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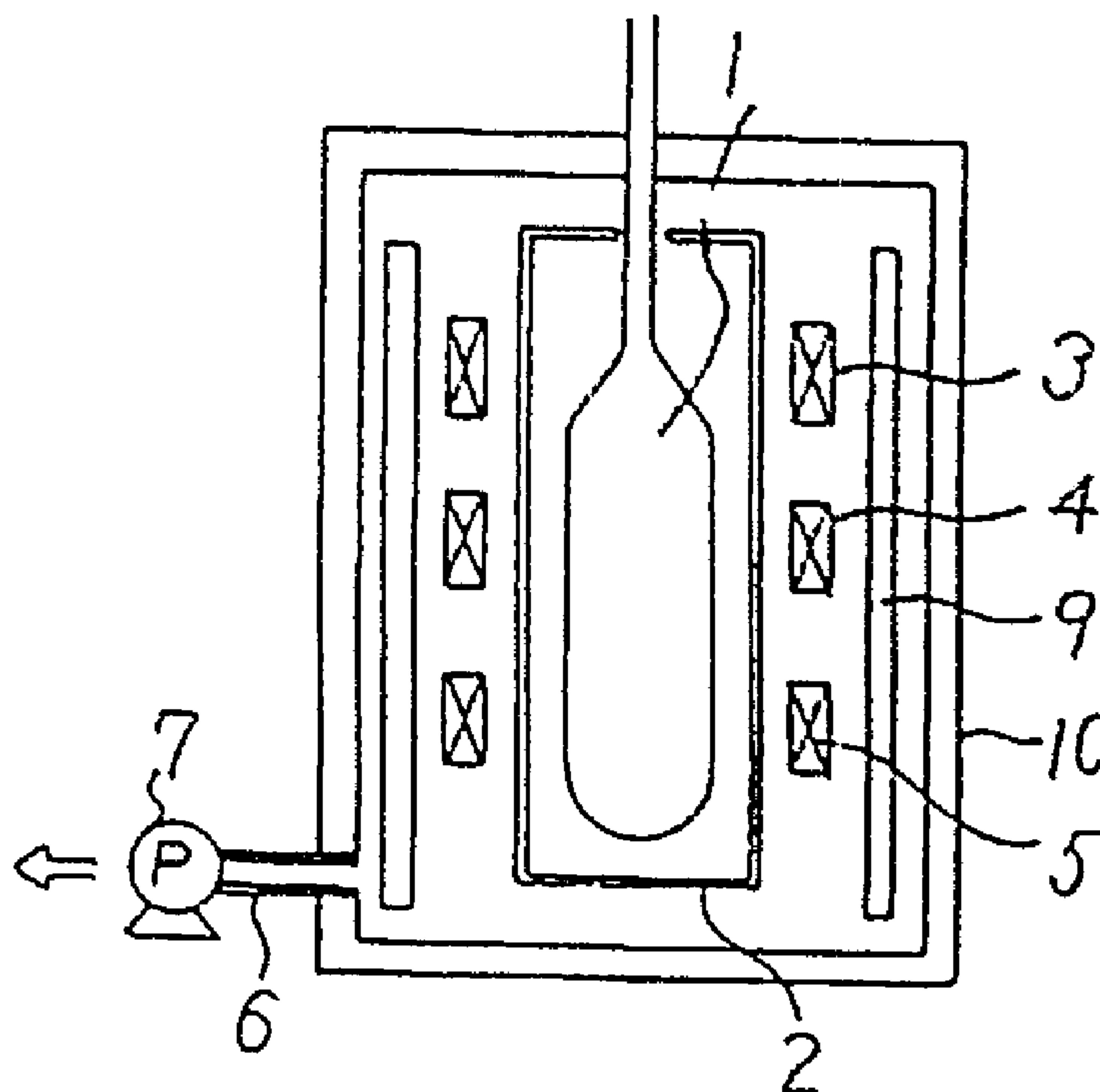
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(54) Title: PROCESS FOR PRODUCTION OF GLASS PREFORM FOR OPTICAL FIBER



(57) Abrégé/Abstract:

The present invention relates to an improved process for providing the production of a glass preform for an optical fiber which includes substantially no bubbles therein and also which has a substantially uniform shape. The process for the production of a glass preform which comprises at least silica for an optical fiber is comprised of producing a body comprising a rod on which fine glass particles are deposited, preferably by a vapour phase reaction and heating the body to vitrify the body under a reduced atmosphere or a vacuum so that the glass preform is produced. The process includes a first heating step of degassing the body at a first heating temperature, a second heating step of shrinking the body at a second heating temperature which is higher than the first heating temperature and which is lower than a third heating temperature, and a third heating step of vitrifying the shrunken body at the third temperature which corresponds to a vitrification temperature of the fine glass particles so as to produce the glass preform.



ABSTRACT

The present invention relates to an improved process for providing the production of a glass preform for an optical fiber which includes substantially no bubbles therein and also which has a substantially uniform shape. The process for the production of a glass preform which comprises at least silica for an optical fiber is comprised of producing a body comprising a rod on which fine glass particles are deposited, preferably by a vapour phase reaction and heating the body to vitrify the body under a reduced atmosphere or a vacuum so that the glass preform is produced. The process includes a first heating step of degassing the body at a first heating temperature, a second heating step of shrinking the body at a second heating temperature which is higher than the first heating temperature and which is lower than a third heating temperature, and a third heating step of vitrifying the shrunken body at the third temperature which corresponds to a vitrification temperature of the fine glass particles so as to produce the glass preform.

PROCESS FOR PRODUCTION OF GLASSPREFORM FOR OPTICAL FIBERBACKGROUND OF THE INVENTION

The present invention relates to a process for the production of a glass preform for an optical fiber. In particular the invention relates to a glass preform comprising at least silica from which an optical fiber is directly drawn without any additional treatment of the preform or the need of an intermediate glass preform from which an optical fiber is drawn after some additional treatment of the preform.

DESCRIPTION OF THE RELATED ART

A glass preform comprising at least silica for an optical fiber is produced by heating, at an elevated temperature, a body comprising a rod on which fine glass particles are deposited by vapour phase reactions in an electrical furnace so that the glass particle deposited body is vitrified (namely consolidated). This deposition process is known in the field as, for example, the outside chemical vapour deposition process and the vapour phase axial deposition (VAD) process. Such a vitrification process has been conventionally carried out by heating the body at an atmospheric pressure in an atmosphere filled with helium gas or an inert gas containing a small amount of a halogen gas. In such a process, a gas contained in the fine glass particles of the body remains confined within the body when the body is vitrified, which causes the problem that the vitrified body (namely the vitrified preform) includes bubbles therein.

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In order to solve such a problem, Japanese Patent Kokai Publication No. 63-21025 discloses a process for the vitrification of a glass preform in which the preform is heated in a vacuum or in a reduced atmosphere. In this process, since the atmosphere is reduced or a strong vacuum is present, the body comprising the fine particles is degassed so that it is expected that no bubbles remain in the vitrified body.

SUMMARY OF THE INVENTION

It is, therefore, an object of the present invention to provide an improved process for the production of a glass preform for an optical fiber which includes substantially no bubbles therein and also has a substantially uniform shape so as to overcome the problem discussed with regard to the prior art.

According to the present invention, there is provided a process for the production of a glass preform which comprises at least silica for an optical fiber comprising producing a body comprising a rod on which fine glass particles are deposited and heating the body to vitrify under a reduced atmosphere so that the glass preform is produced; in which process the heating comprises a first heating step of degassing the body at a first heating temperature, a second heating step of shrinking the body at a second heating temperature which is higher than the first heating temperature and which is lower than a third heating temperature, and a third heating step of vitrifying the shrunken body at the third temperature which corresponds to a vitrification temperature of the fine glass particles so as to produce the glass preform, wherein the second heating step is carried out

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by differently heating portions of the body from one another along the length direction of the body so that a temperature of each portion of the body is controlled independently from one another and a temperature of a lower portion of the body
5 is higher than that of an upper portion of the body.

In the present invention, the fine glass particle deposited body may be produced by any conventional process such as the VAD process, the CVD process and the like. In principle, the atmosphere during the heating steps is
10 continuously or successively evacuated by a conventional means such as a vacuum pump from an apparatus in which the heating steps are carried out. Optionally, the atmosphere may comprise an inert gas, a halogen gas, a halogenated gas or a mixture thereof for a predetermined purpose such as dehydration,
15 fluorine doping and the like, if necessary, according to conventional knowledge, provided that the following specified pressure (degree of vacuum) and the following specified heating temperature are ensured.

For example, the body may be a composite which is
20 produced by depositing the fine glass particles around a glass rod having an at least double waveguide structure of which the refractive index is lower in its outer portion than its core portion.

In accordance with another aspect of the present
25 invention there is provided a process for producing a silica-containing glass preform for an optical fiber, comprising the steps of: depositing glass particles on a rod to form a body, the body having a longitudinal axis and at least a lower portion and an upper portion; and heating the body at a
30 reduced pressure, wherein said heating step comprises: a

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first heating step of degassing the body at a first heating temperature, a second heating step of shrinking the body by subjecting the upper and lower portions of the body to respective second heating temperatures which are higher than
5 the first heating temperature, the second heating temperature for the lower portion of the body being higher than the second heating temperature for the upper portion of the body, and a third heating step of consolidating the body at a third temperature which is higher than the second heating
10 temperatures and which consolidates the fine glass particles.

In accordance with yet another aspect of the present invention there is provided a process for producing a silica-containing glass preform for an optical fiber, comprising the steps of: depositing glass particles on a rod to form a body,
15 the body having a longitudinal axis, upper and lower ends, and a plurality of portions located therebetween; and heating the body at a reduced pressure, wherein said heating step comprises: a first heating step of degassing the body at a first heating temperature; a second heating step of shrinking
20 the body by subjecting each of said plurality of portions to respective second heating temperatures which are higher than the first heating temperature, the second heating temperature for any selected portion being higher than the second heating temperature for the portions located closer to the upper end,
25 and a third heating step of consolidating the body at a third temperature which is higher than any of the second heating temperatures and which consolidates the fine glass particles.

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BRIEF DESCRIPTION OF THE DRAWINGS

The present invention will be described in detail hereinbelow with the aid of the accompanying drawings, in which:

Fig. 1 is a schematic diagram of a heating furnace used in one embodiment of the present process;

Fig. 2 is a schematic diagram showing a conventional heating furnace;

5 Fig. 3 is a graph which schematically shows a temperature condition in a conventional heating process to make a vitrified glass preform;

10 Fig. 4 is a schematic diagram which shows the shape of a produced glass preform in a conventional process which has different diameters along its length;

15 Fig. 5 is a graph showing the relationship between a first heating temperature and an attained degree of vacuum (namely an attained reduced pressure) in a first heating step and also the relationship between the first heating temperature and the number of bubbles contained in a vitrified glass preform per unit length of the preform;

Fig. 6 is a graph showing the relationship between a retention time at a second heating temperature and an outer diameter difference of a vitrified glass preform;

20 Fig. 7 is a graph showing the relationship between a second heating temperature and an outer diameter of a vitrified glass preform; and

25 Fig. 8 is a graph showing the relationship of a temperature difference between a lower heating element and an upper heating element versus an outer diameter difference of a vitrified preform between around a lower portion and around a middle portion of the preform.

Prior to discussing the present invention in detail a discussion of the prior art will be useful.

Fig. 2 schematically shows a conventional apparatus for the production of a glass preform for an optical fiber. The apparatus comprises a vacuum vessel 10 in which is placed a muffle tube 2. The muffle tube 2 surrounds a fine particle deposited body 1. A heating member 8 is positioned outside the muffle tube 2. Thermal insulation (a heat shield) 9 is located between the heating element 8 and a wall of the vessel 10. The vessel 10 is further equipped with an evacuation port to which a vacuum pump 7 is connected through a vacuum line 6 so the vessel can be evacuated or the atmosphere reduced. The glass particle deposited body 1 inserted in the muffle tube 2 is vitrified therein by heating it with the heating member 8.

However, it has been found that bubbles remain in the preform when the apparatus as shown in Fig. 2 is used for the vitrification of a glass preform in the form of a rod in a strong vacuum or a reduced atmosphere and a predetermined temperature condition is used which is schematically shown in Fig. 3. The vitrification temperature is usually in a range of 1550 to 1650°C in a conventional process. The produced glass preform has a longitudinally non-uniform shape in its outer diameter as schematically shown in Fig. 4, which shows both end portions of the preform thicker and the central portion thereof thinner.

In order to produce a glass article having a good quality from the glass rod produced by the above process, the remaining bubbles should be reduced or eliminated and the outer diameter of the vitrified glass rod should be uniform.

5 The present invention will now be discussed.

According to the present invention, the first heating step is preferably continued in a temperature range of 1000 to 1300°C while the atmosphere in the furnace is evacuated with a vacuum pump until the pressure of the atmosphere (the degree
10 of vacuum) reaches 10 Pa or less and more preferably 5 Pa or less.

The second heating step is preferably carried out at a pressure of 10 Pa or less and more preferably 5 Pa or less while keeping the second heating temperature in a range of
15 1320 to 1480°C and more preferably 1350 to 1450°C for from 10 to 120 minutes and more preferably from 40 to 90 minutes while evacuation is continued if necessary.

In addition, a heating member which is located around a muffle tube and which heats the degassed body may comprise a
20 plurality of heating elements which are located separately along the longitudinal direction of the body and which can control the heating temperature of degassed body portions independently from one another so that the heating elements can heat the degassed body portions to different temperatures
25 along the length of the preform. Also, the plurality of the

heating elements may be so arranged that the lower portion of the degassed body is heated to a temperature equal to or higher than a temperature of the upper portion of the degassed body which is located above the lower portion.

5 The third heating step is carried out at a pressure of 10 Pa or less and more preferably 5 Pa or less in a temperature range of from 1490 to 1600°C and more preferably in a temperature range of from 1500 to 1550°C for from 1 to 60 minutes and more preferably from 5 to 20 minutes.

10 The present inventors carried out the heating treatment of a fine glass particle deposited body in a reduced atmosphere while evacuated with the vacuum pump, and obtained a relationship between the first heating temperature and an attained degree of vacuum. The solid line in the graph of
15 Fig. 5 shows such a relationship after 5 hours have passed from the beginning of the heating. As seen from the graph, when the first heating temperature is 1000°C or more, the attained degree of vacuum is 10 Pa or less.

 After the first heating step, the body was heated to
20 1550°C so as to vitrify the body and then the number of bubbles contained in the vitrified body was obtained. The results are also shown in the graph of Fig. 5 by the broken line, which shows the relationship between the first heating temperature and the number of the bubbles finally confined in
25 the vitrified body per one meter length of the body. As seen from the broken line, when the first heating temperature is in

a range of from 1000 to 1300°C, nearly no bubbles are left in the body. On the other hand, when the first temperature is outside of such a specified range, the number of the contained bubbles is sharply increased.

5 Although not bound by any theory, the reason why the above results were obtained could be as follows: The degassing rate itself from the fine glass particle deposited body is larger at a higher first heating temperature. However, the body shrinks more readily at the higher degassing
10 temperature so that gases do not tend to be removed due to the shrinkage. Thus, since the degassing rate is smaller at the first heating temperature below 1000°C or since the shrinkage already begins before a sufficient amount of the gas has been removed at the first heating temperature above 1300°C, the
15 bubbles are likely to be confined in the body. Therefore, on the basis of the results shown in Fig. 5, it is critical, to produce a vitrified glass preform with substantially no bubbles, that the glass body is heated to a temperature range of from 1000 to 1300°C until the pressure inside the furnace
20 reaches 10 Pa or less, and then the body is further heated to a higher temperature so as to vitrify the body.

 In the production of an optical fiber preform, one more factor which is as important as the number of the bubbles contained in the preform is uniformity of the outer diameter
25 of the glass preform along its length. The present inventors have found that there is a strong correlation of the retention time during which the degassed body is keep at the second

heating temperature in a range of from 1320 to 1480°C versus the outer diameter difference between a location around a middle portion and a location around a lower portion of the body (namely, "a diameter around the lower portion of the body" minus "a diameter around the central portion of the body") in the finally produced preform. Such a correlation is shown in Fig. 6. In addition, the inventors have further found a correlation between the second heating temperature and the outer diameter of the middle portion of the glass preform body after final vitrification, and the correlation is shown in Fig. 7. This is because shrinkage along a radial direction of the body is larger at a higher temperature so that such a correlation is obtained. On the basis of the results shown in Fig. 7, it is effective to carry out the second heating step so as to increase the temperature of the lower portion of the degassed body relative to the temperature of the upper portion of the degassed body and/or to decrease the temperature of the upper portion of the degassed body relative to the temperature of the lower portion of the degassed body in order to prevent a larger outer diameter of the lower portion of the glass body.

Therefore, one preferred embodiment according to the present invention may use a heating furnace as shown in Fig. 1 in which a plurality of, for example, three, heating elements 3, 4 and 5 are provided so as to constitute a heating member. Once again the fine glass particle deposited body is denoted 1, a muffle tube is denoted 2, in which the body 1 is inserted

and heated, An upper heating element 3 is provided, along with a middle heating element 4, and a lower heating element 5. A degassing conduit 6 is connected to a vacuum pump 7. A heat shield 9 is located outside the heating elements in a vacuum vessel 10. Once again the heating member is divided into a plurality of heating elements, for example three heating elements 3, 4 and 5, located along the longitudinal direction of the degassed body and the heating temperature of each heating element is controlled independently from one another, so that the lower portion of the body may be heated to a temperature higher than a temperature to which the upper portion is heated. Using the heating furnace as shown in Fig. 1, a relationship, as shown in Fig. 8, was obtained as to the heating temperature difference between the lower heating element and the upper heating element versus the outer diameter difference between a location around the lower portion and the middle portion of the body. It has been found from the results that the diameter difference becomes less than 6 mm when the temperature of the lower heating element is higher than that of the upper heating element by 50°C or more.

The third heating step is carried out in a range of from 1490 to 1600°C which corresponds to the vitrification temperature of the glass particle deposited body. It has been found that a longer period of the third heating step than required causes droop of the vitrified preform due to its

weight since the glass is very soft at the third heating temperature. Therefore, the period of the third heating step should be as short as required, and thus the period is preferably in a range of from 1 to 60 minutes and more preferably from 5 to 20 minutes.

As described above, since the fine glass particle deposited body is heated under such conditions that the gas contained in the body is sufficiently removed according to the present invention, substantially no bubbles are left in the vitrified glass body. In addition, the vitrified glass body having a uniform outer diameter is produced by raising the heating temperature of the lower portion of the fine glass particle deposited body by using a heating member which is divided into a plurality of independent heating elements.

EXAMPLES

The present invention will be, hereinafter, described with reference to the following Examples and Comparative Example. It should be understood that the present invention is not limited to the Examples.

Example 1

Using the heating furnace as shown in Fig. 1, a fine silica glass particle deposited body 1, produced by the VAD process, was heated to be vitrified according to the present invention. The body had an outer diameter of 200 mm and a length of 1000 mm. The body was inserted in a muffle tube 2 located in a vacuum vessel 10 and heated to a temperature of 1200°C while the atmosphere was evacuated from the furnace.

When a temperature of 1200°C was reached, the pressure in the furnace was 20 Pa. However, after the furnace was kept at that temperature for three hours, the pressure was reduced to 3 Pa. Then, the temperature of the upper heating element 3 was increased to 1330°C, the temperature of the middle heating element 4 was increased to 1400°C, and the temperature of the lower heater was increased to 1470°C. Such an increased temperature condition was kept for one hour, and the pressure inside the furnace was constant at about 3 Pa during said one hour. Thereafter, the body was heated to 1550°C and kept at that temperature for 15 minutes, and the pressure inside the furnace was still constant at about 3 Pa. Then the body was cooled and observed. The body was of such a good quality that it had substantially no bubbles over its entire length. The outer diameter of the body was substantially uniform such that it was 90 mm $\pm$ 1.2 mm over its entire length.

Comparative Example 1

The same fine glass particle deposited body as in Example 1 was inserted in a muffle tube 2 placed in a vacuum vessel as shown in Fig. 2, and the pressure inside the furnace was kept at 30 Pa and heated to a temperature of 1600°C for 30 minutes. Then, the body was cooled and taken out of the furnace. The body contained small bubbles having a size of 0.1 mm or less over its entire length and had a deformed shape such that the outer diameter at a location around the center portion was as thin as 76 mm, and the outer diameter at a location around the lower portion was as thick as 90 mm.

Example 2

The same fine glass particle deposited body as in Example 1 was inserted in the muffle tube 2 placed in the apparatus as shown in Fig. 1 which had already been preheated to a temperature of 800°C. When the body was heated to 1300°C and kept at such a temperature for two hours while evacuated in the first heating step, the pressure inside the furnace was 5 Pa. Then, the body was further heated so as to initiate the second heating step in which the upper heating element was set to 1320°C, the middle one to 1390°C and the lower one to 1470°C and such a different temperature condition was kept for one hour. Thereafter, in order to carry out the third heating step, the upper heating element was set to 1550°C, the middle one to 1525°C and the lower one to 1500°C and such a different increased temperature condition was kept for 30 minutes. The preform was then cooled. During the third heating step, the pressure inside the furnace was constant at 5 Pa. The produced glass preform was of such good quality that it had substantially no bubbles over its entire length. The outer diameter of the body was substantially uniform such that it was 90 mm $\pm$ 0.8 mm over its entire length.

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Claims:

1. A process for the production of a glass preform which comprises at least silica for an optical fiber comprising producing a body comprising a rod on which fine glass
5 particles are deposited and heating the body to vitrify under a reduced atmosphere so that the glass preform is produced; in which process the heating comprises a first heating step of degassing the body at a first heating temperature, a second heating step of shrinking the body at a second heating
10 temperature which is higher than the first heating temperature and which is lower than a third heating temperature, and a third heating step of vitrifying the shrunken body at the third temperature which corresponds to a vitrification temperature of the fine glass particles so as to produce the
15 glass preform, wherein the second heating step is carried out by differently heating portions of the body from one another along the length direction of the body so that a temperature of each portion of the body is controlled independently from one another and a temperature of a lower portion of the body
20 is higher than that of an upper portion of the body.
2. The process according to claim 1 wherein the first heating step is continued in a temperature range of from 1000 to 1300°C until a degree of vacuum of reduced atmosphere reaches 10 Pa or less.
- 25 3. The process according to claim 1 wherein the second heating step comprises heating the body in a temperature range

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of from 1320 to 1480°C at a degree of vacuum of reduced atmosphere of 10 Pa or less for from 10 to 120 minutes.

4. The process according to claim 1 wherein the third heating step comprises heating the body in a temperature range
5 of from 1490 to 1600°C at a degree of vacuum of reduced atmosphere of 10 Pa or less for from 1 to 60 minutes.

5. The process according to claim 1 wherein the body is a composite which is produced by depositing the fine glass particles around a glass rod having an at least double
10 waveguide structure of which refractive index is lower in its outer portion than its core portion.

6. A process for producing a silica-containing glass preform for an optical fiber, comprising the steps of:

depositing glass particles on a rod to form a body, the
15 body having a longitudinal axis and at least a lower portion and an upper portion; and

heating the body at a reduced pressure, wherein said heating step comprises:

a first heating step of degassing the body at a
20 first heating temperature,

a second heating step of shrinking the body by subjecting the upper and lower portions of the body to respective second heating temperatures which are higher than the first heating temperature, the second heating temperature
25 for the lower portion of the body being higher than the second heating temperature for the upper portion of the body, and

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a third heating step of consolidating the body at a third temperature which is higher than the second heating temperatures and which consolidates the fine glass particles.

7. The process according to claim 6 wherein the first
5 heating temperature is between 1000°C and 1300°C, and wherein said first heating step is continued until a degree of vacuum of the reduced pressure reaches 10 Pa or less.

8. The process according to claim 6 wherein the second
heating temperatures range between 1320°C and 1480°C, and
10 wherein said second heating step is conducted at a degree of vacuum of the reduced pressure of 10 Pa or less for 10 to 120 minutes.

9. The process according to claim 6 wherein the third
heating temperature is between 1490°C and 1600°C, and wherein
15 said third heating step is conducted at a degree of vacuum of the reduced pressure of 10 Pa or less for 1 to 60 minutes.

10. The process according to claim 6 wherein the body is a
composite, and wherein the rod is glass and has a core portion
and an outer portion and a refractive index which is lower in
20 the outer portion than in the core portion.

11. A process for producing a silica-containing glass preform
for an optical fiber, comprising the steps of:

depositing glass particles on a rod to form a body, the
body having a longitudinal axis, upper and lower ends, and a
25 plurality of portions located therebetween; and

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heating the body at a reduced pressure, wherein said heating step comprises:

a first heating step of degassing the body at a first heating temperature;

5 a second heating step of shrinking the body by subjecting each of said plurality of portions to respective second heating temperatures which are higher than the first heating temperature, the second heating temperature for any selected portion being higher than the second heating
10 temperature for the portions located closer to the upper end, and

a third heating step of consolidating the body at a third temperature which is higher than any of the second heating temperatures and which consolidates the fine glass
15 particles.

12. The process according to claim 11 wherein the first heating temperature is between 1000°C and 1300°C, and wherein said first heating step is continued until a degree of vacuum of the reduced pressure reaches 10 Pa or less.

20 13. The process according to claim 11 wherein the second heating temperatures range between 1320°C and 1480°C, and wherein said second heating step is conducted at a degree of vacuum of the reduced pressure of 10 Pa or less for 10 to 120 minutes.

25 14. The process according to claim 11 wherein the third heating temperature is between 1490°C and 1600°C, and wherein

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said third heating step is conducted at a degree of vacuum of the reduced pressure of 10 Pa or less for 1 to 60 minutes.

15. The process according to claim 11 wherein the body is a composite, and wherein the rod is glass and has a core portion
5 and an outer portion and a refractive index which is lower in the outer portion than in the core portion.

FIG. 1

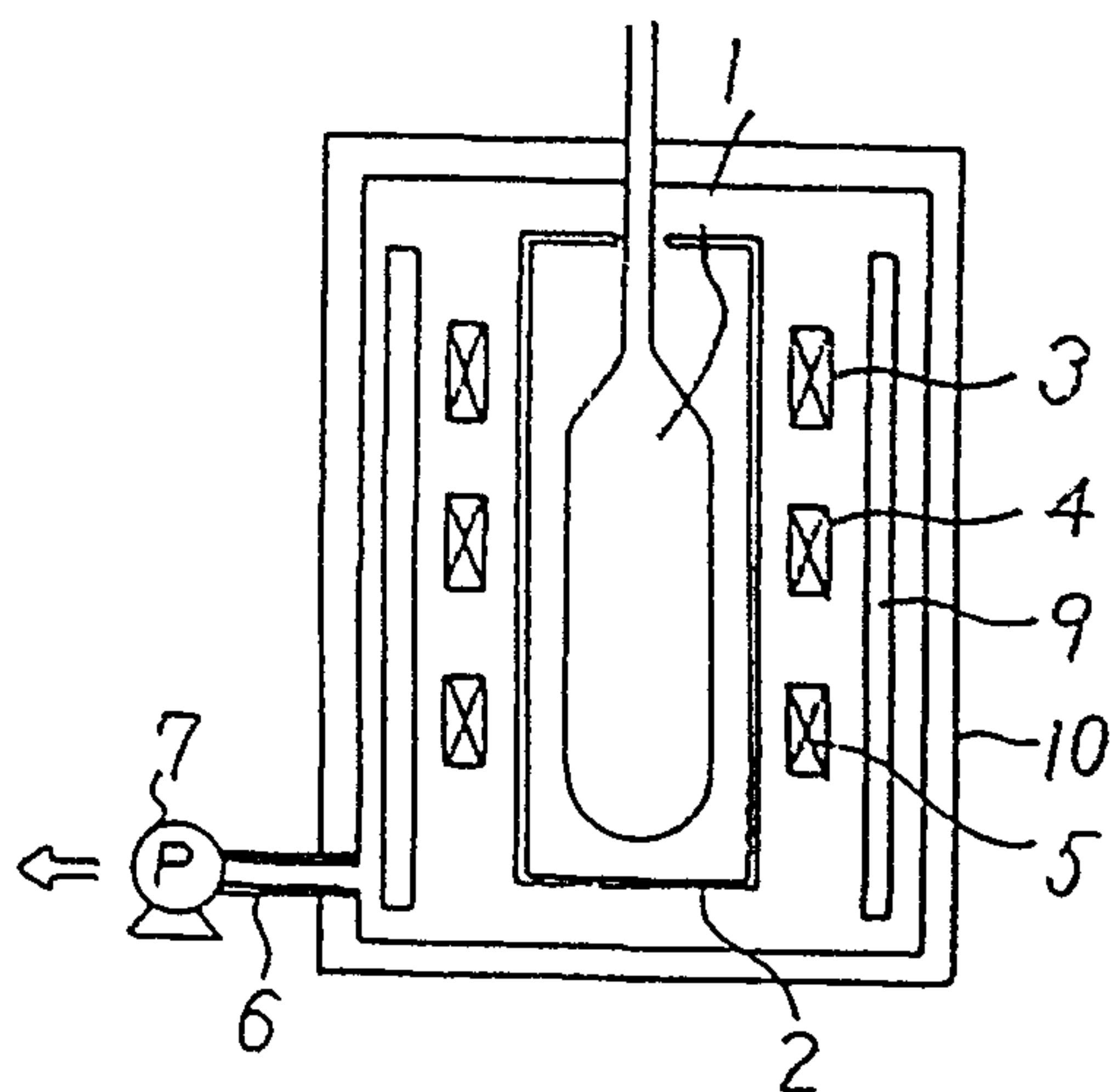


FIG. 2

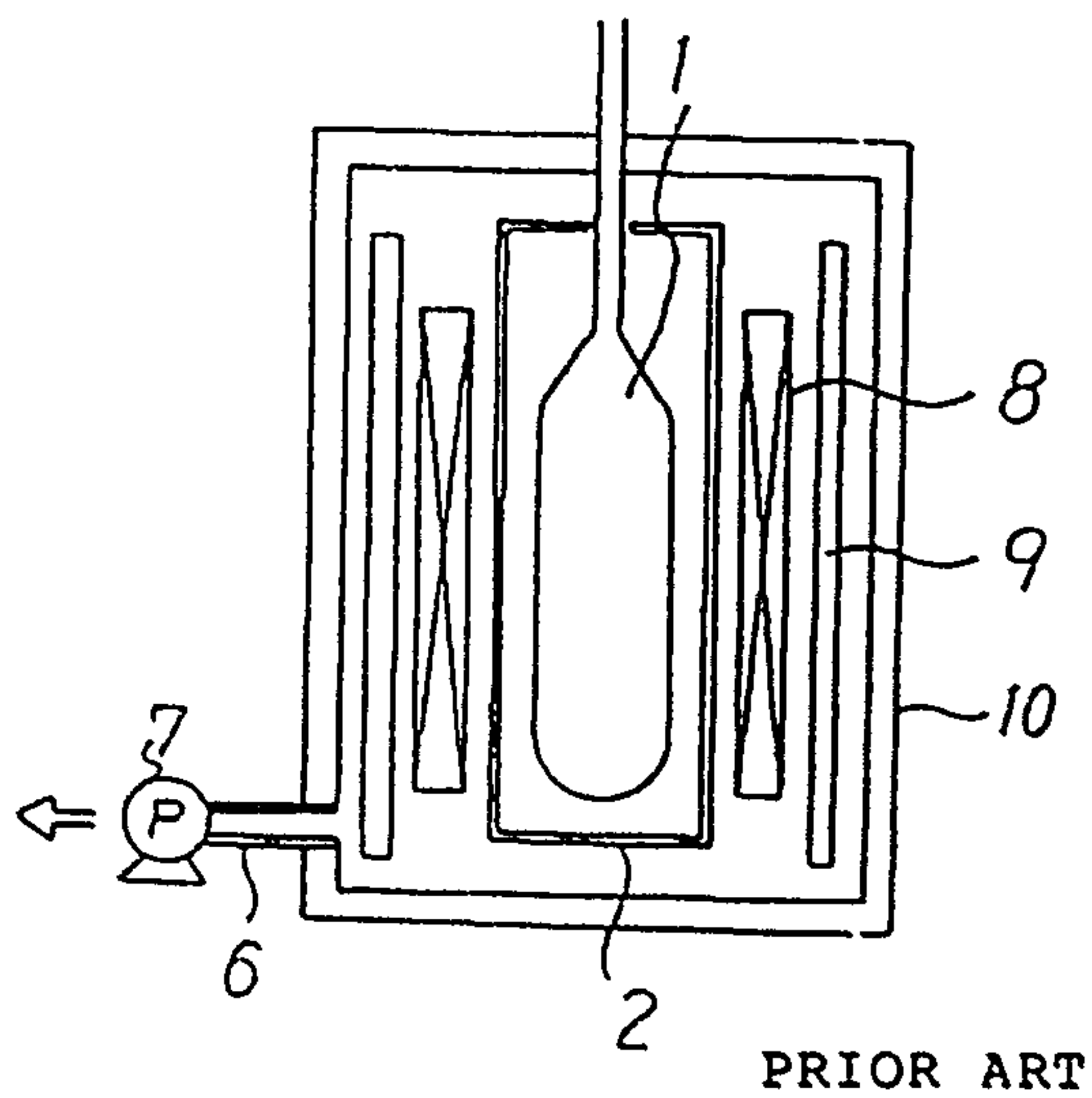


FIG. 3

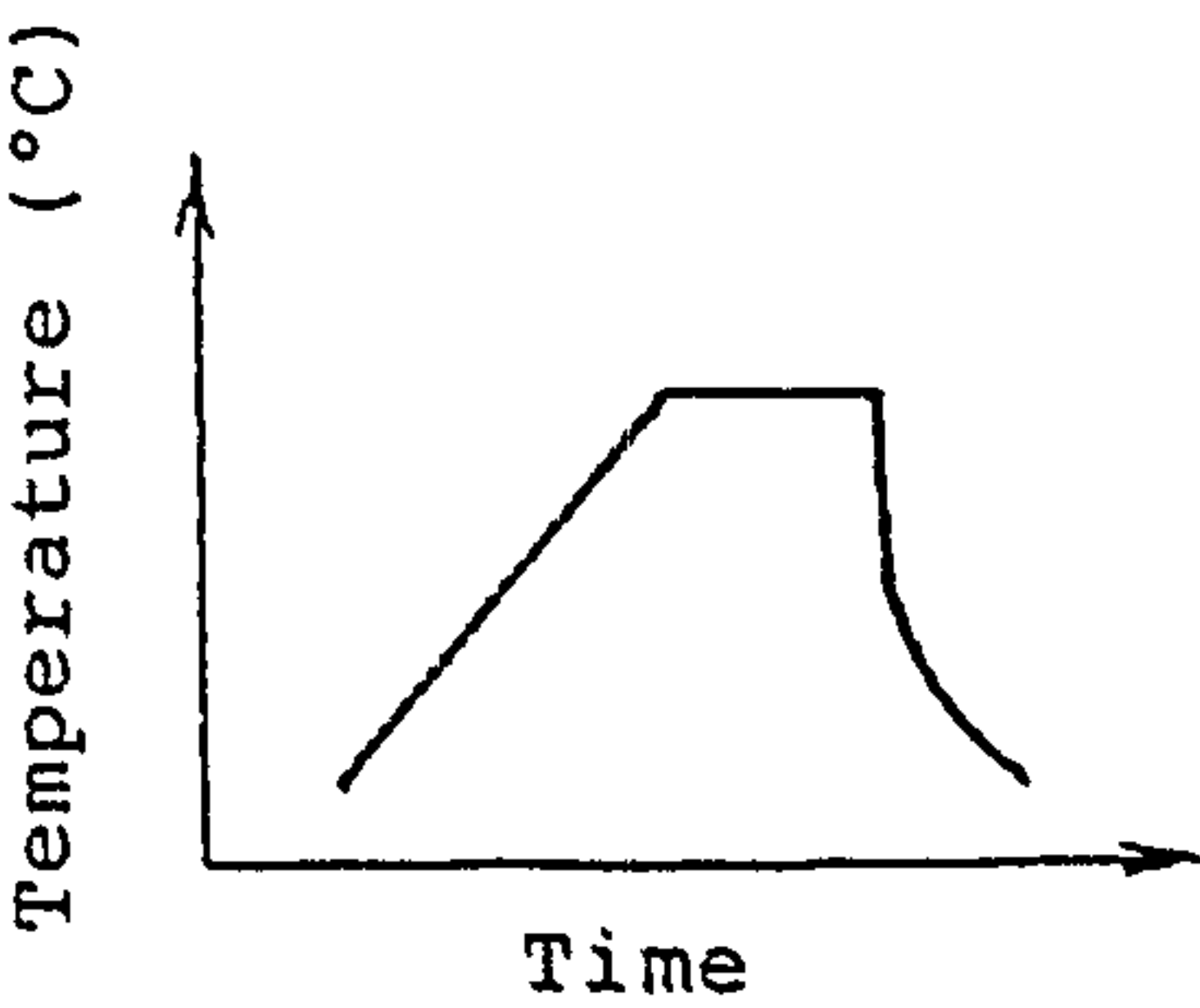


FIG. 4

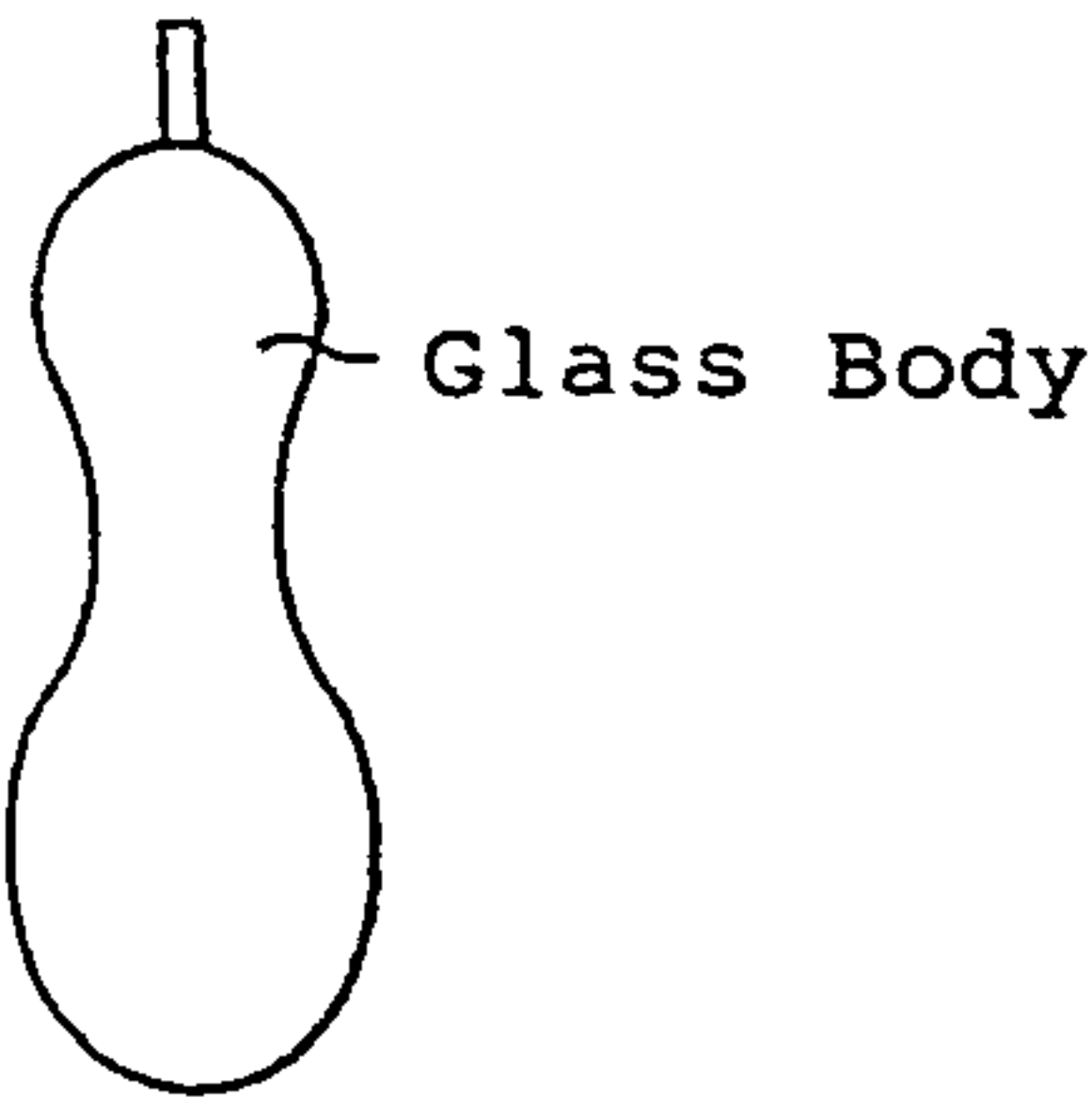


FIG. 5

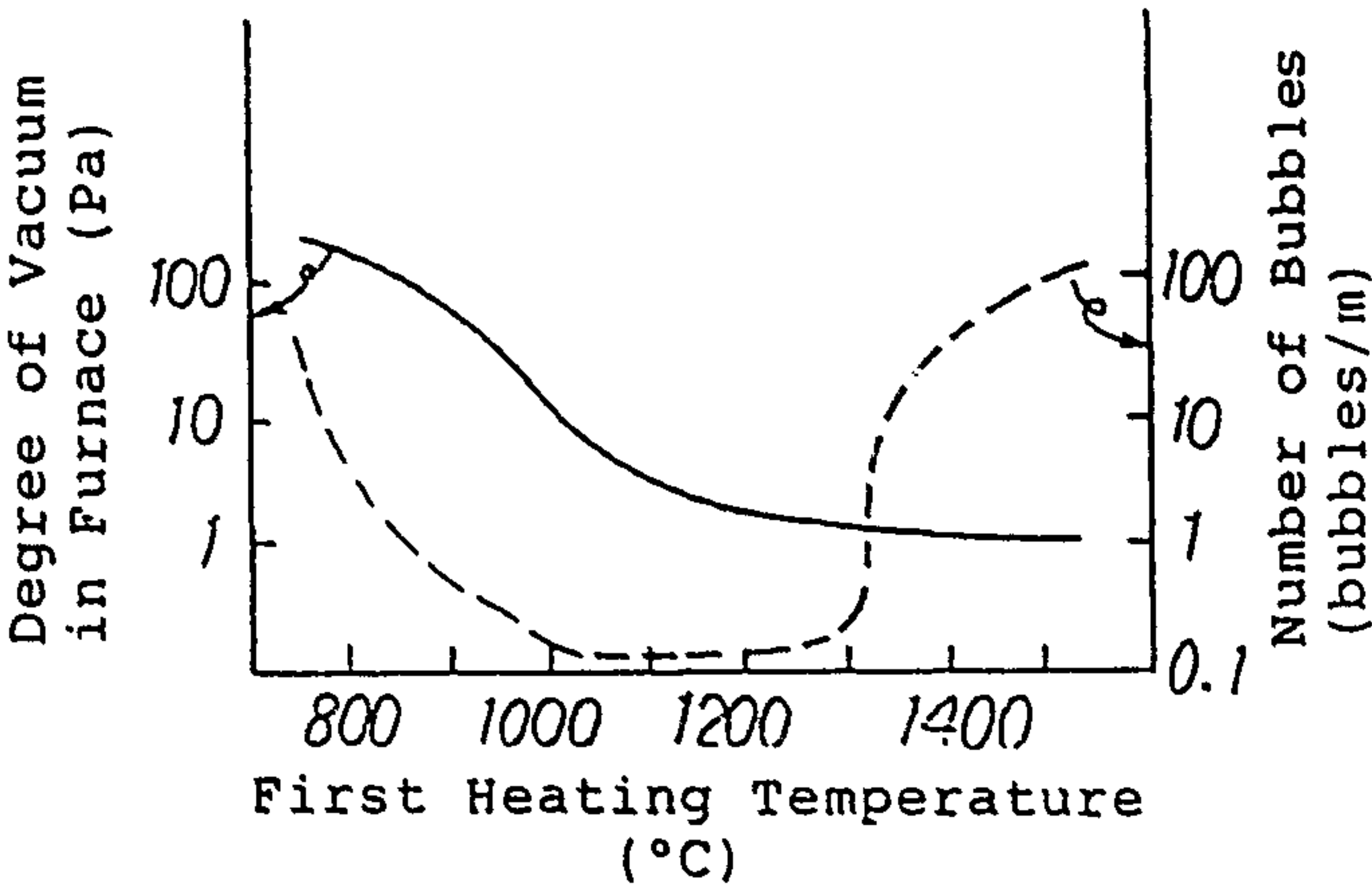


FIG. 6

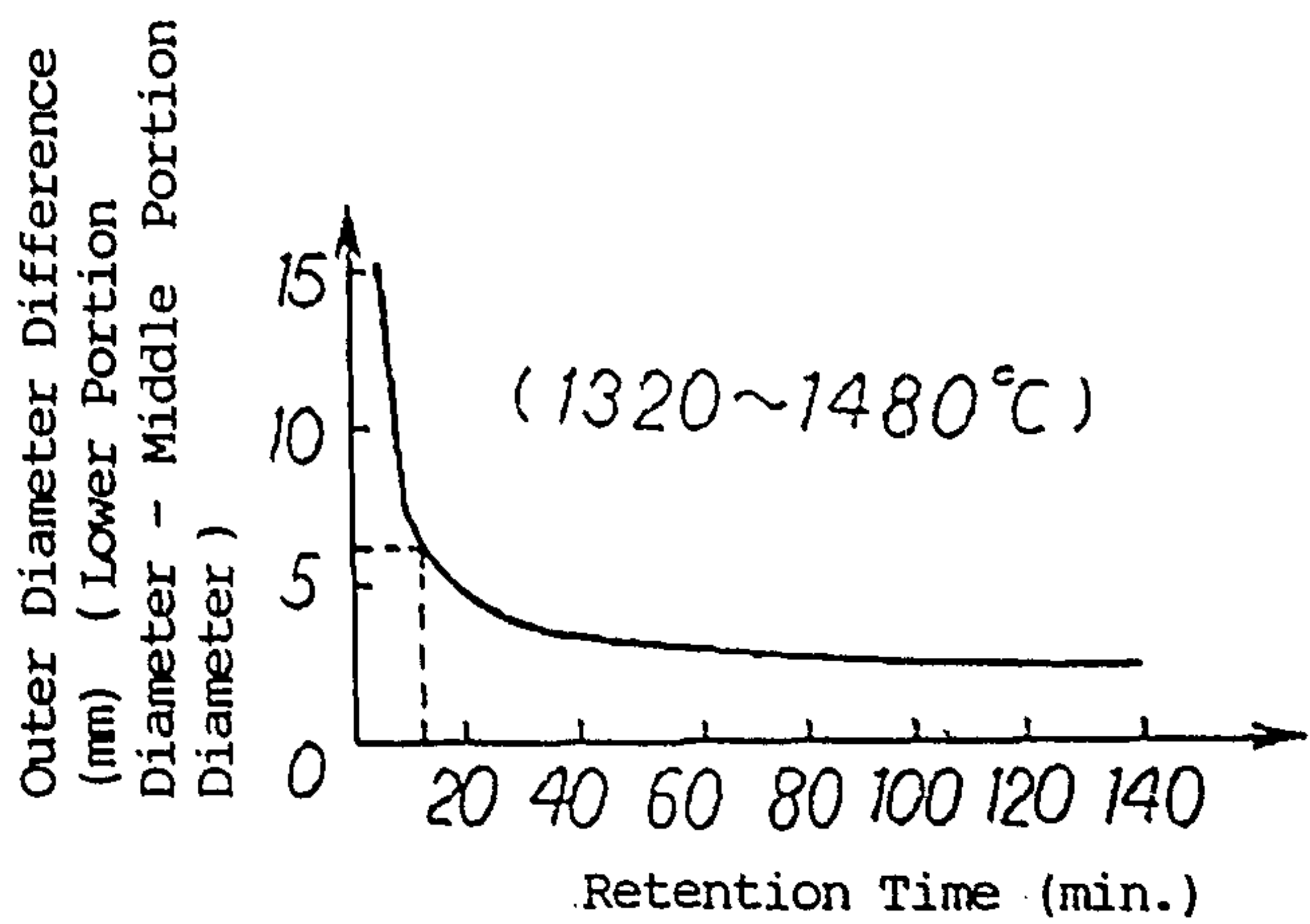


FIG. 7

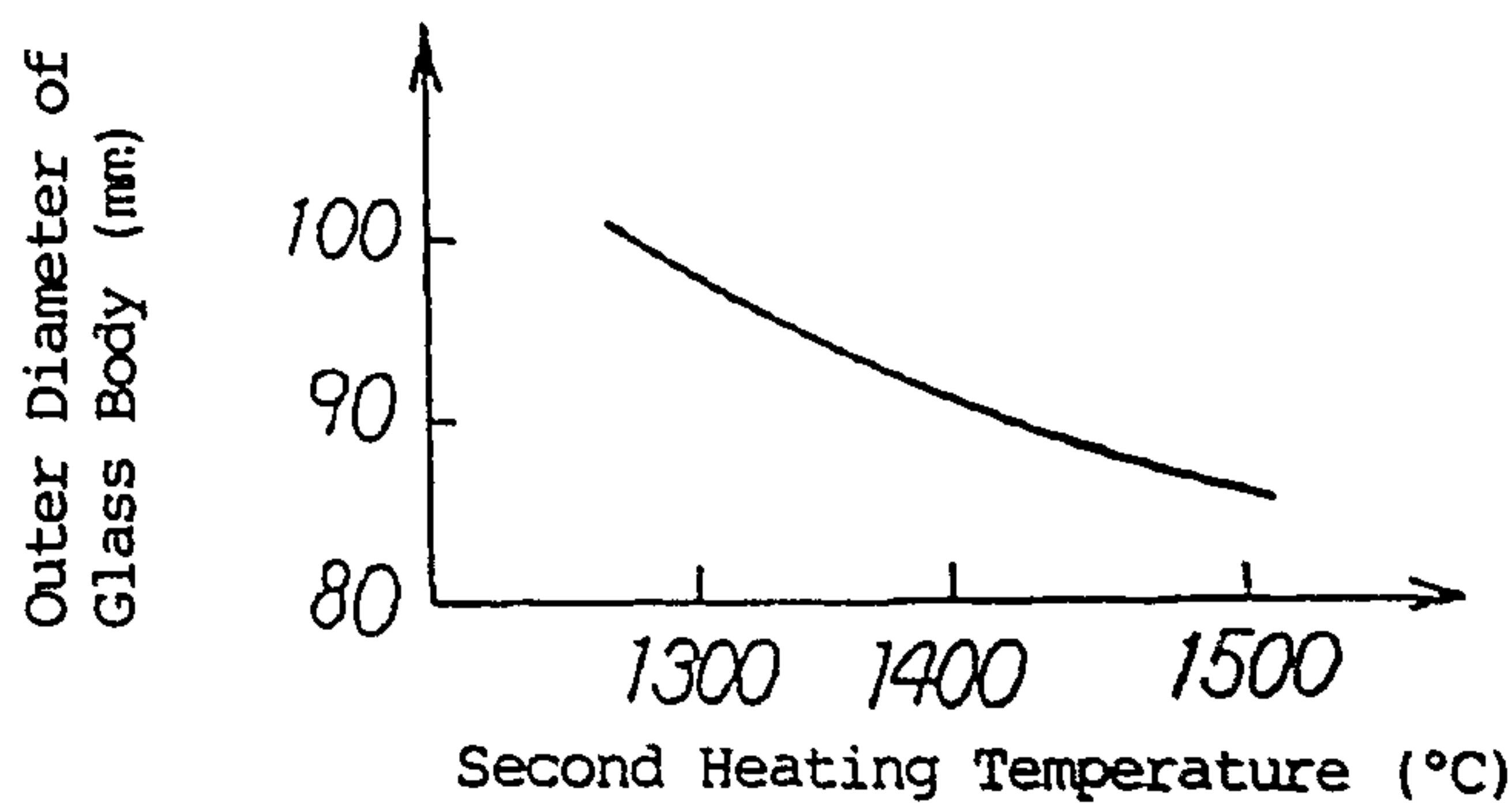


FIG. 8

