# PATENT SPECIFICATION

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## (54) PRODUCTION OF VACUUM BOTTLES OR LIKE CONTAINERS

(71) We, NIPPON SANSO K.K., a Japanese body corporate of 1-16-7, Nishi-shinbashi, Minato-ku, Tokyo, Japan, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for producing a vacuum bottle, flask or like container for example by a vacuum brazing operation.

Heretofore, a metal vacuum bottle has been produced in such a manner that after internal and external shells thereof have been seamed together adjacent their top openings, evacuation of the insulating space between the two shells is carried out through a vacuum suction port previously provided on the bottom part of the external shell. However, it is commonly difficult to maintain the vacuum intensity and thus the thermal insulation effect, for a long period of time because of outgassing from the surfaces of the shells defining the insulating space, or other parts. In order to maintain the desired thermal insulation effect, it is necessary to maintain a high degree of vacuum by repeating a proper evacuation process, but such repetition complicates the steps in producing the bottle as well as increasing cost.

To avoid the aforesaid shortcomings, there has been proposed a process for producing a metal vacuum bottle of the type mentioned above by a vacuum brazing operation, in which a substantially complete degassing treatment can be carried out by subjecting the surfaces of the internal and external shells to a heating treatment and evacuating the space between the two shells; additionally the steps in production are intended to be simplified. Such process comprises the steps of transferring the assembly of a metal vacuum bottle to a vacuum heating furnace for heating and evacuating gases from said assembly. The assembly is previously provided with a filler metal at the seam between the internal and the external shells, and the process includes the step of brazing said seam by heating to a temperature higher than the melting point of the filler metal under the desired intensity of vacuum, for instance, a pressure lower than $10^{-2}$ Torr. According to the above-mentioned process, the assembly is subjected to vacuum baking when the internal and the external shells of the vacuum bottle are brazed together, so that degassing of the shell surfaces is effected at the same time. Thus there can favourably be avoided deterioration in the vacuum thermal insulation effect resulting from outgassing gases as time elapses. Moreover, in a conventional process, there have generally been necessary two steps for producing a vacuum bottle, one of which is brazing the internal and the external shells together and the other of which is evacuation of space between said two shells. In the foregoing proposed process, the aforesaid steps can advantageously be carried out concurrently.

It has however been found that there are difficulties in putting the proposed process into practical use. For instance, it is essential to attain high intensity of vacuum because of the effect of this on thermal insulation; also it is desirable for the vacuum to be obtained as quickly as possible. However, when the vacuum brazing operation is used, because of the influence of flow rates there is a difference between the vacuum furnace and the thermal insulation space of the vacuum bottle so that the evacuation should be carried on for a long period of time in order to acquire the desired vacuum intensity. The troubles are caused by the fact that from the viewpoint of operation, it is not desirable to enlarge the crosssectional area of the vacuum suction port; if the vacuum suction port is enlarged to enhance the evacuation effect, the operation for sealing with a brazing filler metal becomes complicated and causes unreliable sealing. Furthermore, it happens that in many cases, the desired intensity of vacuum after subjecting the vacuum suction port to the vacuum brazing operation may not be maintained owing to residual gases within the thermal insulation space such gases having been generated at the time of the brazing filler metal being melted. Still further, in the vacuum brazing operation for producing a vacuum bottle, the whole body of the vacuum bottle has to be heated at a high temperature, so that, in the case of producing a vacuum bottle of austenitic stainless steel and the like, there may be an undesirable annealing effect. This may make it necessary for the external shell to be made thick, resulting in an increase in weight thereof as well as a considerable rise in the cost of production etc.

According to the invention, there is provided a process for producing a vacuum bottle, flask or like container, comprising inner and outer metallic shells defining an insulating space therebetween having an evacuation outlet sealable by means of filler metal, vacuum getter material being provided for the insulating space, wherein after evacuation the temperature of the shells is raised so as to melt the filler metal and seal the evacuation outlet whilst the vacuum is maintained, and the temperature is subsequently lowered to a level at which the getter material is active, and maintained at this level for a sufficient length of time for the getter substantially to assimilate residual gases in the insulating space.

Some embodiments of the invention will now be described by way of example and with reference to the accompanying drawings, in which:—

Fig. 1 is a longitudinal sectional view of a first form of vacuum bottle assembly;

Fig. 2 is a graph showing the comparison between a vacuum bottle produced in accordance with the present invention and one produced by a conventional process, regarding the temperature change with time of boiled water in each of said vacuum bottles; and

Fig. 3 is a longitudinal sectional view of a second form of bottle assembly.

In Fig. 1, the reference numeral 1 designates a metallic bottle assembly comprising an internal metallic shell 2 and an external metallic shell 3. 4 indicates thermal insulation space between said shells 2 and 3, to be evacuated. 5 denotes a seam formed between the internal and the external shells 2 and 3, provided with a filler metal such as a nickel alloy or another suitable material. 6 indicates further filler metal, identical with the one provided at the seam 5 mentioned above. 7 is a sealing plate made of a metal identical to that of the shells 2 and 3 for sealing an evacuation outlet in the form of a port 8, the sealing plate 7 being mounted on the filler metal 6, which comprises portions opposed to each other at a suitable distance.

Accordingly, the filler metal 6, which will serve to connect the sealing plate 7 to the inner wall of the external shell 3, in the meantime supports the plate 7 so as to provide an appropriate vent between the thermal insulation space 4 and the outside air.

Reference numeral 9 designates a thermal insulation material, for instance aluminum silicate in the form of cotton, and 10 designates a powdery metallic vacuum getter material such as for example titanium hydride or the like positioned within the thermal insulation material 9. The getter material is adapted to discharge gas at a high temperature and then at a certain constant, lower temperature to assimilate gases particularly hydrogen. The assimilation will be by means of absorption, adsorption chemical reaction and so forth. The vacuum bottle assembly as shown is put in a vacuum furnace which is subsequently evacuated. Together with exhaustion of gases within the vacuum furnace, other gases existing in the thermal insulation space of the vacuum bottle assembly 1 and gases ejected from the getter material are discharged into the vacuum furnace from the port 8 through the clearance formed between the filler metal 6 and the sealing plate 7, so that evacuation of the thermal insulation space 4 also takes place. During evacuation the intensity of vacuum within the furnace as well as within the thermal insulation spaces 4 increases and when the intensity of vacuum reaches a predetermined level, heating treatment starts. Said heating treatment is carried on until the internal temperature of the vacuum furnace and thus the shells 2 and 3, becomes uniform at a temperature somewhat lower than the melting point of the filler metal, and a predetermined intensity of vacuum, for instance a pressure of $10^{-2}$ Torr is obtained. In the course of the heating treatment the getter material such as titanium hydride or the like, is adapted to eject any previously assimilated gases, so that it becomes activated.

In the next step, the internal temperature of the vacuum furnace is raised higher than the temperature of the filler metal's liquidus melting point, to melt the filler metal provided at the seam 5 formed between the internal and the external shells 2 and 3 so as to join the two shells by brazing them together. At the same time, the filler metal 6, which has been supporting the sealing plate 7, is also melted so that the sealing plate 7 drops downwards onto the inner wall of the external shell 3 and is brazed by the molten filler metal to seal the port 8.

The assembly thus treated is cooled down to a temperature at which the getter material 10 is active and at its most effective so that substantially all gases within the thermal insulation space 4 as well as gases ejected from the molten filler metal, are assimilated by the getter material 10, the lowered temperature being maintained for a sufficient length of time. As a result the intensity of vacuum in the thermal insulation space 4 is increased and the assembly can then be cooled down to room temperature.

In trials, there has been produced a metal vacuum bottle made of BS 304S15 stainless steel as a trial product, employing a nickel alloy as a filler metal, and the getter material in the form of the powdery titanium hydride enveloped by the thermal insulation material of aluminum silicate in the form of cotton. When the intensity of vacuum within the vacuum furnace reached $10^{-2}$ Torr during vacuum heating, the temperature within the furnace was raised up to about 1050°C to melt the filler metal 6, whereby the vacuum suction port 8 was sealed. Subsequently, the temperature was lowered to about 600°C and the bottle kept at the temperature for approximately 10 minutes, and subsequently cooled to a room temperature.

Fig. 2 is a graph showing the comparison between a vacuum bottle manufactured on trial by a conventional process, having hitherto been proposed, and a vacuum bottle produced in accordance with the present invention, as regarding the change of temperature of boiled water with time elapsed. In this instance, the vacuum bottle made by a conventional process was subjected to an evacuation treatment until the intensity of vacuum within the vacuum furnace reached $10^{-4}$ Torr, whilst the vacuum bottle made by the process in accordance with the invention was sealed when the vacuum intensity reached $10^{-2}$ Torr. However, the thermal insulation property of the vacuum bottle made in accordance with the invention is shown to be much better than that of the vacuum bottle made by the conventional process.

Fig. 3 shows a modified embodiment of the present invention in which the evacuation outlet for the insulating space 4 is provided adjacent a seam 11 to be formed between the internal and the external shells 2 and 3 by means of filler metal 12. Supporting members 13 for the shell 2 are made of a filler metal which is the same as the filler metal 12, and are positioned between the internal and the external shells 2 and 3 at the bottom thereof so as to form a clearance at the seam 11 to be formed. Such a vacuum bottle assembly is treated within a vacuum furnace in a similar way as for the previous embodiment. However, evacuation of the thermal insulation space is effected through the clearance formed at the seam 11 to be formed. When the temperature is raised above the melting point of the filler metal 12, not only this melts, but also the supporting members 13, so that the internal shell 2 drops downwardly to be brazed to the external shell 3.

Subsequently, due to maintaining the furnace temperature at an activating temperature of the getter 10 for a certain period of time, a vacuum bottle having excellent thermal insulation properties similar to those of the previous embodiment, can be produced.

Reference numeral 14 indicates a shielding plate which is previously joined by spot welding to the external shell 3, and 15 designates a supporter made of fine metal wire connected at one end to the outer wall of the internal shell 2. The other free end of the supporter is in contact with the inner wall of the external shell 3, as well as with the supporting member 13 made of filler metal. Thus the free end of the supporter 15 is adapted to be securely adhered to the inner wall of the external shell 3 due to melting of said supporting member 13, so as to hold the internal shell 2 when the external shells 2 and 3 are securely connected with each other.

A metallic vacuum bottle may generally be produced from a base metal such as aluminium or the like having excellent workability, but as such metals have a high heat conductivity, stainless steel having a low heat conductivity is commonly used as the metal. Accordingly, if manufacturing a vacuum bottle by a vacuum brazing process as set forth hereinbefore, it may be necessary to make the external shell with large thickness since annealing occurs during the vacuum brazing, and reduces the strength of the stainless steel.

However, the process for producing a vacuum bottle in accordance with the present invention can be adapted to avoid the drawbacks due to the annealing effect by carrying out the vacuum heating treatment after spraying or coating the getter material over one or both of the walls of the internal and the external shells defining the insulation space 4. In other words, the vacuum heating treatment is carried out after powdery eg. metallic getter has been sprayed or coated on to such walls, or other desired portions. The getter may diffuse into the surfaces of the shells to harden the material thereof, eg. by forming intermetallic compounds. Accordingly, the thermal insulation property of the shell metal can be improved by the effect of the getter, and the strength of the vacuum bottle can be increased.

Regarding the getter material which can be employed, such metals as titanium, zirconium or the like could be used, or alloys, or hydrides, of such metals or mixtures thereof. Other possible metals might be Li, Na, K, Mg, Ca, Ba, Al, Ge, Cu, Y, V, Nb, Ta, Cr, Mn, Fe, Co, Ni, Pd, Uranium and other metals of the Actinide and Lanthanide series, their alloys and hydrides. These possibilities are not exhaustive and any other material could be used which is capable of effectively assimilating hydrogen or other gases in the course of the process steps necessary. Such getters should therefore be able to assimilate hydrogen or another gas at a temperature not higher than the melting point of the filler metal as well as under a desired vacuum degree.

Generally it is anticipated that the filler will melt at a temperature greater than 750°C, and the getter material should be active e.g. to assimilate hydrogen, below this temperature and at the vacuum intensity concerned eg. $10^{-2}$ Torr.

In the above described embodiments, the vacuum bottle assemblies had evacuation outlets for the thermal insulation spaces formed either on the bottom part of the external shell or adjacent the seam to be formed between the internal and the external shells. However, any other optional structures, eg. those which are provided with clearance formed on the bottom part of the internal shell or adjacent the top opening of the internal and the external shells, for effecting evacuation may be used.

As will clearly be understood from the foregoing description, at least with the preferred embodiments there is the advantage of simplifying the working steps in a process for producing eg. a metallic vacuum bottle, by joining the internal and the external shells simultaneously with the evacuation of the thermal insulation space in a vacuum brazing operation. Furthermore there is an improved effect, different from those of conventional processes, due to the use of the getter in the step of vacuum brazing treatment. For example, deterioration in the intensity of vacuum of the thermal insulation space caused by gases generated in the step of brazing can be prevented, whereby a vacuum bottle having an extremely good thermal insulation performance can be obtained. Moreover, since the intensity of vacuum in the thermal insulation space during evacuation whilst heating in the vacuum furnace, does not need to be raised above that of a conventional known vacuum bottle the time necessary for evacuation can be shortened and thus the cost of production can be lowered. Still further, as the working steps for the evacuation outlet can be simplified, defects in the end product can be minimized. In addition, the foregoing effects as well as the strength of the metal of the vacuum bottle can be improved due to spraying or coating the getter material over the wall surfaces of the thermal insulation space as occasion demands, so that it is possible to keep low the weight of the bottle.

WHAT WE CLAIM IS:—

1. A process for producing a vacuum bottle, flask or like container, comprising inner and outer metallic shells defining an insulating space therebetween having an evacuation outlet sealable by means of filler metal, vacuum getter material being provided for the insulating space, wherein after evacuation the temperature of the shells is raised so as to melt the filler metal and seal the evacuation outlet whilst the vacuum is maintained, and the temperature is subsequently lowered to a level at which the getter material is active, and maintained at this level for a sufficient length of time for the getter substantially to assimilate residual gases in the insulating space.

2. A process as claimed in claim 1, wherein the inner and outer shells are joined together by filler metal melting at said raised temperature.

3. A process as claimed in claim 2, wherein the evacuation outlet is defined between portions of the respective shells adjacent the join to be made between them.

4. A process as claimed in claim 1, 2 or 3 wherein the getter material is sprayed or coated on one or both of the shell walls defining the insulating space.

5. A process as claimed in any preceding claim wherein heating and evacuation are carried out in a vacuum furnace.

6. A process as claimed in any preceding claim wherein the temperature at which the filler metal melts is greater then 750°C.

7. A process as claimed in claim 6 wherein the getter material is such that it assimilates hydrogen at a temperature less than 750°C and a pressure less than $10^{-2}$ Torr.

8. A process as claimed in any preceding claim wherein evacuation is carried out, at least in part, at a temperature maintained at a level below the melting point of the filler metal and at which the getter material discharges previously assimilated gases.

9. A process as claimed in claim 4 wherein the shell material is hardened by diffusion of the getter material into the surface thereof.

10. A process for producing a vacuum bottle, flask or like container, substantially as hereinbefore described.

11. A process for producing a vacuum bottle, flask or like container substantially

as hereinbefore described with reference to Fig 1 or Fig 3 of the accompanying drawings.

12. A vacuum bottle, flask or like container, made by a process as claimed in any preceding claim.

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1576670 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

Fig. 1

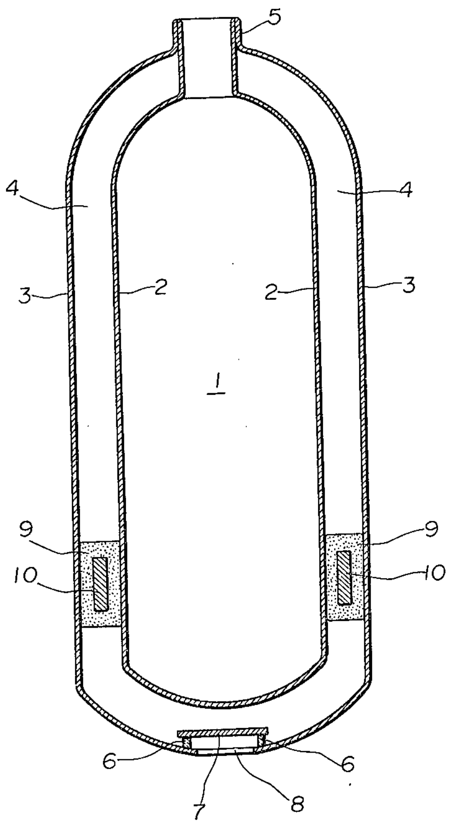
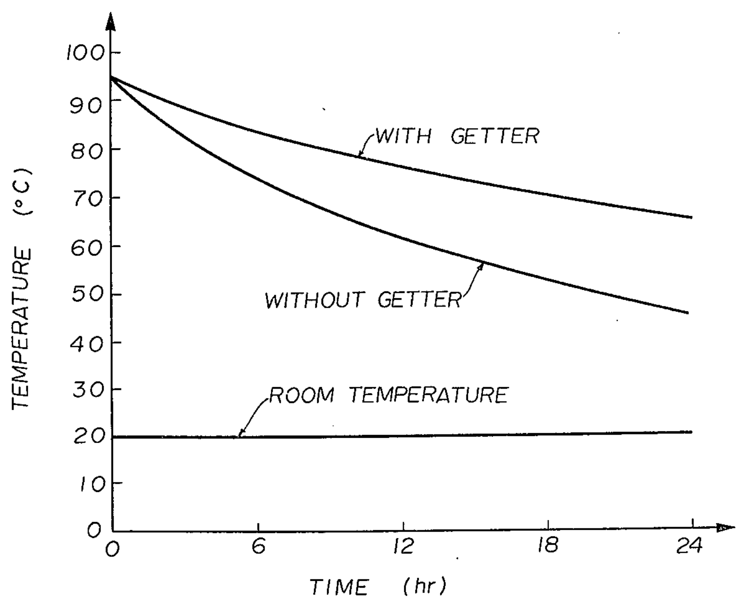


*Fig. 2*


100
90
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TEMPERATURE (°C)
WITH GETTER
WITHOUT GETTER
ROOM TEMPERATURE
0
6
12
18
24
TIME (hr)

1576670 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 3

Fig. 3