

[54] **METHOD OF TREATING ARTICLES
UNDER DIFFERENTIAL VACUUM
CONDITIONS WITH EXTERNAL GAS
FLOW**

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126/343.5 R; 51/317, 319

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[57] **ABSTRACT**

The process of treating articles in a vacuum which includes placing the articles in a first enclosure which is fluidly connected to a second enclosure through a fixed or variable orifice. The first enclosure may be physically located within the confines of the second enclosure or outside the confines thereof. A vacuum source is connected to the second enclosure and draws a vacuum therein to a range of from 10 microns to 6,700 microns and an external gas is continuously introduced into the first enclosure which results in a vacuum therein of from 100 microns to 10,000 microns. The gas may be inert or it may be functional i.e., reactive. It is desirable to keep the ratio of pressure in the first enclosure to pressure in the second enclosure in the range of from about 1.5:1 to about 20:1.

6 Claims, 3 Drawing Figures

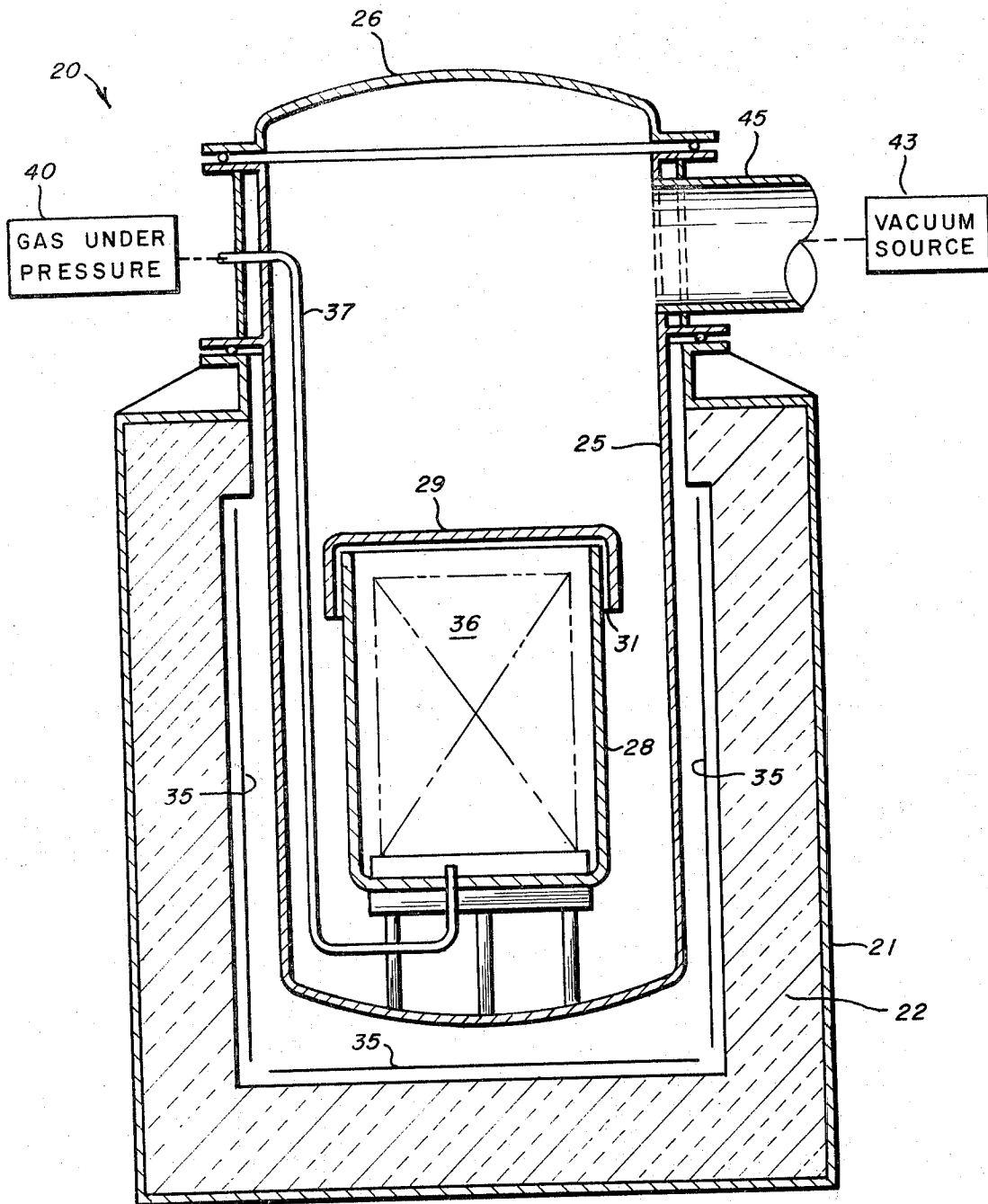
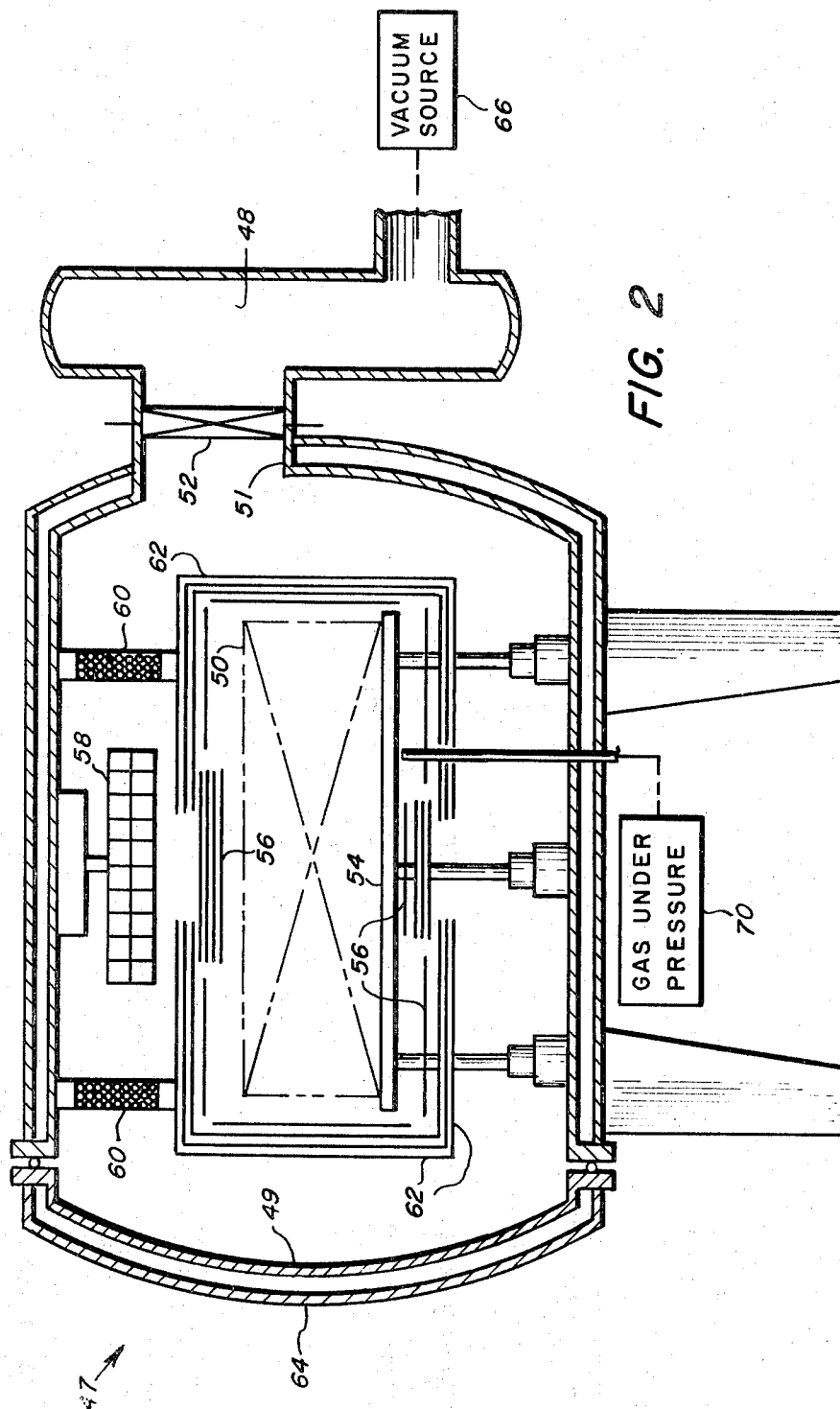


FIG. 1



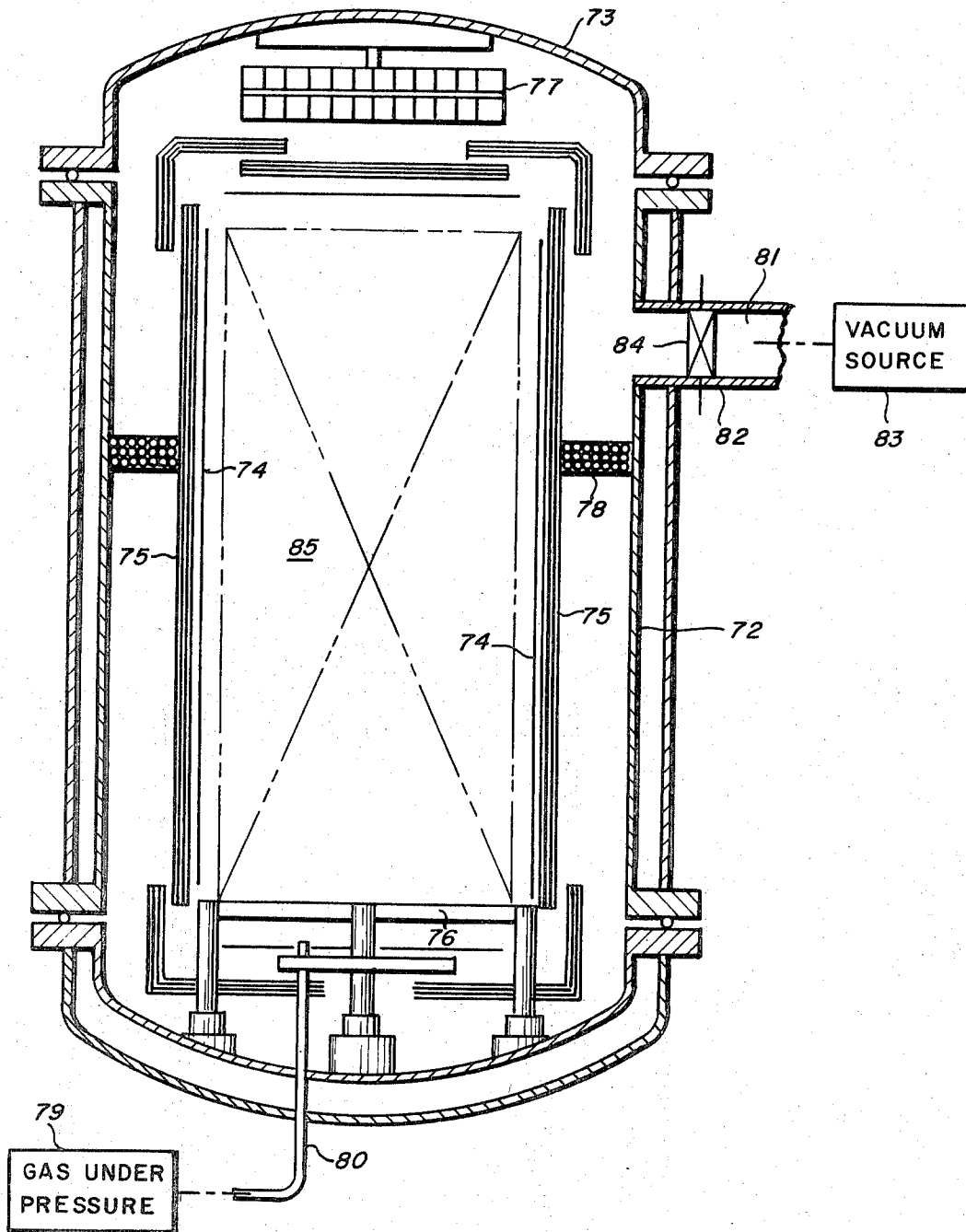


FIG. 3

METHOD OF TREATING ARTICLES UNDER DIFFERENTIAL VACUUM CONDITIONS WITH EXTERNAL GAS FLOW

METHOD OF TREATING ARTICLES IN A VACUUM

Vacuum furnaces and other vacuum surface cleaning devices have been built for many years. Their pattern has remained virtually unchanged throughout the last 20 years. New ideas and improvements have devoted themselves to methods of creating vacua faster or to a lower degree of vacuum (lower ultimate or operating pressure), or they have been devoted to improvements in chamber design or heat source design to obtain better overall conditions of cleanliness or heat distribution within the operating shell.

For many years gas flow in sub-atmosphere chambers has been divided into laminar (viscous) or higher pressure flow, turbulent or intermediate flow, and molecular or low pressure (roughly below 0.001 torr) flow. One torr is equal to a pressure of 1 mm. of mercury or is equal to 1,000 microns of mercury. It has always been the goal of equipment manufacturers and process designers to achieve the lowest possible levels of vacuum because this represented a desirable increase in cleanliness. Hence, most vacuum processes (and in particular vacuum processes involving heat) have been carried on in the sub-micron (i.e., below 0.001 torr) region. Each decade of absolute pressure reduction means one-tenth the contamination level and, hence, a better product will result.

Our invention is based on a common phenomenon whose actual mechanism has heretofore not been fully understood. Hence, the mechanism needed to utilize this phenomenon has not only not been developed, but conditions under which this phenomenon would occur have been studiously avoided.

When a sub-micron level of vacuum has been achieved, the mean free path of the remaining gas molecules has lengthened to the point where there is no actual "flow" pattern or viscous "flow" effect. There is a net flow of molecules from an area of higher pressure to one of lower pressure, but they migrate of their own directional volition by elastic bounce. As density decreases, equilibria form more quickly. Unwanted molecules having a solid or liquid surface and entering the gas phase are more free to return and land on the surface from which they left (or an adjacent one). Molecular motion and direction in the pressure ranges of molecular flow are more or less uncontrollable.

In the process of heat treating and surface cleaning, a vacuum is often used because it places the part in an area of cleanliness (within the vacuum chamber) where offending molecules are supposedly at a minimum. The general desire is to remove from the surface of the part molecules which will have an undesirable interaction with the part surface (or, also, to add ones which will have a desirable effect on the surface). The usual way to achieve this has been to achieve lower and lower vacua. This, unfortunately, leads not only to more complex devices, but it also runs counter to phenomena governing equilibria formation. The real desire to remove the unwanted molecules from the surface to be treated while not allowing them to return. Thus, no equilibrium of unwanted molecules must be allowed to proceed. Only equilibria of molecules desired at the surface of the part must be allowed. Unfortunately,

under molecular flow conditions this is virtually impossible to control.

This invention makes use of the realization of the practical use involved in understanding the meaning of gas flow as it relates to surface interactions. To control the formation of equilibria it is necessary to control the direction of molecules within the gas flow pattern under vacuum conditions. This can only be done by operating the device in a controlled manner at a vacuum level high enough in absolute pressure to be within a narrow band of laminar flow conditions while maintaining the lowest total pressure possible. With specific geometrical relationships the partial pressure(s) of an undesirable gas (or gasses) may be regulated, as may be the partial pressure(s) of desirable gases. Thus, may be controlled the surface interactions with a piece of equipment much more simple and much more precisely controlled than current types of vacuum furnaces or vacuum cleaning devices.

Other objects and a fuller understanding of this invention may be had by referring to the following description and claims, taken in conjunction with the accompanying drawings, in which:

FIG. 1 is an elevational view in section of one form of vacuum furnace which can be utilized to practice the process of the present invention;

FIG. 2 is an elevational view in section of another form of vacuum furnace which can be utilized to practice the process of the present invention;

FIG. 3 is an elevational view in section of still another form of vacuum furnace which can be used to practice the process of the present invention.

Referring to FIG. 1 there is shown a vacuum furnace 20 which may be referred to as an external hot wall design and which comprises an outer wall 21 having insulation 22. A shell 25 forms an outer vacuum chamber (at times herein referred to as a second enclosure) which is provided with a sealable cover 26 to provide for the entrance and exit of articles of manufacture to be treated by the process of the present invention.

The shell 25 is suspended as shown and is adapted to support a work chamber (at times herein referred to as a first enclosure) 28 which has a cover 29. The cover 29 is so dimensioned or fitted to the work chamber as to provide a fixed orifice 31 or gas conductance path between the interior of work chamber 28 and the interior of the shell 25.

The furnace 20 is provided with electrical heating elements 35 for heating articles 36 in the work chamber 28 from ambient to on the order of 3,000° C. A first conduit 37 provides a fluid path from an external source of gas 40 to the interior of the work chamber for a purpose which will be explained in more detail hereinafter.

A vacuum pump 43 is provided and is fluidly connected to the shell 25 (second enclosure) by means of another conduit 45. It will, therefore, be clear that when the vacuum source 43 is activated, the shell 25 is evacuated and, also, the work chamber 28 by way of the orifice 31 formed between the cover 29 and the work chamber 28. In accordance with the teachings of the present invention, the process of treating various articles of manufacture is accomplished by evacuating the first and second enclosures to a pressure of on the order 10 microns of Hg. to 10,000 microns of Hg. The external gas source 40 is utilized to continuously inject gas into the first enclosure to always maintain this en-

closure at a slightly higher pressure than the second enclosure 25. As mentioned in the introduction, the present invention operates at pressures above which molecular flow of gases occurs which is generally above 1 micron; and, since the pressure in the first enclosure must be higher than in the second enclosure, the preferred range of pressure in the first enclosure is from about 100 microns to 10,000 microns and the pressure in the second enclosure is from about 10 microns on the low side of the range up to about 6,700 microns on the high side of the range. The orifice 31 is, also, sized such that when taken with the gas source 40 and the pressure in the first and second enclosures, it will pass from about 0.1 cubic feet per hour (CFH) to about 10 CFH. Specific examples showing the treatment of various articles of manufacture in an apparatus similar to that shown in FIG. 1 will be given. However, the apparatus of FIGS. 2 and 3 will first be described.

The furnace 47 of FIG. 2 differs to some degree from that of FIG. 1 and particularly in that its second enclosure 48 is separate from or outside of the first enclosure 49 and is fluidly connected thereto by means of a conduit 51 as distinguished from FIG. 1 wherein the enclosure 28 is totally within the confines of the second enclosure 25. A variable orifice 52 is positioned between enclosures 48 and 49 and in the conduit 51. Other features of the furnace of FIGS. 2 and 3 include a work support 54, heating elements 56, a circulating fan 58, cooling elements 60 and heat shields 62. A cover 64 is provided so as to gain entrance to enclosure 49 so that the work 50 can be processed according to this invention. A vacuum source 66 is connected to chamber or enclosure 48 and an external gas source 70 is fluidly connected to the first enclosure 49.

The following are examples showing the treatment of various articles of manufacture according to the process of the present invention.

EXAMPLE 1

Heat Treating and Surface Preparation of Cast iron and/or Ductile Iron and/or Steel Castings

The purpose of this process was to prepare the surface of these materials which materials are to be subsequently coated with glass, ceramic or other materials. Adhesion of the additive coatings requires both clean, metallic surfaces and out-gassing of the casting itself, so that entrapped gas does not later cause blistering or breaking of the coating.

Surface oxides of the metal must be removed from the part during the outgassing process.

Concomitant with the outgassing and surface chemical treatment of the part may be the heat treatment of the part changing it to perlitic iron or malleable iron.

Two different furnace configurations were used in various runs of this material. The process parameters were the same for both physical configurations. Configuration one consisted of the following:

1. An external vacuum chamber constituting the retort of a standard hot wall vacuum furnace. A removable sealable cover for entry and exit of parts. Vacuum gauges in both the work chamber and the exhaust chamber appropriate to the process. Gas leads into the bottom of the inner work chamber, and appropriate electrical and mechanical controls. A cover to isolate the inner chamber and the other chamber with appropriate conductance to

meet process requirements. The work parts and the inner chamber were suspended from this cover. Dimensions of the exhaust chamber were 30 inches $\times$ 72 inches long.

2. An inner or work chamber was located inside the outer chamber — dimensions 24 inches, ID $\times$ 48 inches long.
3. A cover was placed on top of the inner chamber with gas flow clearance of 59 CFM along the perimeter between the cover and the side walls of the inner chamber.
4. A vacuum pump of 500 CFM capacity was attached to the exhaust chamber by 15 feet of 6-inch ID pipe. A restricting butterfly-type valve was installed in this line to regulate the flow into the vacuum pump.
5. A one-fourth inch gas inlet line was connected to the diffusion manifold in the bottom of the work chamber.
6. Pressure in the gas source was 10 PSIG. Nitrogen, argon and air were used as the purge gases in various cycles. No difference in result was noted.

Configuration two consisted of the following:

1. A vacuum chamber circa 30 inches ID $\times$ 72 inches high functioning as the work chamber. This, also, functioned as the retort of a standard hot wall furnace.
2. This chamber had at right angles to it a 10 inches ID $\times$ 24 inches long exhaust chamber isolated from the work chamber by a butterfly-type valve to act as a variable restriction in the gas flow from the work chamber into the exhaust chamber. Flow rates from the work chamber to the exhaust chamber were set at 30 CFM.
3. The exhaust chamber was connected to a 500 CFM vacuum pump via 15 feet of 6 inches ID pipe.
4. A gas inlet pipe was connected to a diffusion manifold in the bottom of the work chamber and to a source of nitrogen, argon, and air at 10 PSIG. Suitable metering valves and flow meters were in this line to regulate the incoming gas flow — both as to quantity and type of gas.

NOTE:

In runs of both physical configurations no significant difference was found in the parts. Gas flow parameters were approximately — but not exactly — the same for both configurations.

The process steps were:

1. Two hundred pounds of parts were stacked at random within the work chamber. They were intermixed — cast iron and cast steel. Their condition was dirty and oxidized at random. No prior cleaning processes were used.
2. The closures on both the outer exhaust chamber and the inner work chamber were replaced.
3. The two chambers were evacuated to circa 50 microns of Hg.
4. Gas flow was continuously introduced at 1–2 CFH and pressures were stabilized at 250 microns within the work chamber and 40–60 microns in the outer chamber. In other runs pressures in the work chamber were stabilized at 400 microns and 80 microns in the outer chamber no significant differences in the parts were noted until pressures reached 800 microns within the work chamber.

5. Flow was allowed to proceed for approximately 10 minutes before temperature was raised above ambient.
6. Heating cycle began. Gas flow was reduced from 2 CFH to about 0.1 CFH as heating cycle progressed through 900° F. Severe outgassing was evidenced by total and partial pressure rises. Pressure was maintained between 300–500 microns within the work area and 50–90 microns in the exhaust area.
7. Temperature was raised to 1,800° F, and the parts were maintained at this maximum temperature for 1 hour.
8. As outgassing decreased, gas flow was increased to maintain a stable pressure of 400 microns within the work area and 50 microns within the exhaust area.
9. After one hour soak at 1,800° F, temperature was lowered and heaters turned off. Both chambers were backfilled with nitrogen or argon to a pressure of 5 inches of vacuum. Internal gas was circulated to hasten cooling.
10. It should be noted that on subsequent runs pressures in the work chamber were run at 1 torr and 2 torr with pressures in the exhaust chamber as high as 500 microns. Parts done at these pressures were satisfactory.
11. Parts done under conditions of 10 torr within the work chamber and 2 torr in the exhaust chamber were not satisfactory.

EXAMPLE 2

Fluxless Brazing of Aluminum

The purpose of this process was to braze complex aluminum heat exchangers in a vacuum environment. The parts were constructed of an alloy of silicon-magnesium clad aluminum.

Several series of runs were made in the usual fluxless brazing sequence, i.e.:

1. Parts were put into their supporting jigs.
2. Jigs and parts were placed in a normal vacuum process furnace.
3. The vacuum was reduced to an absolute pressure of 5×10^{-5} torr.
4. Temperature was increased from ambient to 1,100° F and maintained at the 1,100° F upper limit for 5 minutes.
5. Temperature was then reduced to ambient and the parts removed.

The yield under the normal process above has varied from 50 percent good parts to 100 percent good parts. This is normal for production lots under this process. The process as done in a normal vacuum furnace, however, is very dependent on precise control of many factors, e.g.

1. Vacuum levels may not rise above 1×10^{-4} torr.
2. Even slight increases in the partial pressure of water, hydrogen, methane, and certain other gases within the vacuum chamber are critical and can alter the level of acceptable output of parts radically.
3. Presence of these particular gases and increases in their level is especially critical at certain temperature levels.
4. The relationship of temperature to vacuum level is particularly critical.

5. Temperature levels and uniformity must be precisely maintained.

This invention reduces or removes entirely these critical parameters and their related interdependence. Several series of runs were made in the furnace and by the process as described herein. The furnace had the following salient and unusual characteristics in addition to the parts usually found in all vacuum furnaces:

1. Standard hot wall design including a vacuum retort or outer exhaust vacuum chamber envelope.
2. This outer envelope was connected to a 500 CFM Beach Russ mechanical pump — Model 250—MO-500 (as the vacuum source) through 15 feet of 6 inches i.d. pipe.
3. The dimensions of this outer exhaust chamber were circa 30 inches i.d. $\times$ 72 inches long.
4. An inner work chamber, circa 24 inches i.d. $\times$ 48 inches long, with a lid whose clearance was precisely calculated to allow a conductance from inner chamber to outer chamber of 45 CFM.
5. Connected to the inner chamber was a gas inlet line flowing nitrogen at 5 psi absolute and fed through a metering valve so as to regulate flow at the vacuum inlet orifice to 1/60 CFM or a net inlet flow of 1 CFM. This incoming orifice was fed into a gas distribution network at the bottom of the work chamber (below the area in which the parts for treatment were suspended) so that there was roughly an even distribution of inlet gas over the bottom area of the work chamber.
6. Gas flow was laminar in an upward path past the piece parts and out the circumference of the removable top of the work chamber into the outer or exhaust chamber.
7. Pressure in the inner work area was 300–500 microns of mercury.
8. Pressure in the exhaust area was 35–50 microns of mercury.

The process followed in brazing the heat exchangers is as follows:

1. Retort top and work chamber top were removed at ambient pressure while heat exchangers were put into the furnace.
2. Both tops were replaced and furnace was opened to vacuum pump. Both chambers were reduced to about 50 microns pressure.
3. Gas flow was introduced into the work chamber and balanced to obtain 300 microns in the work area and 30 microns in the exhaust area. Cold purge was continued for 15 minutes to remove surface water vapor. This was indicated by an Aero Vac Mass Spectrometer Model AVA -2.
4. Heat level in furnace was increased from ambient to 850° F as quickly as possible.
5. After 5 minutes of soak at 850° F, temperature was elevated to 1,085° F and maintained for several minutes to braze.
6. Temperature was reduced below 1,000° F under flow conditions as described above.
7. Pressure within exhaust and work chambers was increased to a point commensurate with the heat dissipation characteristics of the furnace to hasten cooling — circa 5 inches of vacuum.
8. After sufficient cooling of parts furnace was opened and parts removed.

Several similar runs were made using argon as the purge gas, but no significant differences were noted.

Parts done in this mode of furnace operation consistently exhibited 90–100 percent good brazing. There was no pressure-temperature relationship of such critical dimensions as was noted under the vacuum conditions of the 10^{-5} torr process.

Runs were made with pressures of 1,000 microns in the work area and 200 microns in the exhaust area.

Runs were made at pressures of 2,000 torr in the work area and 500 microns in the work area. Parts began to become unacceptable in this area, because the offending gases to the process began to exhibit too high partial pressure.

EXAMPLE 3

Brazing of OFHC Copper

The purpose of this particular application of the invention was to braze small copper tubulations into a machined copper block of much greater mass than the small appendages. In addition, gaseous hydrogen, water, and oxygen were to be moved from the parts.

Several runs were made under typical brazing conditions in a standard vacuum furnace. Removal of gaseous hydrogen and oxygen from the copper was acceptable at the 10^{-5} torr level of total vacuum provided long periods of time were spent at these levels. However, brazing did not proceed well, and the parts were not satisfactory due to poor wetting. A two-step process has been adopted where the parts are outgassed and cleaned at a vacuum of 10^{-5} torr. The parts are then removed, and actual brazing is then accomplished in a hydrogen furnace. Brazes on the parts wetted well, and the parts were satisfactory. However, the two-step process requires the use of two furnaces. This is standard practice for several important brazing processes.

Several runs were made of this invention unit consisting of the following hardware configurations:

1. A hot wall furnace with external heater design was used.
2. An external vacuum chamber was used as a retort which functioned as the exhaust chamber. This was connected to a mechanical vacuum pump of 450 CFM through 15 feet of 6-inch pipe and an isolation or throttling valve. The volume of this exhaust chamber was in a 2-1 relationship with the internal work chamber.
3. The inner work chamber had a removable lid which did not fit tightly, but which had clearances designed to allow a conductance of 45 CFM between the inner and outer chamber at pressures of 100–1,000 microns.
4. The inner chamber was connected to a gas source which consisted of selectable entries for nitrogen, hydrogen, and argon. Quantities of each gas used varied from 0.5 CFH during the cycle depending on material outgassing at any given temperature setting.
5. A diffusion ring was used as the actual gas entry into the bottom of the work chamber to insure even circulation of the purge or reactive gases throughout the part area of the furnace.

The process sequence as carried out was:

1. The copper parts to be cleaned and brazed were suspended within the inner work chamber above the gas inlets and below the lid. This was done with the furnace open to ambient atmosphere.
2. The retort chamber lid and the work lid were replaced.

3. Both chambers were exhausted to 30 microns of mercury by a Beach-Russ pump connected to the outer retort chamber (or exhaust area).
4. Nitrogen gas flow at the rate of 1 CFH was introduced into the work chamber.
5. Pressures were balanced at 300 microns in the work chamber and 40 microns in the exhaust chamber.
6. Temperature was raised from ambient to 500° F.
7. Pressure in the work chamber rose to 400 microns.
8. Nitrogen purge was stopped.
9. Hydrogen purge started. Flow — 2 CFH. Pressure in work chamber — 500 microns. Pressure in exhaust chamber — 50 microns.
10. Hydrogen purge continued until braze temperature was reached.
11. After brazing was accomplished, the temperature was reduced to 500° F.
12. Hydrogen purge stopped.
13. Nitrogen purge was begun. Flow set at 1 CFH. Pressure in work chamber at 350 microns. Pressure in exhaust chamber at 40 microns.
14. Step 13 continued until partial pressure of hydrogen was reduced to below 5×10^{-9} torr on Aero Vac AVA 2 Analyzer. This was approximately 20 minutes.
15. Furnace was reduced to under 400° F temperature and then the pressure was raised to atmosphere and parts removed. The parts checked showed the brazing to be extremely successful.

FIG. 3 represents another physical arrangement within which the process of the present invention may be carried out. Shown in this Figure is a shell 72 having a cover 73. This shell constitutes or forms the work chamber which includes heating elements 74, heat shields 75, a work support 76, fan 77 and a heat exchanger 78. A source of gas under pressure 79 supplies gas to the work chamber 72 through a conduit 80. An exhaust chamber 81 is formed in a conduit 82 which leads from the work chamber to a vacuum pump 83. A variable orifice in the form of a butterfly valve 84 is located in conduit 82 and the exhaust chamber 81 is located between this valve 84 and the entrance port of the vacuum pump. It will be appreciated that the variable orifice may, also, be a fixed orifice. The work in this FIG. 3 is indicated by the numeral 85.

In this particular physical embodiment the same process conditions are utilized, namely, a pressure of 10 to 6,700 microns in the exhaust chamber and 100 to 10,000 microns in the work chamber. Also, the size and other relationships between the work and exhaust chambers, the size of the orifice between them; the introduction of gas from the external source; and the throughput of the vacuum source are such as to provide the pressure ranges recited above in the work and exhaust chambers.

The following are two examples of the process of the present invention carried out in an apparatus of the type shown in FIG. 3.

EXAMPLE 4

Titanium Brazing

The purpose of this process is to braze metallic parts which have high titanium and aluminum content. Surface cleanliness is imperative and is more than usually

critical because of the high temperatures involved. Many of the braze alloy materials contain high silver content. The usual specifications require a vacuum of 10^{-5} torr for this work.

The equipment for these runs consisted of:

1. A vacuum envelope or chamber 36 inches ID $\times$ 84 inches long with a removal cover. This functioned as the work chamber.
2. A side arm or pipe exit to a mechanical vacuum pump of 500 CFM. This was a cylindrical pipe 30 inches long and 10 inches ID. This was connected to the vacuum pump by a 12 feet long flexible pipe 6 inches ID. This functioned as the lower pressure or exhaust chamber.
3. An external gas flow connection fed from a 50 PSI source of argon (nitrogen used interchangeably on some runs) was distributed inside the work chamber by a diffusion device or ring.
4. A butterfly valve in the 6 inch line connecting the vacuum pump and the cylindrical outer chamber to regulate the gas flow to the vacuum pump. This regulation was, also, accomplished in a fixed manner in other runs by the size of the hole or orifice between the main work chamber and the appurtenant arm into the vacuum pump inlet line.
5. Two discrete pressure areas — the higher pressure work area and the lower pressure exhaust area — were maintained as usual. In this form they were appurtenant to each other rather than one within the other.

Process Steps:

1. Parts were placed within the work chamber and the lid closed.
2. The work chamber was exhausted by the mechanical vacuum pump through the exhaust chamber and the 10 inch orifice between the two vacuum areas to a pressure of 50 microns.
3. Gas flow was introduced into the work chamber in the form of argon or nitrogen from a 50 PSI source and at the rate of 8 CFH.
4. The mechanical vacuum pump was pumping at the rate of 450 CFM at 50 microns.
5. Pressures were balanced at ambient temperature with 300 microns in the work chamber and 50 microns in the pumping line or exhaust chamber.
6. Temperature of the parts was raised to the brazing temperature of $1,690^{\circ}$ in the case of some alloys and $1,975^{\circ}$ in the case of other alloys. Pressure during this period was maintained between 400–500 microns by varying the gas inlet from 8 CFH to 1 CFH depending on the outgassing rates.
7. After brazing was accomplished, temperature was lowered under 8 CFH flow conditions. At $1,400^{\circ}$ F the furnace was isolated from the vacuum pump and the pressure of argon increased to 5 inches of water column to speed cooling. Parts were cooled at this pressure level to below 350° F and then opened to atmosphere and pulled from furnace.

Parts done in this manner were exceptionally bright and well brazed on exit from the furnace. No supplementary cleaning procedures were used due to lack of oxidation.

EXAMPLE 5

In the heat treating of alloy steels the major requirement is a nonreactive atmosphere surrounding the part, so that oxides are not formed on the surface at elevated

temperatures. In the claimed process those oxides and other surface impurities are not formed, but in addition, those present at the start of the process are removed.

The following configuration of equipment was used:

1. External chamber with removable cover operating as work chamber and retort of conventional hot wall design of furnace. Size — circa 36 inches ID $\times$ 72 inches long.
2. A 10 inches ID $\times$ 4 feet long pipe section at right angle to the work chamber with a butterfly-type of valve to act as a restriction to the gas flow into the vacuum source. This functions as the outer or exhaust chamber.
3. A gas inlet into the bottom of the work chamber from an external gas source to provide a continuous flow of gas into the work chamber through a regulation valve and a dispersion grid.
4. Vacuum source — Standard Beach-Russ Company type of mechanical vacuum pump — 500 CFM capacity.

These process steps were followed:

1. Parts were placed in the work chamber and the cover closed.
2. The vacuum pump reduced the chambers to 50 microns Hg pressure.
3. External gas flow was started and balanced so that the pressure in the work chamber was 8,000 microns (8 torr). The pressure in the exhaust area was 800 microns. This resulted in an external gas flow of 9 CFH.
4. Temperature was raised to $1,800^{\circ}$ F. As the temperature rose, various levels of outgassing were noted and the gas inlet flow was adjusted accordingly, averaging 7 CFH flow.
5. Temperature was dropped on the parts by turning off the heating elements. Internal pressure in the work chamber was raised by increasing external gas flow and reducing the vacuum pumping speed by closing the restricting-isolation valve. This was done to hasten cooling.

Parts done in this manner were universally as good as parts done in conventional vacuum furnaces and exhibited much lower oxide coating.

The following is a summation of the system component parts and the process parameters for carrying out the teachings of the present invention.

The key parts of the system used to obtain the results of the process are:

1. A mechanical vacuum pump with the ability to reduce a chamber to an ultimate vacuum of 1–25 microns of Hg. No diffusion pumps or other special low vacuum pumps are used. Normally, rotary piston, oil sealed mechanical pumps such as are manufactured by Beach Russ-Company are used provided they have the throughput capacity required. No traps, cold traps, etc., are used in the pumping system. This is connected to the exhaust chamber by means of a pipe of appropriate diameter which may become the exhaust area, if the proper geometry, proper size ratio between work and exhaust areas is maintained, and proper orifice restrictions between the work area and exhaust area are maintained.
2. A work chamber, capable of maintaining a vacuum of from 100 microns to 10,000 microns of Hg. A

removable lid for entry and exit of parts is required.

3. An exit or exhaust chamber with a pressure of from 10 microns to 6,700 microns, which may physically surround the work chamber or be an appurtenant part to it is required. These chambers must have a fixed or variable gas flow path between them which is in relation to the gas inlet flow and the gas outlet flow into the mechanical pump in such wise as to maintain the two pressure area concept in the appropriate ratio with the work chamber being at higher pressure.
4. A gas flow system from an external gas source to the interior of the work area or chamber which may be regulated from 0.1 CFH to 10 CFM in concert with the speed of the vacuum pumping source and in relation to the size of the two areas and the orifice between them in such wise as to maintain the pressure ratio and the laminar flow conditions from the work chamber to the exhaust chamber. The process is carried out with a variable; but continuous, flow of external gas into the internal or work chamber. Since the pressure balance between the two discrete vacuum areas is extremely important to the process, the quantity of gas introduced may be reduced to compensate for the excessive gas emanating from the furnace and the articles being treated. In case of excessive formation of gas from the articles or furnace interior it may be necessary to temporarily stop the flow of gas from the external source until it is necessary to restart the flow in order to maintain the pressure balance between the two chambers required by the process.
5. Regardless of the physical geometry, the proper relationship between the work area pressure, exhaust are pressure (and their respective volumes), the orifice restriction between them, the gas inlet flow rate, and the gas flow outlet rates to the vacuum pump must be maintained so as to maintain the laminar flow conditions in the work area around the parts to be treated.

It will thus be seen that a unique process has been disclosed for treating parts in a vacuum and obtaining better treatment results than those obtained with much lower vacua and without the necessity of utilizing the highly sophisticated and expensive equipment necessary to obtain vacua below the order of 1 micron.

Although this invention has been described in its preferred form and preferred practice with a certain degree of particularity, it is understood that the present disclosure of the preferred form and preferred practice has been made only by way of example and that numerous changes in the details of construction and the combination and arrangement of parts and steps may be resorted to without departing from the spirit and the scope of the invention as hereinafter claimed.

What is claimed is:

1. The process of treating articles of manufacture comprising the steps of placing the articles within a first enclosure which is fluidly connected to a second enclosure through an orifice, continuously drawing a vacuum in said second enclosure from a vacuum source to produce a pressure of between 10 microns and 6,700 microns in said second enclosure, continuously introducing a gas under pressure from an exterior source into said first enclosure which gas may be inert, or functional so as to always maintain a higher gas pressure in said first enclosure than in said second enclosure and cause continuous gas flow from said first enclosure to said second enclosure through said orifice, said pressure in said first enclosure being between 100 microns and 10,000 microns, said continuous gas introduction into said first enclosure being at such a volumetric flow rate as to maintain said aforementioned pressures in said first and second enclosures.

2. The process of claim 1, wherein the ratio of pressure in said first enclosure to pressure in said second enclosure is in the range of from about 1.5:1 to about 20:1.

3. The process of claim 1, wherein the relationship between said first and second enclosures; the size of said orifice therebetween; and the gas throughput of said vacuum source being in such relationship as to permit the continuous external introduction of gas at flow rates in the range of from 0.1 CFH to 10 CFH.

4. The process of claim 3, wherein said orifice is variable.

5. The process of claim 1, wherein said articles of manufacture are heated to a temperature in the range of from ambient to 3,000° C.

6. The process of claim 1, wherein said gas which is introduced into said enclosure from said exterior source includes nitrogen, argon, oxygen, hydrogen, air or a hydrocarbon gas.

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