

[54] **APPARATUS FOR SINTERING PRESSED POWDER ELEMENTS CONTAINING HYDROCARBONS**

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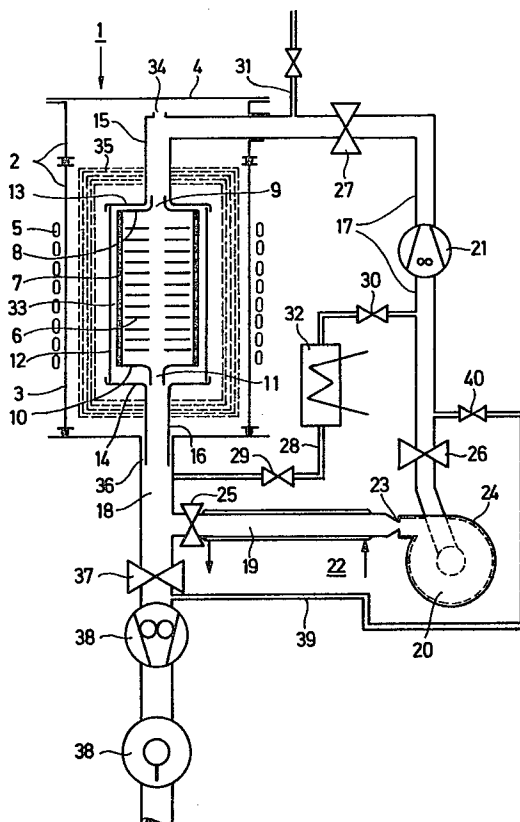
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[57] **ABSTRACT**

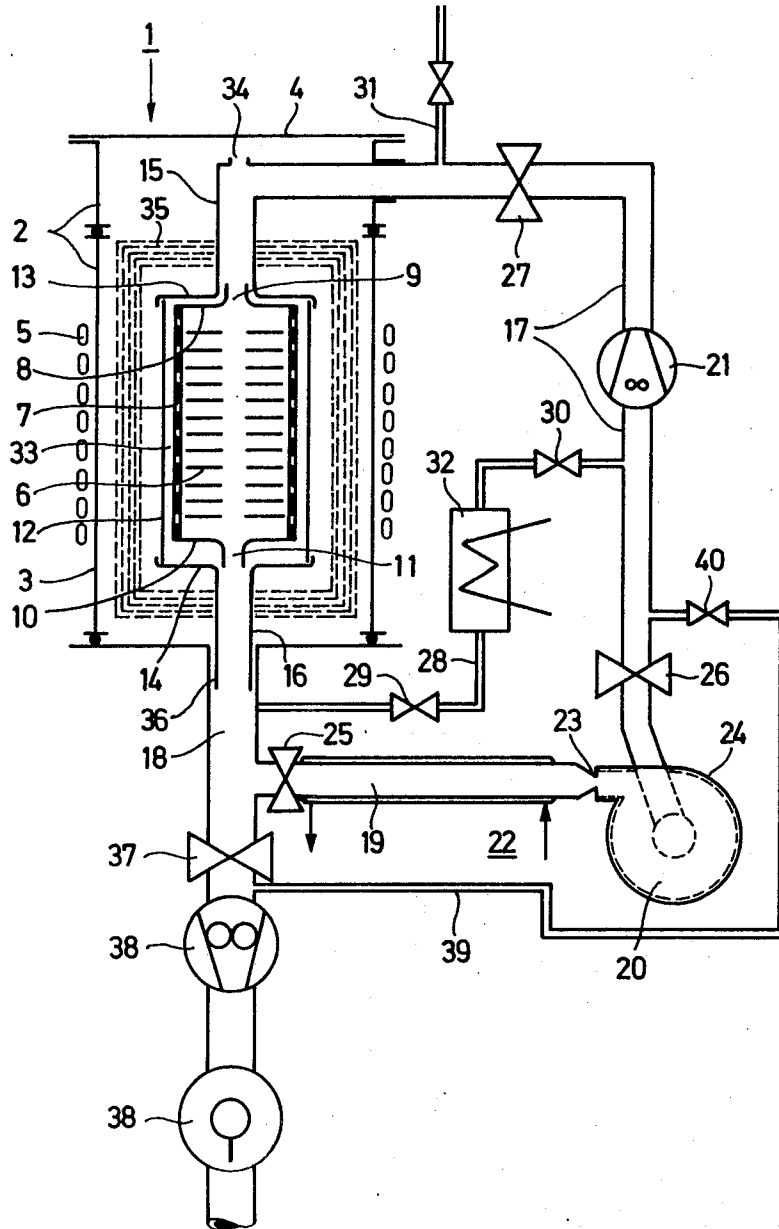
An arrangement for sintering under vacuum pressed powder elements which contain hydrocarbons. The hydrocarbons are driven out under lower pressure and higher temperature than ambient conditions. A flow of cleansing gas is then directed between the periphery of the pressed powder elements and the device for separating the hydrocarbons. This process step is carried out at a temperature between 100° and 800°C., while the pressure is below atmospheric. The sintering operation is then carried out after the hydrocarbons have been substantially driven out.

11 Claims, 1 Drawing Figure



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APPARATUS FOR SINTERING PRESSED POWDER ELEMENTS CONTAINING HYDROCARBONS

BACKGROUND OF THE INVENTION

The present invention relates to an arrangement for sintering pressed powder material elements containing hydrocarbons. In particular, the present invention relates to powder metal elements processed under vacuum. The hydrocarbons are driven out before the sintering operation takes place, under conditions of lower pressure and higher temperature in relation to ambient pressure and temperature.

Prior to pressing and sintering powderbased elements, hydrocarbons are added to these elements in the form of waxes to serve as bonding materials. These hydrocarbons must be removed from the pressed elements prior to sintering them at high temperatures. Without the removal of these hydrocarbons, they become cracked or fractionated within the pressed elements and the resulting carbon remains partially within these elements and functions as an impurity. The carbon residue in the pressed elements is undesirable in many applications. The bonding elements released during the cracking process are drawn into the vacuum pump and damage the pump to the extent that its operating life is substantially reduced, as well as contaminating the pumping oil. In view of these conditions, the dewaxing process was carried out in separate apparatus from the sintering process. This has incurred a more complex process apparatus, in addition to the requirement of transporting the pressed elements from the dewaxing apparatus to the sintering arrangement. Such conventional process arrangement has resulted in considerable operating personnel difficulties and time loss.

Since then it has also been known to carry out the dewaxing and sintering processes in a single apparatus arrangement, through the publication of H. Vollmer in "Performance of a High-Vacuum Induction-Heated Furnace with an Integrated Dewaxing Unit for Sintering Cemented Carbides," the Journal of Vacuum Science and Technology, Volume 8, No. 4.

In this publication, a carrier gas stream is applied during the entire dewaxing process. This carrier gas stream penetrates into the graphite holder for the pressed elements from the housing of the apparatus due to a pressure difference, and the gas stream is from there drawn into a vacuum pump. The gas becomes, thereby, loaded with the wax, and this wax is applied to the oil in the vacuum pump. For the subsequent sintering process, the dewaxing vacuum pump is turned off and a normal conventional pump is turned on.

The conventional arrangement described above has the disadvantage, however, of requiring a considerably large amount of gas which is also expensive and must be continuously maintained at a pressure which is below atmospheric pressure, by a vacuum pump. The requirement for switching pumps, furthermore, necessitates further separation of the process steps of dewaxing and sintering. As a result, the process is prolonged. Enrichment of the pump oil with the wax is, moreover, an undesirable disadvantage, since the oil must be thereby regenerated or exchanged. The wax condenser suggested there for larger apparatus can only delay the enrichment of wax in the pump oil, but it cannot prevent such enrichment.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide an arrangement in which the pressed elements may be dewaxed and sintered within the same apparatus, while avoiding the disadvantages of the conventional apparatus and methods.

It is another object of the present invention to provide an arrangement of the foregoing character which does not require the application of fresh cleansing gas which must then again be removed by pumping.

A further object of the present invention is to provide an arrangement, as described, in which the pump used for the dewaxing phase is required to overcome only a substantially light pressure difference, so that the pump oil is substantially free of hydrocarbons.

It is a still further object of the present invention to reclaim a substantially high proportion of the wax removed from the pressed elements.

It is a particular object of the present invention that the sintering process be directly connected to the dewaxing process through variation in the process parameters of pressure and temperature, so as to maintain down times or lag times at a minimum.

The objects of the present invention are achieved by providing a first process step in which the temperature of the pressed elements is between 100° and 800° C., and is preferably between 250° and 400°C. At the same time, the prevailing pressure is below atmospheric and is preferably between 500 and 0.1 torr. A flow of cleansing or scavaging gas is maintained between the periphery of the pressed elements and a separating arrangement for the hydrocarbons. After the gases have circulated and driven out the hydrocarbons, a second process step is initiated in which the actual sintering process is carried out.

In accordance with the present invention, furthermore, further advantages are realized as, for example, in that the vacuum enclosure does not require heating, since no wax can condense on it. It is possible to use highly porous materials for heat insulation, as well as low cost heating elements. For the purpose of circulating the gas, furthermore, it is sufficient to use a simple conventional circulating pump, since it is not necessary to maintain a substantially large pressure difference. Finally, the emptying of the separating arrangement may take place during the sintering process, so that the emptying or cleaning times do not result in lost time intervals. When discussing the periphery of the pressed elements herein, this description is to designate that the pressed elements are subjected directly to the circulating gas which passes by the elements. For this purpose, it is desirable that the elements be held in supports through which the gas may pass freely, or that the elements be suspended in baskets.

It is desirable that the dewaxing process begin at substantially high pressure of the cleansing or scavenging gas as, for example, at 500 torr. The pressure is thereafter gradually dropped until the end of the dewaxing interval. The reduction of the pressure during this interval is to be such that the percentage rate at which the hydrocarbons are released, remains constant during that interval. During the reduction in pressure, the temperature may be increased gradually to substantially 800°C., so that when the end of the driving out phase is attained, even the more obstinate residual hydrocarbons may be removed. The driving out process con-

nects, thereby, directly to the sintering process, since a temperature of substantially 800°C. can also be specified as the pre-sintering temperature. In accordance with the present invention, moreover, the pressure surrounding the pressed elements is reduced to a level below the final pressure of the first process step, when going from the first process step to the second process step.

The "separating device" can be in the form of various conventional apparatuses. Thus, a surface heat exchanger with fluid cooling can be used for this purpose. Other arrangements which serve to improve the operation of the separating process, are described in further detail below. The separating process runs particularly intensive, when the cleansing or scavenging gas is directed into the separating arrangement at substantially constant pressure for heat transfer to take place below the boiling point region and above the solidification point. It is, furthermore, possible to remove additional components of hydrocarbons from the cleansing or cooling gas, by subjecting the cooled gas from the separating arrangement to a further cooling process through expansion. The expansion process is applied so as to attain velocities below the speed of sound, and after the expansion of the gas the latter is recompressed to the initial pressure. The auxiliary cooling of the gas through the expansion process, leads to a precipitation or removal of the hydrocarbons which appear as a fine snow when using wax. By means of the substantially high flow velocities which have been attained in the expansion process, the mixture of gas and hydrocarbon particles can be directed to a mechanical separating arrangement, in which the components can be separated on the basis of their different masses or centrifugal forces.

It is also an object of the present invention to provide apparatus for carrying out the method of the invention. The apparatus has essentially a vacuum-tight furnace housing provided with a heating arrangement and support for the pressed elements. A pumping arrangement is connected to the furnace housing for maintaining a vacuum therein, and a separating unit or device is provided for separating the hydrocarbons. The separating arrangement and the furnace housing, furthermore, are interconnected with a flow line, in accordance with the present invention.

The separating arrangement is designed, in accordance with the present invention, in a particularly advantageous manner, whereby a series interconnection includes a surface heat exchanger, an expansion device, a solids particle separator, and a compressor. The expansion device is preferably in the form of a nozzle, since this provides the substantially high flow velocities required for the subsequent solids or particle separation. The solids particle separator can, thereby, be in the form of a cyclone separator, for example. The inner walls of this cyclone separator are provided with an irregular or roughened surface structure which may be in the form of, for example, steps, ribs, webs, or a simple grid or mesh on the interior wall of the cyclone. The irregular or roughened surface structure serves the purpose of catching the hydrocarbon particles. At the same time, the expansion device is arranged in an advantageous manner so that its entrance to the particle separator is such that the expanded gas stream is essentially tangential in relation to the wall of the particle or solids separator.

In order that emptying and cleansing of the separating arrangement may be carried out during the sintering process and thereby return the separator to its operative state, a valve is provided within the gas flow line in front and behind the separating unit. For the purpose of shortening the process cycle, furthermore, it is advantageous that upon ending the sintering process, the charge be cooled as rapidly as possible to a temperature at which it is possible to remove the pressed elements without damaging them due to atmospheric effects. To achieve this object, a bypass line is provided, in accordance with the present invention which is parallel to the surface heat exchanger, the expansion device and the solids or particle separator. This bypass line includes, moreover, an auxiliary heat exchanger and at least one valve. The auxiliary heat exchanger is a gas cooling device through which inert gas admitted into the apparatus after completing the sintering process, is continuously cooled. The pump which is used for transporting the cleansing or scavaging gas, as well as for the driving out phase, serves also to circulate the cooling inert gas to the bypass line.

A valve is, furthermore, located in the vacuum or suction line between the furnace housing and the pump unit. The side of the valve connected to the furnace housing communicates with a pipeline which is connected to the suction or vacuum line through a regulating valve. This design makes it possible to remove a portion of the gas and thereby reduce the pressure during the driving out phase and preferably at the end of this phase. The removal of the cleansing or scavenging gas is performed at a location where the hydrocarbons have already been separated.

The interior of the furnace housing has a series of built-in components or units based on mechanical and thermal considerations. Thus, for purposes of supporting the pressed elements, a supporting unit is provided at the central core of the furnace. This supporting unit is, for example, surrounded by an interior housing which is, in turn, arranged concentrically within a heating cylinder. In order to maintain thermal losses at a minimum, a radiation screen is located between the heating cylinder and the furnace housing. This screen is constructed of, for example, fibrous or granular insulating material, or from a plurality of spaced radiation sheet elements. The heating cylinder can, thereby, be heated by the directed application of current. However, it is also possible to provide an induction coil on the exterior of the furnace housing, whereby the heating effect results from high frequency alternating current within the coil.

In order to prevent deposition of hydrocarbons on the internal components particularly in dead corners within the furnace housing, there is provided in accordance with the present invention, that the heating arrangement has a heating cylinder which encloses or surrounds the support for the pressed elements and is within the path of the flow line for the cleansing or scavaging gas. An interior chamber, furthermore, is arranged between the heating cylinder and the support for the pressed elements, which has inlet and outlet openings that are also located within the path of the cleansing or scavaging gas. The cross-sectional area between the heating cylinder and the interior chamber has a magnitude relative to the cross-section of the interior chamber itself, so that increased static pressure prevails within the space between the heating cylinder

and the interior chamber. In view of this pressure drop, hydrocarbons cannot penetrate the space between the heating cylinder and the interior chamber, even when a tight seal is not present. The corresponding design for realizing the pressure drop in an advantageous manner and thereby enhancing the cleansing effect, is obtained by providing openings in the wall of the interior chamber in the region of the support or the pressed elements. The cleansing gas passes through these openings on all sides of the interior chamber and passes uniformly over the pressed elements.

It is also desirable to maintain a still higher pressure within the furnace housing in the space in which the heat screen is located. This feature is accomplished by providing an outlet opening in the part of the gas flow line within the furnace housing. This outlet opening is in front of the entrance to the heating cylinder, when viewed in the flow direction. With this arrangement, the circulating flow line communicates with the space between the furnace housing and the heating cylinder. The furnace housing, furthermore, connects to the circulating flow line at a location spaced from the outlet opening. The applicable part is made to communicate with the flow line, while the corresponding dimensioning of the flow cross section provides for the desired pressure drop in the directions to the core of the furnace.

The novel features which are considered as characteristic for the invention are set forth in particular in the appended claims. The invention itself, however, both as to its construction and its method of operation, together with additional objects and advantages thereof, will be best understood from the following description of specific embodiments when read in connection with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWING

A schematic elevational view through the furnace housing and interconnecting devices arranged to carry out the method in accordance with the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawing, a vacuum sintering furnace 1 has a furnace housing 2. This housing 2 has a lower part 3 and an upper part 4 which may be removed for the purpose of charging the furnace. The housing 2 is made of substantially non-magnetic material such as quartz, for example, and exterior to this housing is an induction coil 5. Within the interior of the housing and at substantially the core of the furnace, is a rack 6 for treatment of the powdered metal elements. This rack 6 is in the form of a multiple number of carrying plates. The rack 6, furthermore, is surrounded by an interior housing 7 in the form of a hollow cylinder which has uniform spacings in the vertical direction. The interior housing 7 has at its top, a cover 8 provided with a central opening 9. At the lower end of this interior housing 7, is a cover 10 with a corresponding opening 11.

The interior housing 7 is surrounded by a heating cylinder 12 which is held or supported within two covering elements 13 and 14. A pipe or tube member 15 communicates with the upper covering element 13. A tube or pipe 16, furthermore, communicates with the lower covering element 14, and leads to a vacuum line 18. The tubes or pipe elements 15 and 16 are part of a cir-

cuit 17 which branches off from the vacuum line 18 and passes through a surface heat exchanger 19, a particle separator 20 in form of a cyclone, and a blower 21. From this blower 21, the circuit returns to the furnace housing 2. The surface heat exchanger 19, has the function and construction of a converter. The parts 19, 20 form a separating arrangement 22. At the entrance of the particle separator 20, is an expansion arrangement 23 in the form of a nozzle, which applies a stream tangentially to the particle separator. The interior surface of the particle separator has a structure 24 in the form of, preferably, laminations or a metallic mesh or grid against which the hydrocarbon solids or particles may strike.

Within the circuit 17, are valves 25, 26 and 27. Upon closing the valves 25 and 26, the separation arrangement 22 can be opened for the purpose of evacuation and cleansing. In parallel with the separation arrangement, is a bypass line 28 containing valves 29 and 30. When the sintering process has ended, an inert gas is circulated through the bypass line, by means of the blower 21. The inert gas is admitted into the circuit through the line 31. The inert gas, furthermore, is conducted through a heat exchanger 32 where the inert gas transfers the heat received from the powdered metal elements or pressed elements in the furnace. Upon closing the valve 27, the entire circuit may be isolated from the furnace housing. At the beginning of the process, the cleansing or flushing gas is also admitted through the line 31.

The opening 9 within the interior housing 7 and the tube or pipe element 15, is dimensioned so that upon flow of the cleansing gas, the ring-shaped space 33 between the interior housing 7 and heating cylinder 12 has substantially a higher pressure than the pressure prevailing within the interior housing 7. The pressure relationship is also achieved by dimensioning of the opening 11 of the interior housing 7 and the tube or pipe element 16. The part of the circuit 17 within the furnace housing 2 has, furthermore, an exit opening 34 through which the cleansing gas passes during the cleansing or flushing phase in the space between the furnace housing 2 and the heating cylinder 12. Within the space between the housing 2 and heating cylinder 12, furthermore, is a flow screen 35. The exit of the cleansing gas from this space or compartment between the housing 2 and cylinder 12, occurs through a ring-shaped opening 36 which is formed from the relative dimensions of the tube or pipe element 16 and the vacuum line 18. The relative cross sections are designed here also so that the pressure within the space containing the flow screen is slightly higher than the pressure within the ring-shaped space 33.

The vacuum line 18 communicates with a pump 38, through the valve 37. The valve 37 and the circuit line 17 are bridged by a line 39 having a valve 40 which controls the flow through the line 39. The line 39 serves the purpose of dropping the pressure within the system during the cleansing or ejection phase, to the pressure level which is to be maintained within the system during the sintering phase. For this purpose, it is required that at least the valve 27 is open.

For the purpose of illustrating an example in the use of the process and arrangement, in accordance with the present invention, Tungsten carbide elements as, for example, cutting discs were subjected to the process. These Tungsten carbide elements were pressed with a

2% by weight proportion of paraffin. Paraffin has a solidification point between 48° and 60° C.

At the beginning of the process, the valves 29, 30 and 40 were closed, and the valves 25, 26, 27 and 37 were opened. By means of the pump 38, the furnace was evacuated to a pressure of 10^{-2} torr. Thereafter the valve 37 was closed. Argon was then admitted through the line 31 for cleansing purposes, until the pressure within the system had increased to 200 torr. The line 31 was then closed. The compressor 21 and, at the same time, the induction coil 5 were set into operation. After 2.5 hours, the sintering elements within the sintering furnace have attained a temperature of 300°C. This temperature was maintained for 30 minutes. During this entire time, a strong flow of cleansing or scavaging gas was maintained by the compressor 21. The compressor delivered 130 m³/hr.

The throttle valve 40 was then opened and the pressure within the system was dropped within an hour to 10^{-1} torr. At the same time, the temperature of the sintering elements was increased to 800° C. After attaining the preceding conditions corresponding to the pre-sintering temperature, the ends of the dewaxing process was attained.

Valves 25, 26 and 40 were next closed, and the valve 37 was opened. By means of the pump 38, the pressure was dropped to 10^{-3} torr. and the temperature was increased to 1,400° C. within 60 minutes. The actual sintering process was, thereby, initiated, and was carried out for a period of 30 minutes. The valve 37 was, thereafter, again closed, and inert gas was admitted to the line 31 until atmospheric pressure was substantially attained. By opening the valves 29 and 30, and setting the blower 21 into operation, the inert gas was circulated through the heat exchanger 32 for the purpose of obtaining rapid cooling. The entire process cycle took place over an interval of substantially 11 hours.

The sintered elements were substantially free of cracked products, while, at the same time, the interior of the furnace housing was also free of cracked products and wax. The paraffin could again be used after being taken from the separating arrangement 22.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can, by applying current knowledge, readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention, and therefore, such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

What is claimed as new and desired to be protected by Letters Patent is set forth in the appended claims:

1. A vacuum-furnace arrangement for sintering pressed powdered elements containing hydrocarbons under vacuum and elevated temperatures, comprising in combination a vacuum tight furnace housing with heating means; support means within said housing for supporting said elements; first pumping means connected to said housing for producing a vacuum within said housing; separating means for separating said hydrocarbons; cycle of flow interconnecting means be-

tween said housing and said separating means; and second pumping means within said cycle of flow interconnecting means for circulating the furnace atmosphere through the said separating means.

2. The arrangement as defined in claim 1 wherein said separating means comprises a series interconnected arrangement of a surface heat exchanger; expansion means; a solids particle separator; and blowing means.

3. The arrangement as defined in claim 2, wherein said solids particle separator comprises a cyclone separator.

4. The arrangement as defined in claim 3 wherein said expansion means is directed into said solids particle separator means so that the expanded flow from said expansion means is substantially tangential to the wall of said solids particle separator means.

5. The arrangement as defined in claim 3 wherein said cyclone separator has interior walls with irregular surface.

6. The arrangement as defined in claim 4 including valve means in said flow interconnecting means and in front of said separating means and in back of said separating means.

7. The arrangement as defined in claim 1 wherein said heating means comprises a heating cylinder substantially surrounding said elements; and an inner housing between said heating cylinder and said support means, said inner housing having inlet and outlet openings communicating with said flow interconnecting means.

8. The arrangement as defined in claim 7 wherein the wall of said inner housing has openings in the region of said support means for said elements.

9. The arrangement as defined in claim 7 wherein said interconnecting means has an outlet opening in the portion emerging from said furnace housing, said flow interconnecting means communicating with the space between said furnace housing and said heating cylinder.

10. The arrangement as defined in claim 1, including vacuum line means between said first pumping means and said furnace housing; valve means in said vacuum line, pipe line means in said cycle of flow interconnecting means to said vacuum line behind said valve means in said vacuum line when viewed from said housing.

11. An arrangement for sintering pressed powdered elements containing hydrocarbons under vacuum comprising, in combination, a vacuum tight furnace housing with heating means; support means within said housing for supporting said elements; pumping means connected to said housing for producing a vacuum within said housing; separating means for separating said hydrocarbons; flow interconnecting means between said housing and said separating means, said separating means comprising a series interconnected arrangement of a surface heat exchanger, expansion means, a solids particle separator, and blowing means; and auxiliary heat exchanging means and valve means connected in parallel with said surface heat exchanger, expansion means and solids particle separator means.

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