Phase boundary detection for dilution refrigerators

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We describe a device to conveniently measure the positions of the phase boundaries in a dilution refrigerator. We show how a simple modification of a standard capacitive level gauge (segmentation of one of the electrodes) permits a direct calibration of the capacitance versus phase boundary position. We compare this direct calibration with the indirect procedure that must be adopted for a conventional capacitive level gauge. The device facilitates the correct adjustment of the $^3$He/$^4$He fraction in the dilution refrigerator.

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I. BACKGROUND

Dilution refrigerators (DR) have made the temperature region below 0.5 K widely available to the research community. While the continuing development of the technologies involved have made these machines very reliable, good diagnostic devices are essential for their continuing and reliable operation.

Various problems can impede the correct operation of a dilution refrigerator. Leaks to vacuum, blockages and thermal shorts usually lead to catastrophic failure, and are easy to diagnose, but difficult to solve. In contrast, not having the right amounts of $^3$He and $^4$He in the system is easily solved, but may not be readily recognized.

For optimal performance of the DR, the total amount of mixture, and the $^3$He/$^4$He fraction of the mixture must be tuned, which means putting the phase boundaries in the still (between the diluted liquid and the vapour phase) and in the mixing chamber (between the concentrated $^4$He and the $^3$He diluted in $^4$He phases) at the correct positions. The still level is determined by the total quantity of mixture in the DR. If this level is too low (below the still heater or in the heat exchanger) it will be difficult to pump and circulate the $^3$He. If the level is too high (in the pumping line of the still) again it will be difficult to pump the $^3$He and large amounts of $^4$He may be circulated. Both circumstances will degrade the performance of the DR. At the phase boundary in the mixing chamber level the dilution cooling takes places and it is the coldest position in the mixing chamber. This phase boundary must not be up in the heat exchangers, since the cooling power of the DR will suffer. It may be advantageous in some cases to be able to control the position of the phase boundary to put it close to the sample.

Tuning the amounts of $^3$He and $^4$He is not a one time operation because changes may occur over time, leading to degraded performance. If the sample is mounted in the mixing chamber, the mixing chamber volume will depend on the sample size, which will lead to a different optimal quantities of $^3$He and $^4$He. For small DR’s with large $^4$He circulation pumps (to get high flow rates and cooling powers) the large fraction of dead volume behind the pumps makes the $^3$He/$^4$He fraction in the DR depend on the operating conditions. If the circulation rate, and therefore the inlet pressure of the $^4$He return line, is varied the amount of $^3$He in the DR will change too.

For all of these reasons, a good measurement of the positions of the phase boundaries in the still and in the mixing chamber is very useful for finding optimal working conditions of the DR. Although it is possible to infer the positions of the phase boundaries using only thermometers at strategic places [1], it is preferable to have real level gauges in the still and the mixing chamber, providing a direct measurement.

II. PHASE BOUNDARY LEVEL GAUGES

We focus here on capacitive level gauges [2] which may be parallel plates or concentric cylinders. The principle of operation is simplicity itself: the capacitance is given by

$$C(x) = C_s + C(0)[\epsilon_a + x(\epsilon_b - \epsilon_a)]$$  \hspace{1cm} (1)

with $x$ the fractional level, $\epsilon_{a,b}$ the relative dielectric constants of the phases above and below the phase boundary at $x$, and $C_s$ the inevitable stray capacitance. Because of this stray capacitance, the most convenient mode of operation is to measure simply $C(0)$ and $C(1)$, the ‘empty’ ($x=0$) and ‘full’ ($x=1$) capacitance readings. After these measurement the device is fully calibrated: the sensitivity of the device, $dC/dx = C(1) - C(0)$ and a fixed point (e.g. $C(0)$) are known.

In the case of phase-boundary detection in dilution refrigerators, it is difficult to perform this simple calibration under operating conditions. We may not be willing to fill up the still, since it is difficult to remove the excess $^4$He later. Usually it is also not feasible to add enough $^3$He to the mixture so that the phase boundary lowers enough to cover the whole level gauge in the mixing chamber.
One can then resort to an indirect calibration using phases with a known dielectric constant and Eq. (1) (one can use $^4\text{He}$ and vacuum in the still or $^3\text{He}/^4\text{He}$ mixture and vacuum in the mixing chamber). But the dielectric constants are a function of temperature (through the density), which must be accurately measured during the calibration. The stray capacitance may not be constant over the course of the experiment; it may vary with the level of the helium bath or with the temperature of the level gauge itself. The electrodes may not be exactly parallel, invalidating Eq. (1). In the mixing chamber small calibration errors are exacerbated because the difference between the dielectric constants of the phases is five times less than in the still.

These kind of difficulties may be the reason that despite being extremely useful diagnostic devices, capacitive phase boundary detectors are not widely used, especially in the mixing chamber. To solve the calibration problem we have used a segmented capacitor as shown in Figure 1. It is constructed from two pieces of standard circuit board separated by $\approx 0.5$ mm. One of the electrodes is segmented horizontally, by etching away a number of 4 mm wide “fingers” leaving only a thin strip of copper at the edge for electric contact. In effect, we have a stack of small capacitors, connected in parallel. When a phase boundary moves up or downwards in the device, the capacitance will change in steps. A calibration of the device can now be done by moving the phase boundary only one or two segments upward or downwards. The sensitivity $dC/dx$ around the position where the phase boundary was varied follows immediately. The advantage of this scheme is that it is a direct calibration of the sensitivity, which does not involve knowledge of the dielectric constants. In the still a segmented capacitor is especially convenient. As soon as the first segment is covered during the condensation, as indicated by the first plateau in the capacitance reading, the level gauge is calibrated and ready for use.

For a mixing chamber phase boundary level gauge, the phase boundary between the concentrated $^3\text{He}$ phase and the diluted phase in the mixing chamber can be varied by adjusting the amount of $^3\text{He}$ in the DR. One can perform a so-called ‘one-shot’, achieved by temporarily closing off the returning $^3\text{He}$, while continuing to pump $^3\text{He}$ out of the still. In this situation the amount of $^3\text{He}$ in the DR will diminish, and the phase boundary in the mixing chamber will move upwards. On the other hand, condensing more $^3\text{He}$ will move the phase boundary downwards. For the calibration to succeed, it is necessary to move the phase boundary monotonically up or downwards, but this is not difficult to achieve.

III. RESULTS

We have performed an experiment with the level gauge depicted in Figure 1 in the mixing chamber of one of our plastic dilution refrigerators. In addition to the present device, there were four vibrating wire viscometers (horizontal stretched wires) at known positions in the region of the phase boundary. For the purposes of this experiment they served as additional (discrete) level indicators of the phase boundary, because the viscosity of pure $^3\text{He}$ is much larger than that of the diluted phase. Figure 2 shows the result of one particular ‘one-shot’. The capacitance steps are clearly resolved, allowing us to infer the calibration constant. Controlling the phase boundary over almost the whole length of the mixing chamber allowed us to observe almost all steps (see inset in Figure 2). A linear fit gives the sensitivity of our device ($0.0162$ pF/segment, $dC/dx = 0.287$ pF). The (expected) linearity shows that our electrodes were indeed parallel.

To know the absolute position of the phase boundary, it is still necessary to measure a capacitance value at one known position of the interface. The ‘empty’ value is a convenient point (only diluted phase in the mixing chamber; the phase boundary is above the level gauge).
In our experiment, we also used the large change of the resonance width of our horizontal viscometers when the phase boundary passed them. Both methods lead to an ‘empty’ value $C(0) = 30.061 \text{ pF}$. In the case of a still level gauge the most convenient point would be the empty value $C(0)$ (only vapour phase in the gauge).

After the calibration, we were able to monitor and control the phase boundary in the mixing chamber to within a fraction of a segment (a segment corresponded to 3.9 mm). This knowledge greatly helped us to operate the DR with different flow rates (from 100 to 1000 $\mu\text{mol/s}$) because the dead volume behind our large $^3\text{He}$ circulation pump was a considerable fraction of the total volume of the DR, leading to large variations of the phase boundary in the mixing chamber as a function of the flow rate.

The segmented capacitor gives us an opportunity to compare its direct calibration with the procedure which has to be adopted for a conventional non-segmented capacitor. If it is not feasible to measure ‘full’ and ‘empty’ values under operating conditions, one can calibrate the gauge with phases of known dielectric constants. We calculated the dielectric constants of the various helium phases using the Clausius–Mossotti relation with the molar volumes found in reference [3].

We measured the value of the level gauge in vacuum, $C_v = 28.832 \text{ pF}$, and the value with only diluted $^3\text{He}$ phase, $C(1) = 30.061 \text{ pF}$. The sensitivity for the $^3\text{He} - ^4\text{He}$ phase boundary is then

$$dC/dx = \frac{(C(1) - C_v) \epsilon_{34} - \epsilon_3}{\epsilon_{34} - 1} = \frac{(C(1) - C_v) \cdot 0.238}{298 \text{ pF}} = 0.293 \text{ pF}$$

where $\epsilon_{34} = 1.0562$ and $\epsilon_3 = 1.0428$ for $T \leq 100 \text{ mK}$. This result must be compared to a directly measured value of 0.287 pF. Thus it turns out that this indirect calibration, is actually quite good in the case of our level gauge. We note that for a calibration of a conventional level gauge in the still there are no alternatives, short of filling the whole length of the capacitor.

The level gauge can also be calibrated by pure $^4\text{He}$ at a known temperature (density). In practice, this is not as easy as it sounds, because at the beginning of a DR experiment we may be unwilling to fill the DR with (difficult to remove) pure $^4\text{He}$. Nevertheless, it is interesting to see how accurate such a calibration is. Note that the dielectric constant of $^4\text{He}$ depends strongly on temperature (through the density) above $T \approx 1.5 \text{ K}$. We used $^4\text{He}$ at $T = 1.17 \text{ K}$ (measured with a calibrated Ge thermometer, $\epsilon_4 = 1.0573$), and vacuum as the calibration phases. The result was $C(0) = 28.832 \text{ pF}$ and $C(1) = 30.106 \text{ pF}$. The scaled sensitivity for the phase boundary in the mixing chamber is $(C(1) - C(0)) \cdot 0.234 = 0.298 \text{ pF}$, which must be compared to the 0.287 pF measured directly.

Our conclusion is that a conventional non-segmented capacitor can be used as a level-gauge, if careful measurements of the ‘empty’ (vacuum) and ‘full’ (with a phase of known dielectric constant) capacitance values are made. The use of a segmented capacitive phase boundary level gauge is more convenient. It makes a direct calibration of $dC/dx$ in units of pF/segment possible under operating conditions. This calibration does not involve knowledge of the dielectric constants, and can be done by moving the phase boundary only one or two segments. In addition, the level gauge can be checked for non-linearities.

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