



1. A cryocooling system comprising: a sample well and a sample chamber, each of the sample well and sample chamber defining an internal volume that is sized and shaped to hold a cryocooling gas at cryogenic temperatures and a pressure below 1 atm; a cryocooler in thermal communication with the sample well, the cryocooler being adapted to cool a cryocooling gas inside the sample well to less than or equal to 10 Kelvin; an impedance tube having a first end and a second end, the first end being connected to and in fluid-flow communication with the internal volume of the sample well and the second end being connected to and in fluid-flow communication with the internal volume of the sample chamber to allow cryogenic gas to move from the sample well to the sample chamber, the impedance tube having a first hydraulic resistance; and a vacuum tube having a first end and a second end, the first end being connected to and in fluid-flow communication with a vacuum pump via a vacuum port and the second end being connected to and in fluid-flow communication with the internal volume of the sample chamber, the vacuum tube having a second hydraulic resistance; wherein the first hydraulic resistance being higher than the second hydraulic resistance.
2. The cryocooling system of claim 1, wherein the ratio of the first hydraulic resistance to the second hydraulic resistance is between 40:1 and 50:1.
3. The cryocooling system of claim 1, wherein the vacuum tube has an outer surface and the impedance tube is coiled around the outer surface of the vacuum tube.
4. The cryocooling system of claim 1, wherein the sample chamber is nested within the sample well.
5. The cryocooling system of claim 1, further comprising a controller that is configured to isolate the internal volumes of the sample well and sample chamber from a supply of cryocooling gas after the cryocooling gas inside the sample chamber has reached a first temperature, and open the vacuum port and activate the vacuum pump when the cryocooling gas inside the sample chamber has reached a second temperature, wherein the second temperature is lower than the first temperature.
6. The cryocooling system of claim 1, further comprising a sample well shroud that holds the sample well in a vacuum and a sample chamber shroud that houses the sample chamber in a vacuum.
7. The cryocooling system of claim 6, wherein the sample chamber includes a sample plate that is configured to hold a sample substance to be cooled outside of the sample chamber.
8. The cryocooling system of claim 7, wherein the sample plate is made from copper.
9. The cryocooling system of claim 1, further comprising a return gas tube in fluid-flow communication with the sample well, the return gas tube being configured to receive exhausted cryocooling gas removed from the sample chamber via the vacuum tube and feed the exhausted cryocooling gas into the sample well.
10. A method of cryocooling using a cryocooling system, the cryocooling system including a sample well, a sample chamber, an impedance tube in fluid-flow communication with the sample well and the sample chamber, a cryocooler in thermal communication with the sample well, a gas inlet valve connected to the sample well, and a vacuum tube in fluid-flow communication with the sample chamber via a vacuum port, the method comprising: introducing a cryocooling gas into the sample well and the sample chamber via the gas inlet valve; activating the cryocooler to cool the cryocooling gas in the sample well and the sample chamber; closing the gas inlet valve when a temperature of the cryocooling gas in the sample chamber has reached a first cryogenic temperature; opening the vacuum port when the temperature of the cryocooling gas in the sample chamber is has reached a second cryogenic temperature; and withdrawing cryocooling gas from the sample chamber through the vacuum tube to cause the temperature of the cryocooling gas in the sample chamber to fall below a third cryogenic temperature, wherein the third cryogenic temperature is lower than the second cryogenic temperature, and the second cryogenic temperature is lower than the first cryogenic temperature.
11. The method of claim 10, further comprising inserting a sample to be cooled in the sample chamber.



systems have two chamber systems that require restrictions in the mass flow rate of a fluid from one chamber to another in order to create pressure differences between two different chambers. If there is no viscosity in the fluid, there is no practical way to restrict mass flow rates to create the required pressure drops. Furthermore, a helium fluid having infinite thermal conductivity means that the temperature of the volume of fluid is practically uniform, making it particularly difficult to reduce its temperature below the critical temperature (i.e., its Lambda point).

[0003] Current cryocooling systems that use helium-4 as the cooling medium are able to achieve temperatures as low as 1.7 K by reducing the pressure of the helium-4. However, such systems have difficulty achieving such low temperatures for long periods of time (e.g., greater than 60 minutes) without inducing a phase change in the helium-4 into a super fluid. By contrast, Helium-3 does not have a known Lambda point (it is believed to be as low as 2.5 mK), and thus does not present a superfluid issue. Having such properties, helium-3 has been a popular medium for cryocooling applications that require temperatures below 1.7 K.

[0004] Unfortunately, helium-3 is very rare. Sources of natural gas that contain helium-3 are limited, and atmospheric helium migrates into space and is lost. Because helium-3 is such a limited resource and its demand for use in cryocooling applications has increased dramatically, the cost of helium-3 has also increased dramatically. Accordingly, there is a need for a cryocooling system that is capable of maintaining temperatures at or below 1.7 K in a sample chamber for an extended period of time without having to use helium-3.

## SUMMARY

[0005] In view of the foregoing background, a cryocooling system is disclosed. The system includes a sample well and a sample chamber, each of which defining an internal volume that is sized and shaped to hold a cryocooling gas, such as helium-4, at cryogenic temperatures and a pressure below 1 atm. The sample chamber is also sized and shaped to hold a sample substance to be cryocooled. An impedance tube with a first and second end connects the interior of the sample well to the interior of the sample chamber to allow cryocooling gas to move from the sample well to the sample chamber. A vacuum tube is connected to the interior of the sample chamber at a first end and to a vacuum pump via a vacuum port at a second end. The vacuum tube is sized and shaped to allow cryocooling gas within the sample chamber to be pumped out of the sample chamber by the vacuum pump.

[0006] In one aspect of the invention, the impedance tube has a first hydraulic resistance and the vacuum tube has a second hydraulic resistance, wherein the first hydraulic resistance is higher than the second hydraulic resistance. In one embodiment, the ratio of the first hydraulic resistance to the second hydraulic resistance is between 40:1 and 50:1.

[0007] In another aspect, the sample chamber is nested within the sample well. In one embodiment, the vacuum tube has an outer surface, and the impedance tube is coiled around the outer surface of the vacuum tube to conserve space inside the sample well and increase the hydraulic resistance of the impedance tube.

[0008] In another aspect, the cryocooling system includes a controller that is configured to isolate the internal volumes of the sample well and sample chamber from a supply of cryocooling gas after the cryocooling gas inside the sample chamber has reached a first temperature, and open the vacuum port and activate the vacuum pump when the cryocooling gas inside the sample chamber has reached a second temperature, wherein the second temperature is lower than the first temperature.

[0009] In another aspect, the sample well and sample chamber are held in shrouds that hold the sample well and sample chamber in a vacuum. In one embodiment, the sample chamber includes a sample plate made from copper that holds a sample substance outside of the sample chamber and in the shroud's vacuum.

[0010] In another aspect, the cryocooling system includes a return gas tube that is in fluid-flow communication with the sample well so as to recycle exhausted cryocooling gas back into the cryocooling system.



[0021] FIG. 4 is a sectional view taken along line 4-4 of FIG. 3;

[0022] FIG. 5 is an enlarged partial view of area A-A of FIG. 4;

[0023] FIG. 6 is an exploded perspective view of a sample stick from the cryocooling system shown in FIGS. 3-5;

[0024] FIG. 7 is a perspective view of a second exemplary embodiment of a cryocooling system;

[0025] FIG. 8 is a sectional view taken along line 8-8 of FIG. 7;

[0026] FIG. 9 is an enlarged partial view of area B-B of FIG. 8;

[0027] FIG. 10 is a side elevational view of a third exemplary embodiment of a cryocooling system;

[0028] FIG. 11 is a sectional view taken along line 11-11 of FIG. 10;

[0029] FIG. 12 is an enlarged partial view of area C-C of FIG. 11;

[0030] FIG. 13 is a chart showing the results of a sample test of the cryocooling system shown in FIGS. 3-5, which is described in detail in Example 1 further below; and

[0031] FIG. 14 is a chart focusing on a portion of the results shown in FIG. 13.

#### DETAILED DESCRIPTION OF THE INVENTION

[0032] The following disclosure is presented to provide an illustration of the general principles of the present invention and is not meant to limit, in any way, the inventive concepts contained herein. Moreover, the particular features described in this section can be used in combination with the other described features in each of the multitude of possible permutations and combinations contained herein.

[0033] All terms defined herein should be afforded their broadest possible interpretation, including any implied meanings as dictated by a reading of the specification as well as any words that a person having skill in the art and/or a dictionary, treatise, or similar authority would assign particular meaning. Further, it should be noted that, as recited in the specification and in the claims appended hereto, the singular forms "a," "an," and "the" include the plural referents unless otherwise stated. Additionally, the terms "comprises" and "comprising" when used herein specify that certain features are present in that embodiment, but should not be interpreted to preclude the presence or addition of additional features, components, operations, and/or groups thereof.

[0034] The following disclosure is intended to be read in connection with the accompanying drawings, which are to be considered part of the entire written description of the invention. The drawing figures are not necessarily to scale and certain features of the invention may be shown exaggerated in scale or in somewhat schematic form in the interest of clarity and conciseness. In this description, relative terms such as "horizontal," "vertical," "up," "down," "top," "bottom," as well as derivatives thereof (e.g., "horizontally," "downwardly," "upwardly," etc.) should be construed to refer to the orientation as then described or as shown in the drawing figure under discussion. These relative terms are for convenience of description and normally are not intended to require a particular orientation. Terms including "inwardly" versus "outwardly," "longitudinal" versus "lateral" and the like are to be interpreted relative to one another or relative to an axis of elongation, or an axis or center of rotation, as appropriate. Terms concerning attachments, coupling and the like, such as "connected" and "interconnected," refer to a relationship wherein structures are secured or attached to one another either directly or indirectly through intervening structures, as well as both moveable or rigid attachments or relationships, unless expressly described otherwise, and includes terms such as "directly" coupled, secured, etc. The term "operatively coupled" is such an attachment, coupling, or connection that allows the pertinent structures to operate as intended by virtue of that relationship.





the higher temperature helium-4 tends to be gaseous, positioning the intake portion of the impedance tube 20 in the upper portion of the sample well 12 ensures that only gaseous helium-4 travels from the sample well 12 to the sample chamber 14. This is to keep the pressure in the sample well 12 steady, as liquid helium-4 at the bottom of the sample well 12 provides the gaseous helium-4 by vaporizing at constant pressure.

[0049] FIGS. 3-6 and 7-9 illustrate first and second embodiments, respectively, of the cryocooling system 10 discussed above and illustrated in FIG. 1. The elements illustrated in FIGS. 3-5 and 7-9, which correspond to the elements described above with respect to the diagram shown in FIG. 1, have been designated by corresponding reference numbers increased by one hundred and two hundred, respectively. Any element referenced below and identified in the attached drawings should be assumed as having the same or similar structure and function as its corresponding element shown in previous figures, except where specifically indicated otherwise below.

[0050] FIGS. 3-5 show one embodiment of a cryocooling system 210 capable of performing the cryocooling method 100 discussed above. The cryocooling system 210 includes a vacuum shroud (i.e., upper shroud 228 and lower shroud 230) that encases the cryocooler 216, the sample well 212, and the sample chamber 214 within a vacuum and is equipped with a radiation shield 232 that prevents light and other outside sources of energy from affecting the temperature of the parts therein. The cryocooler 216 is a standard two-stage cryocooler well known in the art and is connected to the sample well 212 via first and second heat exchangers 234a, 234b which correspond to the first and second stages of the cryocooler 216 and allow for the transfer of energy from the gas inside the sample well 212 to the cryocooler 216. In one embodiment, the cryocooler 216 has a first stage that can reach 20 K and a second stage that can reach 2.5 K. The sample chamber 214 is positioned below and away from the cryocooler 216.

[0051] As seen in FIGS. 4 and 5, the cryocooling system 200 is designed to be a compact, space-saving embodiment of the cryocooling system 10 shown in FIG. 1. As such, the sample chamber 214 of the cryocooling system 200 is nested within the sample well 212, and the vacuum tube 222 runs from the sample chamber 214 up through the sample well 212 and all the way to the top of the upper shroud 228, where the vacuum port 224 is located. The gas inlet valve 218 is located proximate to the vacuum port 224, which allows a user to operate both from one location.

[0052] As seen in FIG. 6, in one embodiment, the sample chamber 214, the impedance tube 220, and the vacuum tube 222 are part of a sample stick 240 that can be inserted into the sample well 212. The sample chamber 214 is attached to the end of the vacuum tube 222 that includes several conduction rings (i.e., conduction rings 242a, 242b) located along the length of the vacuum tube 222 and has a sample stick cap 244 just below the vacuum port 224 that closes off and seals the top of the sample well 212. The conduction rings 242a, 242b are located at points along the length of the vacuum tube 222 that correspond to the position of the first and second heat exchangers 234a, 234b connected to the cryocooler 216 to assist the cryocooler 216 in cooling the cryocooling gas in the sample well 212. Because the length of the vacuum tube 222 extends beyond the height of the upper shroud 228, the portion of the impedance tube 220 that receives gas from the sample well 212 is similarly long in order to maintain its higher hydraulic resistance. This portion of the impedance tube 220 is wrapped around the vacuum tube 222 to conserve space inside the sample well 212.

[0053] As seen in FIGS. 4 and 5, the impedance tube 220 has a length of  $L_{sub.i}$  and a diameter of  $2R_{sub.i}$ , while the vacuum tube 222 has a length of  $L_{sub.v}$  and a diameter of  $2R_{sub.v}$ . In one embodiment, the impedance tube 220 and vacuum tube 222 are made from stainless steel, with the impedance tube 220 having a length of 37 inches ( $L_{sub.i}=37$  in), a diameter of 3/16 inch ( $R_{sub.i}=3/32$  in), and a wall thickness of approximately 0.020 inches, and the vacuum tube 222 having a length of 26.25 inches ( $L_{sub.v}=26.25$  in), a diameter of 1 inch ( $R_{sub.v}=0.5$  in), and a wall thickness of 0.016 inches.

[0054] FIGS. 7-9 illustrate a second embodiment of a cryocooling system 310 configured to perform the cryocooling method 100 shown in FIG. 2. In this embodiment, the sample well 312 and the sample chamber 314 are located in separate shrouds (i.e., the sample well shroud 327 and the sample chamber shroud 329) and connected by a tubular shroud 331 that houses a portion of the impedance tube 320 connecting the sample well 312 to the sample chamber 314. In addition, the sample chamber 314 includes a sample plate 315 located at the

top of the sample chamber 314 upon which the sample which is to be cooled may sit. In this manner, the cryocooled gas inside the sample chamber 314 is able to cool the sample through the sample plate 315. This allows the sample to be placed in a vacuum space within the radiation shield 332 while being cooled through the sample plate 315.

[0055] As a result of not being nested within the sample well 312, the sample chamber 314 is able to be placed in a vacuum within the sample chamber shroud 329. This arrangement allows for better control over the environment of the sample chamber 314 compared to the vapor environment of the sample chamber 214 shown in FIGS. 3-5, allowing for the sample chamber 314 to reach temperatures of 1.5 kelvin or less over a duration of several hours.

[0056] FIGS. 10-12 illustrate a third embodiment of a cryocooling system 410 configured to perform the cryocooling method 100 shown in FIG. 2. The cryocooling system 410 is similar to the cryocooling system 310 shown in FIGS. 7-9, except that the tubular shroud 431 is shorter than its counterpart shown in FIGS. 7-9. This embodiment conserves space while maintaining the advantages of having a sample chamber 414 located outside of the vapor environment of the sample well 412. The cryocooling system 410 also includes a return gas tube 423 that runs from the vacuum port 424 through the sample chamber shroud 429, the tubular shroud 431, and into the sample well 412. The return gas tube 423 allows exhausted gas that was once pumped out of the sample chamber 414 via the vacuum tube 422 to be fed back into the sample well 412 as part of a recycling system. The return gas tube 423 can also operate as the path through which cryocooling gas is initially introduced to the sample well 412 and the sample chamber 414.

[0057] Multiple variations of the above-described embodiments can be made without departing from the present invention. For instance, in one embodiment of the cryocooling method 100 described above, the clean out step 102 occurs by pumping air out of the cryocooling system 10 through the vacuum tube 22 and the vacuum port 24 while cryocooling gas is introduced to the sample well 12 via the gas inlet valve 18. In an alternative embodiment, the clean out step 102 can pump air out of the gas inlet valve 18 while the cryocooling gas is introduced to the sample chamber 14 via the vacuum tube 22.

[0058] In one embodiment of either the cryocooling system 310 shown in FIGS. 7-9 or the cryocooling system 410 shown in FIGS. 10-12, the sample to be cryocooled may be placed within the sample chamber 314, 414, rather than on top of the sample plate 315, 415, to provide a vapor environment instead of a vacuum environment. In another embodiment, the sample plate 315, 415 may be placed at the bottom of the sample chamber 314, 414, thereby making greater contact with the cooler helium-4 that has fallen toward the bottom of the sample chamber 314, 414. In one embodiment of the cryocooling system 210 shown in FIGS. 3-5, the sample chamber 214 may sit at the bottom of the sample well 212, which can be made of copper and act as a cold plate, allowing the sample to be placed in a vacuum in a manner similar to those of the cryocooling systems 310, 410 of FIGS. 7-12.

[0059] In embodiments where a controller 25 is used, a heater (not shown) may be added to the cryocooling system 10 to help control the temperature and pressure inside the sample well 12 and sample chamber 14.

[0060] The disclosure is further illustrated by the following examples, which are not to be construed as imposing limitations on the scope of the present invention. Various other aspects, embodiments, modifications, and equivalents thereof which, after reading the description herein, may suggest themselves to one of ordinary skill in the art without departing from the spirit of the present invention.

#### Example 1

[0061] A sample test was conducted using the cryocooling system 210 shown in FIGS. 3-5. Charts showing the results of this second test are exhibited in FIGS. 13 and 14. In this test, the impedance tube 220 had a 28.35 inch length, a 0.125 inch outer diameter, and a 0.016 inch wall thickness, while the vacuum tube 222 had a 26.50 inch length, a 0.750 inch outer diameter, and a 0.016 inch wall thickness.



