should be able to be provided cost-effectively and at a low technical complexity for the corresponding use. In particular, it should be possible to adapt the thermal conductivity of the graphite funnel to different technical requirements, within particular limits, at a low technical complexity and with a high flexibility. It should thus be possible, without major technical complexity, to adapt the thermal properties of the graphite funnel to the intended use.

It should also be possible to introduce the filling material at a lower technical complexity than in the prior art.

Where it is disclosed in connection with the present invention that the gap is “filled”, this includes embodiments in which the cavity is completely filled as well as those in which a sub-volume of the cavity is filled.

The object of the present invention is achieved by providing a double-walled graphite funnel which has an upper opening and a lower opening, the openings having different clear widths and being arranged mutually parallel, and the double-walled graphite funnel being formed from an inner funnel and an outer funnel, the inner funnel and outer funnel consisting of graphite, the inner funnel being configured in terms of the dimensions and shape thereof in such a way that said inner funnel can be received in the outer funnel, the inner funnel being received in the outer funnel in such a way that a gap is formed between the inner funnel and the outer funnel, the gap being delimited, adjacent to the opening having a smaller clear width, by a contact surface formed between the inner funnel and the outer funnel, and the gap being accessible from the outside adjacent to the opening having the larger clear width,

wherein the gap between the inner funnel and the outer funnel is filled with a pourable filling material.

An embodiment of the double-walled graphite funnel is preferred in which the inner funnel and the outer funnel have in the region of the contact surface thereof elements by means of which the funnels can be secured to one another.

A double-walled graphite funnel is also preferred in which the outer funnel has the form of a frustum outer surface.

A double-walled graphite funnel is also preferred in which the outer funnel substantially has the form of a frustum outer surface which deviates from the frustum shape at the outer circumference thereof by way of circumferential reductions and/or circumferential extensions.

A graphite funnel is further preferred in which the inner funnel and the outer funnel have the same or a different shape.

A double-walled graphite funnel is also advantageous in which the gap formed between the inner funnel and the outer funnel is sealed by a trapping arrangement.

It is further preferred for the trapping arrangement of the double-wall graphite funnel to comprise at least one graphite trapping element.

In particular, a double-walled graphite funnel is advantageous in which the graphite trapping element is a graphite ring.

A double-walled graphite funnel is also advantageous in which the trapping arrangement comprises at least one sealing element selected from heat-resistant materials.

A double-walled graphite funnel is preferred according to the invention in which the pourable filling material has a typical thermal conductivity of 0.02 W/mK to 75 W/mK, preferably of 0.02 W/mK to 10 W/mK, at room temperature.

A graphite funnel is further preferred in which the pourable filling material is selected from materials based on elemental carbon, in particular graphite chips.

The object of the invention is also achieved by providing a kit for manufacturing a graphite funnel according to the invention, as described above along with the preferred embodiments thereof, wherein the kit comprises an inner funnel, an outer graphite funnel and filling material.

A kit is preferred which further comprises a trapping arrangement comprising at least one graphite trapping element and at least one sealing element.

Surprisingly, it has been found that the double-walled graphite funnel according to the invention has major advantages over prior art graphite funnels.

Thus, the use of pourable filling material makes it possible to fill the formed gap uniformly without major technical complexity. Depending on the insulating properties to be achieved, a suitable pourable insulating material can be selected. Material mixtures of pourable materials may also be used so as to achieve the desired thermal conductivity of the graphite funnel according to the invention. Pourable materials are for example powders, granulates, chips and mixtures thereof.

Further, the graphite funnel according to the invention can additionally be individualised in terms of the insulating properties thereof by the selection of filling materials having different particle size and bulk density.

Materials suitable as a filling material are for example materials based on elemental carbon, such as graphite powder or graphite chips, but also process-compatible filling materials having a typical thermal conductivity at room temperature in the range of 0.02 to 50 W/mK.

The pourable filling material may be an insulating material. Depending on the application, the typical thermal conductivity of insulating materials at room temperature is in the range of 0.02 W/mK to 10 W/mK. If these materials are exposed to operating temperatures of approximately 1500 °C, this corresponds to values of 0.08 W/mK to 40 W/mK.

It is thus advantageous that the fragmented insulating materials used in the art, such as soft felt and hard felt, no longer have to be used as insulating material. This leads to a considerable reduction in costs.

The use of graphite chips has been found to be particularly advantageous. Thus, a graphite funnel according to the invention which is provided for use in a conventional Czochralski system can be filled with graphite chips which have a thermal conductivity corresponding to that of graphite felts.

If for example graphite chips are used as a pourable insulating material, the double-walled graphite funnel can be subjected to cleaning processes for graphite which are known in the art. In this case, a sufficiently high purity can be achieved to make the graphite funnel according to the invention also useable in the field of semiconductor technology.

As a result of the combination according to the invention of the double-walled graphite funnel formed by the inner and the outer funnel, in other words of a graphite shaped body, and of the comparatively cost-effective filling material, a cost-effective price level can be achieved. As a result of the flexibility of the pourable filling material, a broad use for differently configured shaped bodies is possible. Thus, the graphite funnel according to the invention may for example also have protrusions and regions of the gap which are hard to access, but still have insulation even in these regions. Introducing planar insulating material into these regions, if it is even possible to do so, is at the very least associated with high complexity.

As a result of the variable combination, the design complexity and storage of semi-finished products and finished parts are reduced, for example. Further assembly of individual parts, as when hard or soft graphite felts are used, is not required.

The graphite funnel according to the invention can be provided as a fully mounted unit. However, a kit from which the user assembles the graphite funnel may also be provided.

The inner and outer funnel both consist of graphite. The two funnels may be constructed from identical or if required from different graphite material.

If the double-walled graphite funnel according to the invention is provided to a user, because of the fully mounted unit the user is not required to assemble various individual parts. A

suitable filling material is selected for this purpose, and the user acquires the graphite funnel according to the invention in accordance with his requirements, for example in a suitable size and shape and with suitable thermal properties such as the typical thermal conductivity. If the double-walled graphite funnel is provided for use in the Czochralski process, the gap formed between the inner and outer funnel is filled with a pourable insulating material.

The graphite funnel according to the invention may, if required, be subjected to the cleaning method known in the art for graphite materials. In this way, the graphite funnel according to the invention may also be provided at a high purity.

The provision of the graphite funnel with a trapping arrangement simplifies the handling thereof. The trapping arrangement prevents filling material from being able to fall out of the gap, for example during transport of the graphite funnel. As a result, provision of the graphite funnel for use at a place other than the place of manufacture is also supported. Further, the trapping arrangement prevents filling material from escaping during use of the graphite funnel through an oversight, as could occur for example if the graphite funnel according to the invention were tipped.

The trapping arrangement is preferably selected in such a way that the graphite funnel according to the invention can be used without further modification after trapping using the trapping arrangement. For this purpose, material which withstands the process conditions to which the graphite funnel according to the invention is exposed, and which is simultaneously suitable for sealing, is preferably used in the trapping arrangement.

If a trapping arrangement is provided, this trapping arrangement may be formed by removal of material from the inner or outer funnel. Thus, for example, a graphite ring can be obtained in that an annular structure is separated from the upper edge of the inner or outer funnel. In this embodiment, the graphite ring is thus obtained for example by cutting it off from the inner or outer funnel.

This embodiment is advantageous in particular because the graphite ring thus has the required dimensions to be placed back on the funnel in an exact fit, since the funnel and graphite ring previously formed a unit.

In a preferred embodiment, the access to the gap between the inner and the outer funnel is formed or enlarged by separating the graphite ring from the inner or outer funnel.

Brief description of the drawings

In the drawings:

Fig. 1 is a longitudinal section through an embodiment of the graphite funnel 1 according to the invention;

Fig. 2 is a longitudinal section through a partial view of an embodiment of the graphite funnel 1 in the region of the trapping arrangement; and

Fig. 3 is a three-dimensional exploded view of an embodiment of the graphite funnel 1.

The present invention is described in greater detail by way of the accompanying drawings.

Fig. 1 shows an embodiment of the double-walled graphite funnel 1 according to the invention in longitudinal section.

In the embodiment shown, the inner funnel 4 and the outer funnel 3 have a different shape. The inner and the outer funnel have substantially the shape of a frustum outer surface, the outer surfaces of the two funnels having different angles of inclination in the example shown. In the lower region of the inner and the outer funnel, they come into contact with one another. So as to fix the inner funnel 4 and the outer funnel 3 to one another, in this embodiment fixing aids are formed by means of which the outer funnel 3 and the inner funnel 4 are fixed to one another. In this case, a circumferential web 5 and a groove 6 are shown as fixing aids, the web 5 on the outer funnel 3 engaging in a groove 6 on the inner funnel 4 (tongue-and-groove principle).

The gap spanned by the arrangement of the inner funnel 4 and the outer funnel 3 is denoted as 7. The gap 7 is filled with a pourable material 8.

It is further shown that the double-walled graphite funnel 1 is sealed using a trapping arrangement 13. In this embodiment, as well as a graphite trapping element, in this case the trapping arrangement comprises a graphite ring 9 and also two sealing elements 10 and 11. The sealing element 10 is formed annular, and in this embodiment the sealing element 11 is a strip-shaped element which is arranged annularly. The sealing elements are arranged between the inner funnel 4 and the outer funnel 3 and consist for example of graphite felt.

In the embodiment shown of the double-walled graphite funnel 1, the graphite trapping element formed as a graphite ring 9 was originally part of the inner funnel 4.

The trapping arrangement 13 can be fixed to the double-walled graphite funnel 1 according to the invention using aids, for example in that at least one connection aid 2 such as a pin is introduced into the upper edge of the outer funnel 3 through the trapping arrangement 13. This connection aid 2 is preferably a connection aid which can be removed without damaging the double-walled graphite funnel 1. As a result, the trapping arrangement 13 of the filled graphite funnel 1 can be removed, and the filling material can be replaced or topped up.

The use of a trapping arrangement 13 is advantageous for ensuring that no filling material escapes during the handling or transport of the graphite funnel according to the invention. The trapping arrangement shown in this embodiment is purely exemplary, and trapping arrangements without sealing elements or having more than two sealing elements may also be used.

In this embodiment, an outer funnel 3 is shown which does not deviate from the frustum shape by way of circumferential reductions or circumferential extensions. However, circumferential deviations of this type may be required depending on the construction of the system for which the double-walled graphite funnel 1 according to the invention is provided.

Fig. 2 is a detailed view of the trapping arrangement 13 having two sealing elements, specifically a graphite felt upper ring 10 and a graphite felt inlay 11, which are arranged between the inner funnel 3 and the outer funnel 4. The termination is formed by a graphite trapping element, in this case a graphite ring 9.

Fig. 3 is a three-dimensional exploded view of an embodiment of the double-walled graphite funnel 1 according to the invention. It is shown how the inner funnel 4 is received in the outer funnel 3 and the sealing elements 10 and 11 are arranged between the outer funnel 3 and the inner funnel 4. The graphite ring 9 is shown above the funnels 3 and 4.

Example

Methods for manufacturing graphite funnels, such as are used as the outer funnel 3 and inner funnel 4 in the present invention, are known in the art.

Graphite semi-finished product, such as may be obtained under trade name CZ3 or CZ5 from the SGL CARBON GROUP, is used. The manufacture uses shaping methods known in the art, specifically turning and milling.

Since the double-walled graphite funnel according to the invention is provided for differently dimensioned and differently constructed systems, meaning for example different Czochralski systems, variants in different sizes and various designs are provided. The example described in the following merely outlines one possible embodiment of the double-walled graphite funnel according to the invention, and should not be interpreted as limiting the invention to this embodiment.

An outer funnel 3 and an inner funnel 4 have been manufactured from graphite semi-finished product. The inner funnel 4 has an upper diameter of 480 mm and a lower diameter of 220 mm. In this embodiment, the height of the inner funnel 4 is 309 mm.

The outer funnel 3 has an upper diameter of 500 mm and a lower diameter of 328 mm. The height of the outer funnel 3 is 309 mm. The inner funnel 4 and outer funnel 3 are in the shape of a frustum outer surface, differing in the angle formed between the outer surface line and frustum axis in each case.

The size and configuration of the inner and outer funnel results in a gap volume of approximately 7 litres. This volume is filled with a pourable filling material.

In the present embodiment, as the filling material 8, graphite chips obtained from SGL Carbon GmbH and having the following properties are used:

Thermal conductivity: 0.12 W/mK

True density according to DIN 51901: 2.02 g/cm³

Blaine specific surface area according to DIN 66126: 7200 cm²/g

Bulk density according to DIN 52912: 0.55 g/cm³

Average particle size, from Cilas Granulometer 715: 16 µm

The thermal conductivity is thus expediently determined by the Hot Disk® method, in which a disc-shaped sensor element, the eponymous Hot Disk®, which consists of a conductor spiral and is electrically insulated, is introduced into the bulk – in this case the graphite chips. During the measurement process, the sensor is electrically heated, and the rise in temperature over time is recorded. The sensor is simultaneously a heating element and a resistance thermometer. The thermal conductivity, among other things, can be calculated from the recorded measurement data.

However, the use of this material is merely exemplary; other filling materials may also be used, such as other graphite chips.

The gap 7 is filled with the graphite chips by discharge without a protective gas. During packing, the powder or granulate is compressed primarily by means of a vibration device.

After filling, the double-walled graphite funnel 1 is sealed. For this purpose, in this embodiment, a trapping arrangement 13 is used which comprises an annular sealing element 10, an annularly arranged strip-shaped sealing element 11 and a graphite trapping element, in this case a graphite ring 9. The annularly arranged, strip-shaped sealing element 11 is shaped upright with respect to the ring. The sealing elements used in this example consist of soft graphite felt having a thickness of 5 mm made of GFA 5 from SGL CARBON GmbH.

Subsequently, the graphite ring 9 is placed on the double-walled graphite funnel 1 according to the invention, and the gap is thus closed.

In this embodiment, the graphite ring 9 is made of an identical material to the outer funnel 3 and inner funnel 4. The graphite ring 9 is dimensioned in such a way that it can cover the gap 7 between the inner and outer funnel in the region of the opening 12 having the larger

clear width. Further, the graphite ring 9 is preferably configured in such a way that it is positioned on the graphite funnel 1 according to the invention, optionally fixed thereto. Fixing may be provided by means of a connecting element 2, but may also be provided by engagement of a groove formed in the graphite ring 9 in the funnel edge, by the tongue-and-groove principle.

In the present embodiment, the trapping arrangement 13 is fixed to the graphite funnel 1 according to the invention using a connection aid 2. For this purpose, an opening, through which a pin 2 is introduced into the edge of the outer funnel, is formed in the graphite ring 9. However, it is also possible to use other connection aids.

There is further the possibility of using a graphite ring 9 into which a through-opening or a plurality of through-openings for at least one connection aid are introduced.

The graphite funnel 1 according to the invention described in this example is suitable for use in Czochralski processes.

As a result of the chemical inertness of the filling material, it is strongly suspected that the graphite funnel according to the invention still has the original insulating properties even after a relatively long duration of use. This suggests that it will probably not be necessary to replace the filling material over the course of the service life. However, this does depend on the process conditions of the system in which the graphite funnel is used. In any case, the duration for which the graphite funnel according to the invention is usable will exceed the period after which insulating material in the prior art funnels has to be replaced.

The present invention provides a double-walled graphite funnel which has major advantages over the prior art. The graphite funnel according to the invention is filled with a filling material which influences the thermal properties of the graphite funnel. In one embodiment, the disclosed double-walled graphite funnel is filled with graphite chips and as a result of the insulating properties is suitable in Czochralski systems for shielding the monocrystals cultivated therein. Among other things, it is advantageous that the filling can take place individually and cost-effectively at a low technical complexity.

List of reference numerals

1	Double-walled graphite funnel
2	Connection aid
3	Outer funnel
4	Inner funnel
5	Fixing aid, web
6	Fixing aid, groove
7	Gap
8	Pourable filling material
9	Graphite trapping element, graphite ring
10	Sealing element
11	Sealing element
12	Upper opening
13	Trapping arrangement
14	Lower opening

Claims**1. Double-walled graphite funnel (1)**

which has an upper opening (12) and a lower opening (14),
the openings (12, 14) having different clear widths and being arranged mutually parallel,
and the double-walled graphite funnel (1) being formed from an inner funnel (4) and an outer funnel (3),
the inner funnel (4) and outer funnel (3) consisting of graphite,
the inner funnel (4) being configured in terms of the dimensions and shape thereof in such a way that said inner funnel can be received in the outer funnel (3),
the inner funnel (4) being received in the outer funnel (3) in such a way that a gap (7) is formed between the inner funnel (4) and the outer funnel (3),
the gap (7) being delimited, adjacent to the opening (14) having a smaller clear width, by a contact surface formed between the inner funnel (4) and the outer funnel (3),
and the gap (7) being accessible from the outside adjacent to the opening (12) having the larger clear width,
characterised in that the gap (7) between the inner funnel (4) and the outer funnel (3) is filled with a pourable filling material (8).

2. Double-walled graphite funnel (1) according to claim 1, characterised in that the inner funnel (4) and the outer funnel (3) have in the region of the contact surface thereof fixing aids (5, 6) by means of which the outer funnel (3) and the inner funnel (4) can be secured to one another.

3. Double-walled graphite funnel (1) according to any of the preceding claims, characterised in that the outer funnel (3) has the shape of a frustum outer surface.

4. Double-walled graphite funnel (1) according to either claim 1 or claim 2, characterised in that the outer funnel (3) has substantially the shape of a frustum outer surface which deviates from the frustum shape at the outer circumference thereof by way of circumferential reductions and/or circumferential extensions.

5. Double-walled graphite funnel (1) according to any of the preceding claims, characterised in that the inner graphite funnel (4) and the outer funnel (3) have the same or a different shape.
6. Double-walled graphite funnel (1) according to any of the preceding claims, characterised in that the gap (7) formed between the inner funnel (4) and the outer funnel (3) is sealed by means of a trapping arrangement (13).
7. Double-walled graphite funnel (1) according to claim 6, characterised in that the trapping arrangement (13) comprise at least one graphite trapping element (9).
8. Double-walled graphite funnel (1) according to claim 7, characterised in that the graphite trapping element (9) is a graphite ring.
9. Double-walled graphite funnel (1) according to any of claims 6 to 8, characterised in that the trapping arrangement (13) comprises at least one sealing element (10, 11) which is selected from heat-resistant materials.
10. Double-walled graphite funnel (1) according to any of the preceding claims, characterised in that the pourable filling material (8) has a typical thermal conductivity of 0.02 W/mK to 75 W/mK, preferably of 0.02 W/mK to 10 W/mK, at room temperature.
11. Double-walled graphite funnel (1) according to any of the preceding claims, characterised in that the pourable filling material (8) is selected from materials based on elemental carbon, in particular graphite chips.
12. Kit for manufacturing a graphite funnel (1) according to any of claims 1 to 11, characterised in that the kit comprises an inner funnel (4), an outer funnel (3) and filling material (8).
13. Kit according to claim 12, characterised in that the kit further comprises a trapping arrangement (13) comprising at least one graphite trapping element (9) and at least one sealing element (10, 11).