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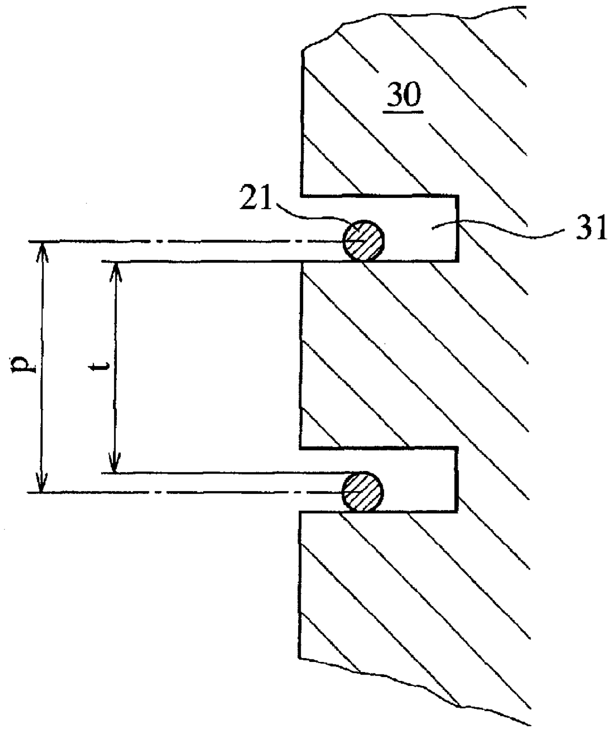
(54) MoSi2-based coil heater and tubular heater module having the same

(57) The invention is directed to an $MoSi_2$-based coil heater including a coil having an inner diameter (D) not smaller than 300 mm, in which the inner diameter (D) of the coil and an interheater distance (t) satisfy the following condition:

$$0.9 \leq t/(D/2)^{1/2} \leq 4.0.$$

FIG. 3



EP 2 701 458 A1

Description

FIELD OF THE INVENTION

[0001] The present invention relates to an $MoSi_2$-based coil heater and a tubular heater module using the same.

BACKGROUND OF THE INVENTION

[0002] An $MoSi_2$ (molybdenum disilicide)-based heater is used in a tubular heat treatment furnace which can be used at a high temperature in the air. For example, a semi-cylindrical wave-type multi-shank heater and a cylindrical spiral (coil-shaped) heater disclosed in JP-A-8-143365 are known as the $MoSi_2$-based heater. The semi-cylindrical wave-type multi-shank heater has been heretofore used in a large-scale tubular heat treatment furnace. When the bore diameter of the semi-cylindrical wave-type multi-shank heater becomes large, there is however a problem of troublesome installation, etc. because it is necessary to accommodate the semi-cylindrical wave-type multi-shank heater hung on a plurality of hooks. On the other hand, the cylindrical spiral (coil-shaped) heater has a problem of thermal deformation and temperature uniformity when the coil inner diameter of the heater is not smaller than 300 mm, so that the cylindrical spiral (coil-shaped) heater has not been realized in the existing circumstances.

SUMMARY OF THE INVENTION

[0003] The invention has been accomplished in consideration of the aforementioned circumstances. An object of the invention is to provide an $MoSi_2$-based coil heater which is excellent in durability and temperature uniformity and which has an inner diameter not smaller than 300 mm.

[0004] The present inventors have found from eager researches that an $MoSi_2$-based coil heater high in temperature uniformity, little in thermal deformation and excellent in durability can be obtained by defining the relation between the inner diameter (D) of a coil (in other words, inner diameter (D) of a helix made by a coil) and the interheater distance (t) in a predetermined range even if the inner diameter (D) of the coil is not smaller than 300 mm, so that the present inventors have hit upon the invention.

[0005] A subject of the invention is an $MoSi_2$-based coil heater including: a coil having an inner diameter (D) not smaller than 300 mm; wherein: the inner diameter of the coil and the interheater distance (t) satisfy the condition: $0.9 \leq t/(D/2)^{1/2} \leq 4.0$.

[0006] Preferably, the $MoSi_2$-based coil heater according to the invention is disposed in coil-shaped grooves formed at an inner circumference of a ceramic mold.

[0007] In an $MoSi_2$-based coil heater according to the invention in which the inner diameter (D) of a coil and the interheater distance (t) satisfy the aforementioned relational expression, temperature uniformity can be kept high in combination with a rapid temperature rise effect by the $MoSi_2$-based heater to be able to contribute to improvement in miniaturization and yield of devices. Moreover, by satisfying the aforementioned relational expression, the $MoSi_2$-based coil heater can expand and contract freely in the grooves formed in the inner circumferential surfaces of the ceramic molds so that the $MoSi_2$-based coil heater can be kept in a free state. Thus, it is possible to provide a tubular heater module which avoids trouble such as breaking due to restriction in the ceramic molds. The $MoSi_2$-based coil heater and the tubular heater module according to the invention are effectively used in a heat treatment apparatus used in a semiconductor manufacturing process, a glass or metal melting furnace, or the like.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008]

Figs. 1A, 1B and 1C are views showing an example of a process of producing a semicircular intermediate material by bending an $MoSi_2$-based heater wire material heated electrically in manufacturing of an $MoSi_2$-based coil heater according to the invention, in which Fig. 1A shows a step before bending, Fig. 1B shows a step in the middle of bending, and Fig. 1C shows a step after completion of bending.

Fig. 2 is a view showing an example of a process of producing a coil-shaped heater by bonding semicircular members to each other by electric welding in manufacturing of the $MoSi_2$-based coil heater according to the invention.

Fig. 3 is a view showing part of a section of a tubular heater module according to the invention, in which the $MoSi_2$-based coil heater is disposed in grooves formed in each ceramic mold.

Fig. 4 is a view showing a criterion for making determination based on a life test as to whether the heater is deformed or not.

DETAILED DESCRIPTION OF THE INVENTION

[0009] A coil heater according to the invention can be produced in such a manner that intermediate members each not larger than a semicircle (a half circle) are produced from a wire material with a length up to about 2000 mm obtained by extrusion-molding an $MoSi_2$-based powder material and sintering the $MoSi_2$-based powder material in a non-oxidizing atmosphere in a vacuum furnace, and the intermediate members are formed into a coil shape by electrically butt bonding, that is, so-called diffusion bonding. Incidentally, a method of producing a coil heater according to the invention is not limited to the aforementioned method.

[1] Production of $MoSi_2$-Based Heater Wire Material

[0010] An $MoSi_2$-based heater wire material according to the invention is produced in such a manner that a green body for extrusion molding, containing $MoSi_2$ powder, binder, water, etc. is made and molded into a rod-like raw material of the order of meter (m), and the rod-like raw material is dried, debound and sintered. A water-soluble binder such as methylcellulose, swelling bentonite, or the like can be used as the binder. Sintering is performed in a temperature range of about 1350°C to about 1600°C in a non-oxidizing atmosphere though sintering varies according to the composition of the $MoSi_2$-based material and a target organization. It is preferable that the wire diameter of the $MoSi_2$-based heater wire material is in a range of 2 mm to 12 mm. When the wire diameter is larger than 12 mm, it is difficult to produce the $MoSi_2$-based heater wire material because cracks occur in the $MoSi_2$-based heater wire material in the drying step after extrusion. A terminal wire material having a diameter about twice as large as the heater wire diameter is generally used for the $MoSi_2$-based heater in order to suppress heating of a terminal portion. In consideration of producibility of the terminal wire material, it is preferable that the wire diameter of the $MoSi_2$-based heater wire material is not larger than 12 mm. It is more preferable that the wire diameter is in a range of 2 mm to 8 mm. It is further preferable that the wire diameter is in a range of 3 mm to 6 mm.

[2] Production of Semicircular Member

[0011] As intermediate members for manufacturing the $MoSi_2$-based coil heater, semicircular members are produced by electric bending. Figs. 1A, 1B, and 1C schematically show a process of producing a semicircular intermediate member. First, opposite ends of an $MoSi_2$-based heater wire material 1 are fixed to clamp portions 2 of an electric bending machine, and the wire material 1 is heated to a plasticized state by electrification through the clamp portions 2 (Fig. 1A). Then, while the heater wire material 1 is electrically heated, the clamp portions 2 are moved while pulled along guides 3 made up of several pins (the number of pins is adjusted depending on the size of the coil) (Fig. 1B). Finally, the two clamp portions 2 are moved in a direction of 90° with respect to the initial linear direction of the heater wire material 1 until the two clamp portions 2 become parallel with each other. It is preferable that the temperature is in a range of 1400°C to 1550°C, and the tensile load gives a force small enough so as not to expand the heater wire material and not to reduce the diameter of the heater wire material. After completion of bending, the heater wire material 1 is cut so as to be shaped like a semicircle and end surfaces 4 are polished so as to become perpendicular to a line tangential to the semicircle to thereby form a semicircular member 11. It is a matter of course that another shape, such as a 1/3 circular shape or a 1/4 circular shape, not larger than the semicircular shape may be used though the number of junctions increases in a bonding process as a postprocess.

[3] Production of Coil Heater

[0012] A coil heater is produced by diffusion bonding of semicircular members 11 to each other. Fig. 2 schematically shows a process of producing a coil heater by diffusion bonding. For diffusion bonding, places near to the end surfaces 4 of the semicircular members 11 are fixed by clamp portions 6 so that the places can be pressed perpendicularly to each joint surface, that is, in a direction tangential to each junction. While a predetermined pressure is applied on each joint surface 4 through the clamp portions 6, the joint surface 4 is supplied with electricity and pressurized at a high temperature so that each junction 5 is formed by welding. The clamp portions 6 are designed to be able to fix the semicircular members 11 with a curvature and selected in accordance with the curvature of the coil. The semicircular members 11 are bonded while shifted by a predetermined pitch whenever bonding is completed by a half turn. When a coil heater with a predetermined number of turns is completed, terminals are bonded to opposite end portions of the coil heater by diffusion bonding likewise.

[0013] In the invention, the coil inner diameter (D) and the interheater distance (t) in the coil heater satisfy the relation: $0.9 \leq t/(D/2)^{1/2} \leq 4.0$. Here, the interheater distance (t) is defined as the length of a gap between adjacent heaters of the coil (in other words, a distance between adjacent coils of a helix). When $t/(D/2)^{1/2}$ is smaller than 0.9, deformation becomes large undesirably. When $t/(D/2)^{1/2}$ is larger than 4.0, temperature uniformity is lowered undesirably in addition to increase of deformation. It is preferable that $0.9 \leq t/(D/2)^{1/2} \leq 3.0$ is satisfied.

[0014] One side of each terminal is machined to have the same diameter as the heater wire material so as to be able to be bonded to the heater wire material. Moreover, it is preferable that the terminal is bent into an L shape so that the other side of the terminal is exposed outside from a ceramic mold.

[4] Ceramic Mold

[0015] A heat insulating material used in the tubular heater module according to the invention is a tubular ceramic mold. An alumina material high in heat resistance is preferred as the material of the tubular ceramic mold. Although a resistance heater generally expands at heating time and contracts at cooling time, wire breaking may be often caused by abnormal heating due to local deformation when the expansion/contraction is restricted. Accordingly, an idea to prevent the expansion/contraction from being restricted is required for disposing the $MoSi_2$-based coil heater in the mold. In this respect, the $MoSi_2$-based heater according to the background art is U-shaped and takes a method of hanging down the $MoSi_2$-based heater by staples. In Fig. 3 showing part of a section of the tubular heater module according to the invention, an $MoSi_2$-based coil heater 21 is disposed in a free state in coil-shaped grooves 31 formed in a ceramic mold 30. That is, though the $MoSi_2$-based coil heater 21 is supported by one side surface of each groove, the $MoSi_2$-based coil heater 21 is allowed to move freely on the grooves without protruding out of the grooves. For this reason, it is preferable that the inner surface of each of the grooves 31 in the ceramic mold exhibits little reaction to the $MoSi_2$-based coil heater 21 and has a hardness sufficient to be not deformed. With respect to the size of each groove, the groove has a sufficient width and a sufficient depth to prevent the $MoSi_2$-based coil heater 21 from being restricted. Though not shown in the drawings, staples may be put across some of the grooves to prevent the $MoSi_2$-based coil heater from protruding out of the grooves 31.

<u>EXAMPLES</u>

Example 1

[0016] 15 % by volume of bentonite and a predetermined amount of water were added to $MoSi_2$ having a mean particle size of 2.7 μm, and kneaded to obtain a molding green body. Further, the green body obtained thus was molded into 3.4 mmΦ and 6.8 mmΦ rods by use of an extrusion molding machine. The rods were cut into a length of 800 mm. After dried, the rods were sintered at 1500°C for 2 hours in a nitrogen atmosphere to obtain about 3 mmΦ and about 6 mmΦ rod-like sintered compacts. Opposite ends of a pseudo-semicircular intermediate member having an inner diameter of 300 mm were clamped and the intermediate member was molded from the rod-like sintered compact of 3 mmΦ×700 mm by bending (bending temperature 1450°C) according to the method shown in Figs. 1A, 1B and 1C. The intermediate member was cut into a semicircular shape, and two cut surfaces of the intermediate member were polished so as to be on one plane. The semicircular members were bonded like a coil having a pitch (P) of 23 mm by butt resistance welding according to the method shown in Fig. 2. A coil heater having 20 turns was produced from 40 semicircular members. Further, terminals produced from the 6 mmΦ rod-like sintered compact by machining were bonded to opposite ends. Grooves having a groove width of 6 mm and a depth of 10 mm were formed at intervals of a pitch (P) of 23 mm in a pair of semi-cylindrical ceramic molds having an inner diameter of 294 mm, an outer diameter of 460 mm and a height of 500 mm. The coil heater having 20 turns was set to be entirely located inside the grooves in one semi-cylindrical ceramic mold. The other semi-cylindrical ceramic mold was put thereon. Joint surfaces of the molds were bonded to each other by a heat-resistant ceramic adhesive agent. Incidentally, grooves through which terminals passed were processed in the joint surfaces of the molds.

<Measurement of Temperature Distribution>

[0017] Alumina heat-insulating materials having a thickness of 100 mm were disposed in a lower portion (bottom portion) and an upper portion (lid portion) of the produced tubular heater module, and a B thermocouple for control of the temperature of the heater module was set in a position 10 mm far toward the center from the inner circumferential surface of the heater module and 250 mm far from the lid portion. Measurement of the temperature distribution was performed in the condition that the temperature of the heater module was set at 1500°C, and another B thermocouple for measurement of the temperature distribution was set in a position 50 mm far toward the center from the inner circumferential surface of the heater module and in a range of 300 mm between a position 100 mm far from the lid portion and a position 100 mm far from the bottom portion. The temperature changed slightly in accordance with the positional relation with respect to the coil but the difference ΔT between the maximum and minimum of the temperature was not larger than 3°C.

<Life Test>

[0018] As a life test for the heater module, the temperature was changed in a range of room temperature to 1500°C. A pattern of cooling to the room temperature after keeping the temperature at 1500°C for 1 hour was repeated by 500 cycles. There was no deformation particularly observed in the coil heater. There was no wire breaking. As shown in Fig. 4, whether deformation was observed or not, was determined based on the positional relation of the coil heater after the test to the heater grooves of the molds. When there was any place where a section of the heater protrudes inward from the inner circumferential ends of the heater grooves beyond the semicircular region, determination was made that deformation was present.

Examples 2 to 16 and Comparative Examples 1 to 6

[0019] Coil heaters and cylindrical heater modules were produced in the same method as in Example 1 except that the interheater distance t with respect to each coil inner diameter D was set at each distance shown in Table 1 in the condition that the wire diameter of the heater wire material was still 3 mm and the coil inner diameter D of the coil heater was set at 300 mm, 600 mm and 900 mm in respective Examples as shown in Table 1. With respect to the size of each ceramic mold, an inner diameter and an outer diameter corresponding to the coil inner diameter were set but a height was set at 500 mm in all examples. Accordingly, the number of turns was set to be a number corresponding to the interheater distance t. Grooves having a groove width of 6 mm and a depth of 10 mm were formed in the pair of semi-cylindrical ceramic molds in respective Examples as in Example 1, but those were formed at the intervals of a pitch (P) of 13 mm in case the interheater distance t was 10, a pitch (P) of 23 mm in case the interheater distance t was 20, a pitch (P) of 33 mm in case the interheater distance t was 30, a pitch (P) of 43 mm in case the interheater distance t was 40, a pitch (P) of 53 mm in case the interheater distance t was 50, a pitch (P) of 63 mm in case the interheater distance t was 60, a pitch (P) of 73 mm in case the interheater distance t was 70, a pitch (P) of 83 mm in case the interheater distance t was 80 or a pitch (P) of 93 mm in case the interheater distance t was 90. In each example and each comparative example, the temperature distribution was measured and the life test was further performed in the same manner as in Example 1. Results thereof, inclusively of the result in Example 1, are shown in Table 1. Here, as a result of the temperature distribution, the case where the temperature distribution is not larger than 3°C is evaluated as ○○, the case where the temperature distribution is larger than 3°C but not larger than 5°C is evaluated as ○, and the case where the temperature distribution is larger than 5°C is evaluated as x. As a result of the life test, the aforementioned case where there is no deformation is evaluated as ○, and the case where there is any deformation is evaluated as x. As comprehensive evaluation, the case where either the result of the temperature distribution or the result of the life test is × is evaluated as ×, the case where both the result of the temperature distribution and the result of the life test are ○ is evaluated as ○, and the case where the result of the temperature distribution is ○○ is evaluated as ○○.

[Table 1]

	Inner Diameter D, mm	Interheater Distance t, mm	$t/(D/2)^{1/2}$	Temperature Distribution	Accelerated Life Test	Comprehensive Evaluation
Example 1	300	20	1.6	○○	○	○○
Example 2	300	30	2.4	○○	○	○○
Example 3	300	40	3.3	○	○	○
Example 4	600	20	1.2	○○	○	○○
Example 5	600	30	1.7	○○	○	○○
Example 6	600	40	2.3	○○	○	○○
Example 7	600	50	2.9	○○	○	○○
Example 8	600	60	3.5	○	○	○
Example 9	600	70	4	○	○	○
Example 10	900	20	0.9	○○	○	○○
Example 11	900	30	1.4	○○	○	○○
Example 12	900	40	1.9	○○	○	○○

(continued)

	Inner Diameter D, mm	Interheater Distance t, mm	$t/(D/2)^{1/2}$	Temperature Distribution	Accelerated Life Test	Comprehensive Evaluation
Example 13	900	50	2.4	○○	○	○○
Example 14	900	60	2.8	○○	○	○○
Example 15	900	70	3.3	○	○	○
Example 16	900	80	3.8	○	○	○
Comparative Example 1	300	10	0.8	○	×	×
Comparative Example 2	300	50	4.1	×	×	×
Comparative Example 3	600	10	0.6	○	×	×
Comparative Example 4	600	80	4.6	×	×	×
Comparative Example 5	900	10	0.5	○	×	×
Comparative Example 6	900	90	4.2	×	×	×

[0020] When $t/(D/2)^{1/2}$ was smaller than 0.9, the temperature distribution was good but deformation still occurred. When $t/(D/2)^{1/2}$ was larger than 4.0, temperature distribution and deformation were undesirable as a result. When $t/(D/2)^{1/2}$ was in a range of 0.2 to 2.9, a particularly desirable result was obtained.

Claims

1. An $MoSi_2$-based coil heater comprising a coil having an inner diameter (D) not smaller than 300 mm, wherein the inner diameter (D) of the coil and an interheater distance (t) satisfy the following condition:

$$0.9 \leq t/(D/2)^{1/2} \leq 4.0.$$

2. The $MoSi_2$-based coil heater according to Claim 1, wherein the inner diameter (D) of the coil and the interheater distance (t) satisfy the following condition:

$$0.9 \leq t/(D/2)^{1/2} \leq 3.0.$$

3. The $MoSi_2$-based coil heater according to Claim 1, wherein a wire diameter of the coil is from 2 mm to 12 mm.

4. The $MoSi_2$-based coil heater according to Claim 2, wherein a wire diameter of the coil is from 2 mm to 12 mm.

5. A tubular heater module comprising:

   a ceramic mold; and
   the $MoSi_2$-based coil heater according to any one of Claims 1 to 4 disposed in coil-shaped grooves formed at an inner circumference of the ceramic mold.

FIG. 1A

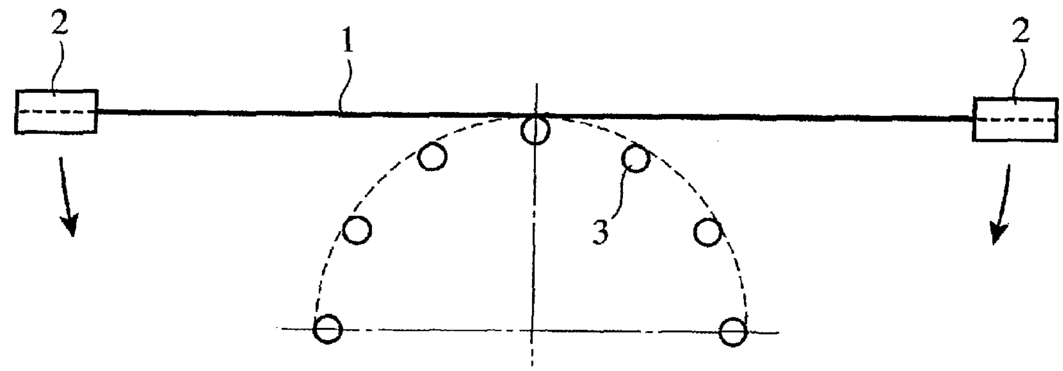


FIG. 1B

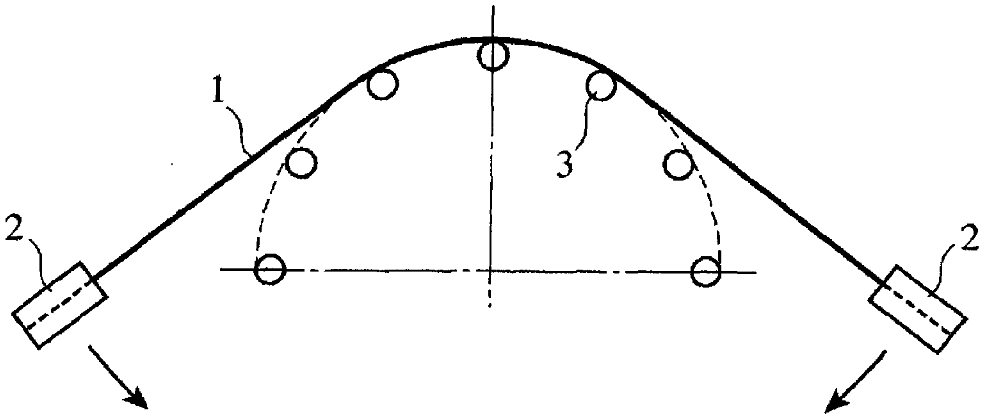


FIG. 1C

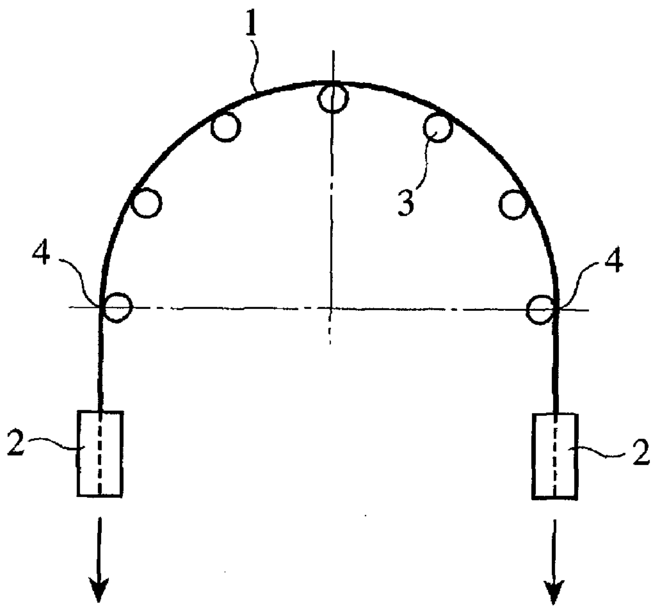


FIG. 2

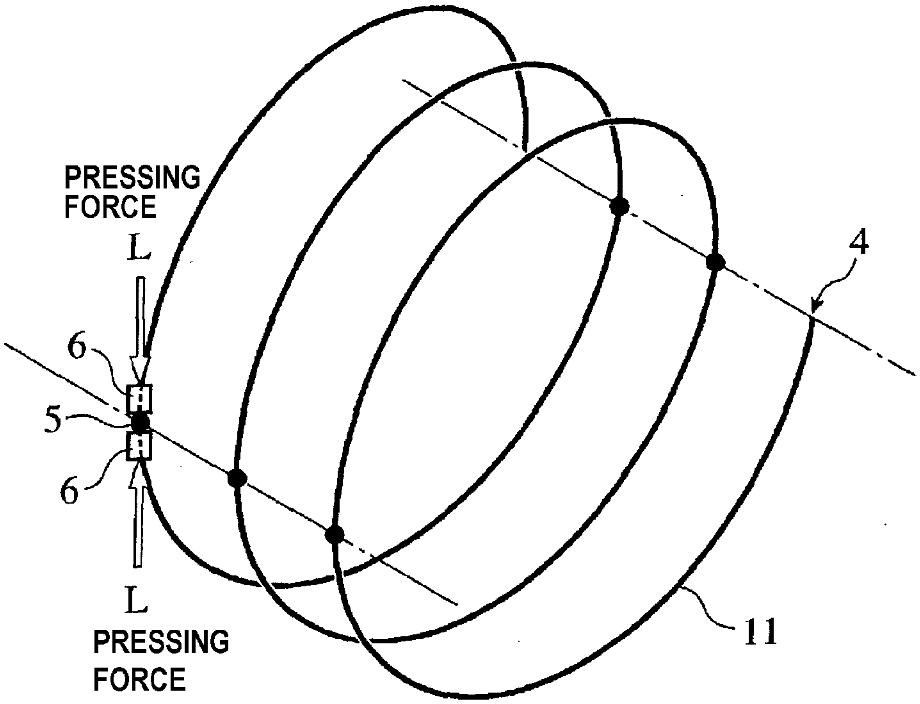


FIG. 3

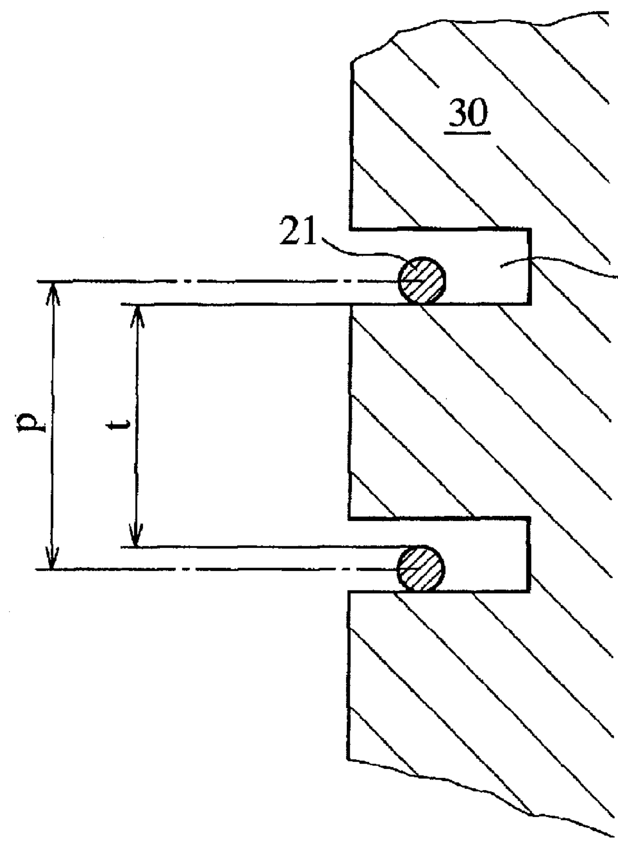
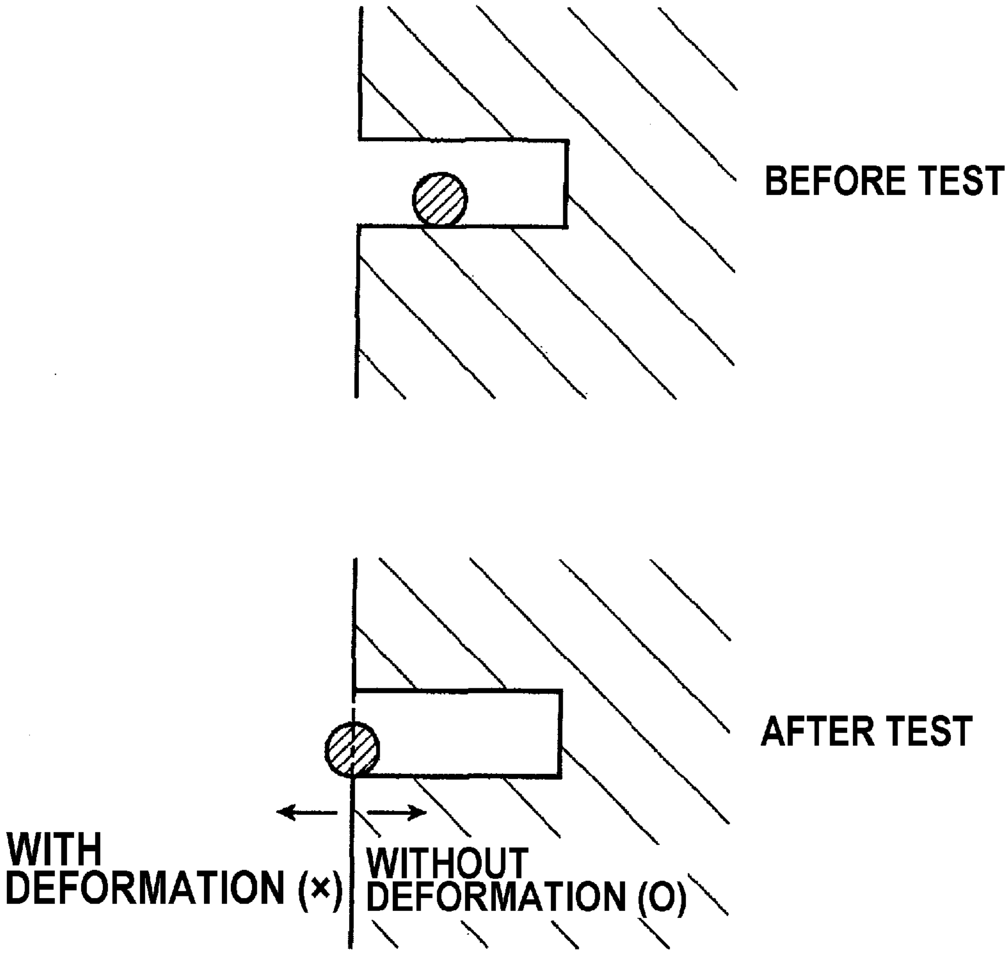


FIG. 4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EUROPEAN SEARCH REPORT

Application Number
EP 13 30 6161

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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A	US 2007/145038 A1 (VISSA RAMGOPAL [US] ET AL VISSA RAMGOPAL [IN] ET AL) 28 June 2007 (2007-06-28) * paragraph [0045] - paragraph [0047]; figures 2,3,6 * -----	1-5	TECHNICAL FIELDS SEARCHED (IPC)
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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	6 November 2013	Gea Haupt, Martin

CATEGORY OF CITED DOCUMENTS

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

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