THE POMERANCHUK EFFECT

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by

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INTRODUCTION

A central part of the story of the discovery of superfluid ^{3}He is the cooling technique used for the experiments, the Pomeranchuk Effect. Although it is not an especially useful technique for obtaining low temperatures today, it contains my favorite example of the use of the Clausius-Clapeyron equation. The cooling technique is fun to describe in undergraduate physics classes on thermodynamics.

In 1950, I. Pomeranchuk, a well known particle theorist, suggested that melting ^{3}He could be cooled by squeezing it[1]. At the time of his suggestion ^{3}He was quite rare and had not yet even been liquefied. He observed that at low enough temperatures the thermal phenomena in condensed ^{3}He would be dominated by spin properties instead of phonon properties. The liquid of ^{3}He would obey Fermi statistics with an entropy proportional to the temperature, much like the free electrons in a good metal. On the other hand, the entropy of solid ^{3}He would be that of the disordered collection of weakly interacting spin 1/2 nuclei. At temperatures greater than the [then] expected nuclear magnetic ordering temperatures less than 1 μK, the entropy per mole of solid ^{3}He would be $S = R \log_e 2$, independent of temperature until the high temperature phonon modes of the solid become important. (The Debye temperature of solid ^{3}He is approximately 30 K.)

The idea of the method is represented in Figure 1. The entropy of solid ^{3}He exceeds that of liquid ^{3}He at temperatures less than 0.3 K. If the mixture is compressed without heat input it will cool as liquid is converted into solid.

DISCUSSION OF POMERANCHUK'S PROPOSAL

Fifteen years passed before anyone took up the suggested cooling technique[2]. There were several reasons. The most important was the availability of ^{3}He. It comes from tritium decay. Tritium was being produced for the most deadly part of the weapons industry. By 1965 copious quantities had been made. Low temperature physicists took advantage of the waste product of the arms race, the ^{3}He extracted from the gases prepared for hydrogen bombs.

The second reason for the late date of attempts at the method was the skep-
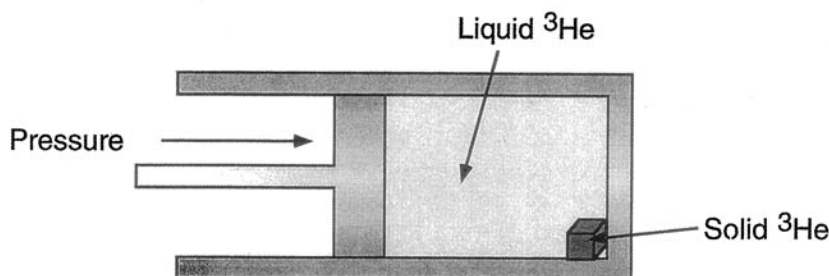
$$\frac{\Delta V}{V} = \frac{1.3}{24} = 5\% \quad \text{(Fraction volume change for complete conversion of liquid to solid)}$$

Figure 1. Pomeranchuk's suggestion for cooling a melting mixture of ^{3}He. The solid phase has a higher entropy than the liquid at low temperatures. As the liquid-solid mixture is compressed, heat is removed from the liquid phase as solid crystallites form. The fractional change of volume required to completely convert liquid into solid is approximately 5%. Unlike melting water, the solid phase forms at the *hottest* part of the container.

ticism of experimentalists about practical considerations. The entropies of the liquid and solid phases are illustrated in Figure 2. Liquid ^{3}He is rather accurately described by Landau's Fermi Liquid Theory[3,4]. At low temperatures[5,6] the entropy per mole of the liquid at melting pressure is approximately given by $S \approx 3RT$. The entropies of the liquid and solid phases are equal at 0.32 K. At lower temperatures Pomeranchuk's suggestion for cooling will work.
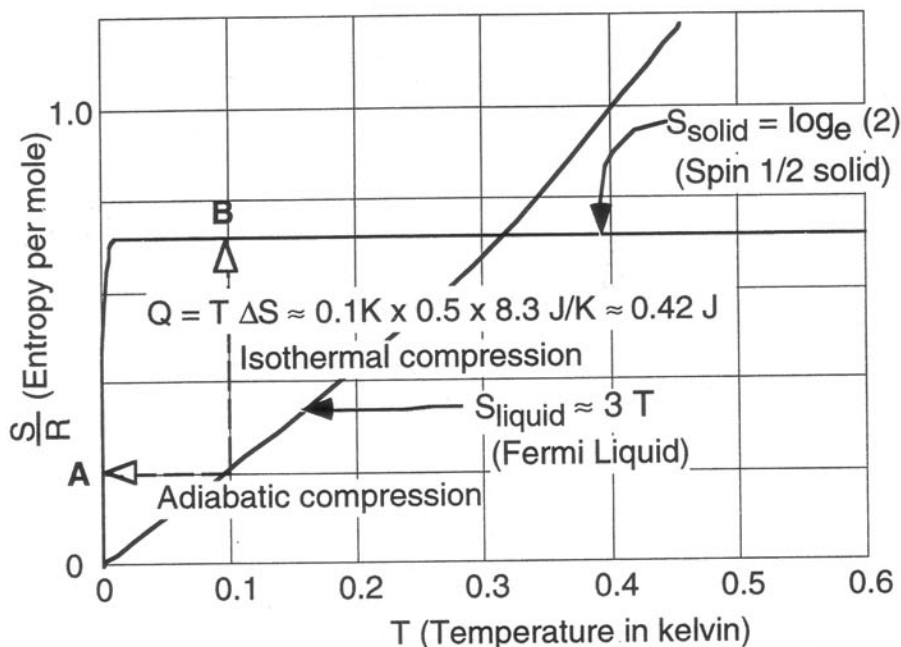

Figure 2. The entropies of liquid and solid ^{3}He. At T < 0.32 K, liquid ^{3}He has a lower entropy than the solid phase. The figure shows an example at T = 0.1 K. The latent heat associated with converting 1 mole of ^{3}He liquid into solid is 0.42 Joules, a substantial amount of heat removal at these low temperatures.

The adiabatic cooling path is indicated with the arrow **A** marked on the vertical axis. In the example, liquid compressed at an initial temperature of 0.1 K, with an entropy of 0.2R, will form a liquid-solid mixture which eventually cools to very low temperatures. The maximum amount of heat which can be removed is the latent heat of conversion of the liquid to solid and is indicated by the isothermal path, labelled **B**. The latent heat per mole at T = 0.1 K is 0.42 Joules.
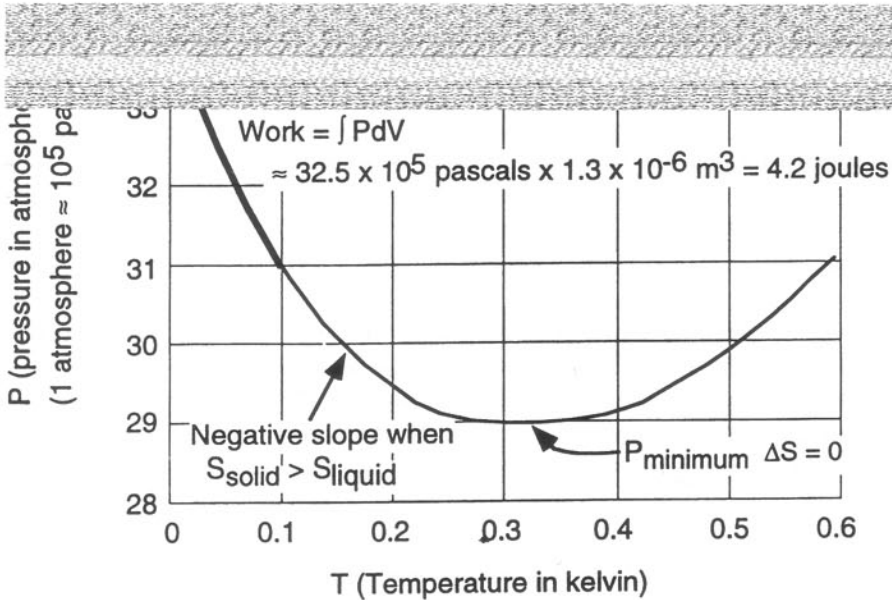

Figure 3. The melting pressure of ^{3}He. The figure can be constructed from Figure 2 through the Clausius-Clapeyron equation [see text]. The molar volume of liquid ^{3}He exceeds that of the solid by 1.3 cm^3 per mole. Thus, the slope of the melting curve is negative at temperatures less than 0.32 K. The work of compression in forming the solid is approximately 4.2 Joules, an order of magnitude larger than the heat which might be extracted.

The cooling effectiveness of the method must be compared with the possible heat losses in the process. At this point the natural skepticism of experimentalists arises. The amount of work involved during the compression is large. The melting pressure versus temperature of ^{3}He is illustrated in Figure 3. The melting curve may be calculated from the liquid and solid entropies using the Clausius-Clapeyron equation,

$$\left.\frac{dP}{dT}\right)_{melting} = \frac{S_{liquid} - S_{solid}}{V_{liquid} - V_{solid}} , \qquad [1]$$

where, $S(T)_{liquid}$ and $S(T)_{solid}$ are the molar entropies at melting. V_{liquid} and V_{solid} are the molar volumes of the two phases at melting. The difference, $V_{liquid} - V_{solid}$ is nearly independent of temperature and has the value 1.3 cm^3 per mole. We will return to Equation [1] later when I describe our experiments designed to measure the entropy of solid ^{3}He.

The work performed in converting the liquid to solid, starting at 0.1 K can be obtained from the integral, $\int PdV$ along the melting curve. It's value is approximately 4.2 Joules. An order of magnitude more work must be done than will be extracted during the cooling process. The ratio, W/Q, of work to heat extracted is near a minimum at the temperature illustrated. When the process is performed at lower starting temperatures, as was the usual practice, W/Q becomes larger than 100. The challenge of the experimental design, thus apparently became the avoidance of frictional heat losses during the compression process.

COMPRESSIONAL COOLING IN PRACTICE

In the Spring of 1966, David Lee invited me to join him at Cornell to begin experiments on the cooling of solid ^{3}He using the compressional cooling technique. The goal was to reach the temperature of the nuclear magnetic ordering transition in solid ^{3}He. My Ph. D. thesis at Duke University with Horst Meyer[7] had been concerned with NMR measurements of the size of the exchange interaction in solid ^{3}He. We knew from these measurements that the magnetic phase transition in solid ^{3}He at the melting pressure should occur at temperatures closer to 1 mK than 1 µK. Despite my certainty that Pomeranchuk's technique for cooling was probably doomed to failure, I was anxious to join Dave in searching for the transition. As a back-up we would attempt to cool solid ^{3}He with magnetic cooling schemes.[2] The latter had not yet been used successfully to obtain such low temperatures in liquid or solid helium but we began a parallel effort to use nuclear demagnetization[8].

By the time I arrived at Cornell in October 1966, the method had been successfully demonstrated by Anufriev in Moscow.[9] A cross section of Anufriev's apparatus is represented in Figure 4. The volume available for liquid ^{3}He is exaggerated in the figure. The ^{3}He space contained 30 cm^2 of metal foil to
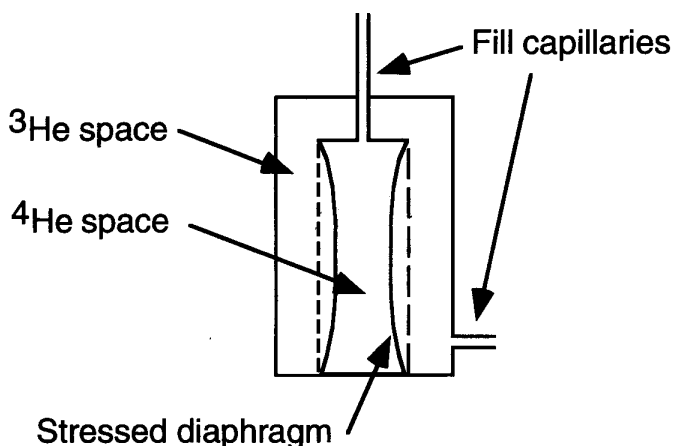

Figure 4. Cross-sectional representation of the compressional cooling cell used by Anufriev. Both inner and outer chambers have a rectangular shape. Thin stainless diaphragms of the inner cell were displaced inward with the application of ^{3}He at the melting pressure. The walls of the stressed diaphragm were then forced outward by filling the inner chamber with pressurized ^{4}He.

act as heat exchangers to cool the outside wall of the chamber. The outer chamber was first filled with liquid ^{3}He at the melting pressure at temperatures greater than 0.32 K.. As the cell was cooled using the demagnetization of a paramagnetic salt, a block formed in the fill capillary trapping a fixed quantity of ^{3}He in the cell. At low temperatures carefully cooled ^{4}He was admitted within the inner chamber to act as a hydraulic fluid. Pressurized liquid ^{4}He forced the diaphragm walls outward to decrease the volume available for the melting ^{3}He. The cell cooled to temperatures less than 15 mK, the minimum temperature which could be measured with the thermometers in contact with the exterior of the cell.

It is interesting to notice the use of liquid ^{4}He as the hydraulic fluid for forcing changes in the ^{3}He cell dimensions. At temperatures less than 0.3 K, the normal fluid fraction of ^{4}He liquid is very small. The heat capacity and thermal conductivity of liquid ^{4}He are negligible. Anufriev's pioneering experiment tested the method, showed that it would work, and demonstrated the use of liquid ^{4}He as a hydraulic fluid at low temperatures. The fears of excessive frictional heating associated with the movement of a metal diaphragm were unfounded. We now know that practically all metals have a very high quality factor at temperatures less than 4 K. In the subsequent years, *every* apparatus built upon the principle of forcing a metal diaphragm or bellows to move produced successful cooling. There is an important caveat in using liquid ^{4}He. The melting pressure of ^{4}He is 4 atmospheres less than that of ^{3}He. Spring tension or pressure amplifiers must be used in the experimental design so that ^{4}He does not solidify during the compression process.

POMERANCHUK CELLS AT CORNELL

The Cooling Cell of Jim Sites

Our first venture with Pomeranchuk cooling came with the thesis project of Jim Sites. The goal of his experiment was to measure the magnetic susceptibility of melting solid ^{3}He at temperatures near or below the nuclear magnetic phase transition. We had long discussions about the design of an apparatus. I have often regretted that we did not follow one of Dave Lee's original suggestions, the use of a weight to compress a bellows filled with melting ^{3}He. A heavy mass would be suspended by a wire and slowly lowered on the bellows in a controlled manner. The mass would have to be a metal of high density with negligible magnetic properties. Gold seemed to be the only really suitable metal. Imagine the profit if we had purchased 5 kilograms of gold at the 1967 price, $30 per troy ounce!

Before we completed our first experiments at Cornell the Wheatley group in La Jolla reported highly successful compressional cooling of ^{3}He down to temperatures less than 2 mK. [10, 11] Their design employed a tube of ^{3}He with an ellipsoidal cross section. The tube was surrounded by pressurized liquid ^{4}He. The entire cooling assembly was placed within the mixing chamber of a dilution refrigerator[2] and pre-cooled, over a matter of days, to 24 mK.

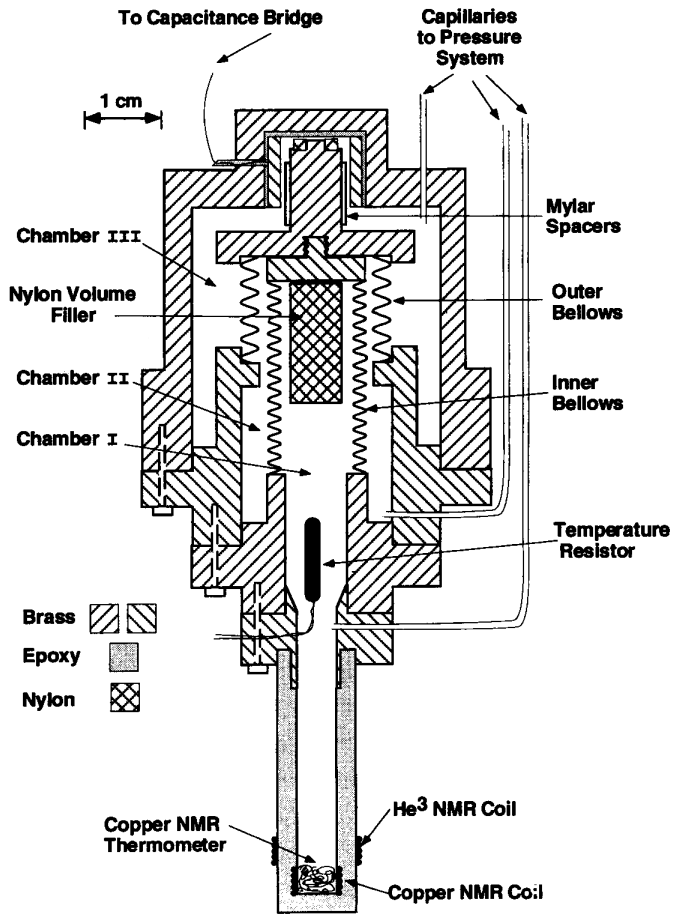The design first used at Cornell was one suggested by our colleague John

Figure 5. The Pomeranchuk cooling cell of Jim Sites.

Reppy. It is illustrated in Figure 5. A simplified schematic of the apparatus is shown in Figure 6. The cell contained two concentric beryllium-copper bellows and three helium chambers.[12, 13] The ^{3}He sample was contained in the innermost chamber, Chamber I, and ^{4}He in the outer two chambers. Initially all three chambers were pressurized to approximate the melting curves of their contents, and the sample cell was precooled with a dilution refrigerator to 25 mK. Compression of the ^{3}He was achieved by releasing the pressure on the ^{4}He in Chamber II. We had thought that there might be some advantage in removing fluid from the cell. The position of the bellows assembly was monitored by measuring the change in capacitance of a short rod attached to the 'Top Plate' of the assembly. The minimum temperature recorded by the copper NMR thermometer was 7 mK. Extrapolating from the size of the magnetic susceptibility of the solid ^{3}He, the average temperature of the solid was sometimes as low as 2 mK. Like the Wheatley group[10], our attention was focused solely on the solid ^{3}He. The liquid component was viewed merely as the cooling agent!

Our first Pomeranchuk cell had several significant disadvantages. The most important was that it tended to warm up quite rapidly after only several hours and achieved far less volume change than the maximum 5% required for conversion of all of the liquid into solid. Apparently, solid helium was being trapped within the convolutions of the bellows to become crushed as the bellows contracted. With regard to our desire to measure the temperature dependence of the magnetic susceptibility of the solid the cell had two further design flaws. The place where solid grew in the cell was unpredictable and the time constant for the copper NMR thermometer was very long. Fortunately, some of the solid ^{3}He nucleated on the "rat's nest" of copper wires at the bottom of the NMR tail section. The thermal equilibrium time for nuclear magnetization in metals, T_1, is inversely proportional to temperature. In copper the product $T_1 \circ T$ is approximately 1 sec-K. With only 10 minutes available at the bottom temperatures near 2 mK, the copper thermometer never caught up with the temperature changes.

A final conceptual mistake in our first compressional cooling cell is that there was no provision for the direct measurement of the ^{3}He pressure. We could monitor the volume change by keeping track of the bellows displacement. But the valuable thermodynamic information available with a knowledge of the melting pressure was not available in this set of experiments.
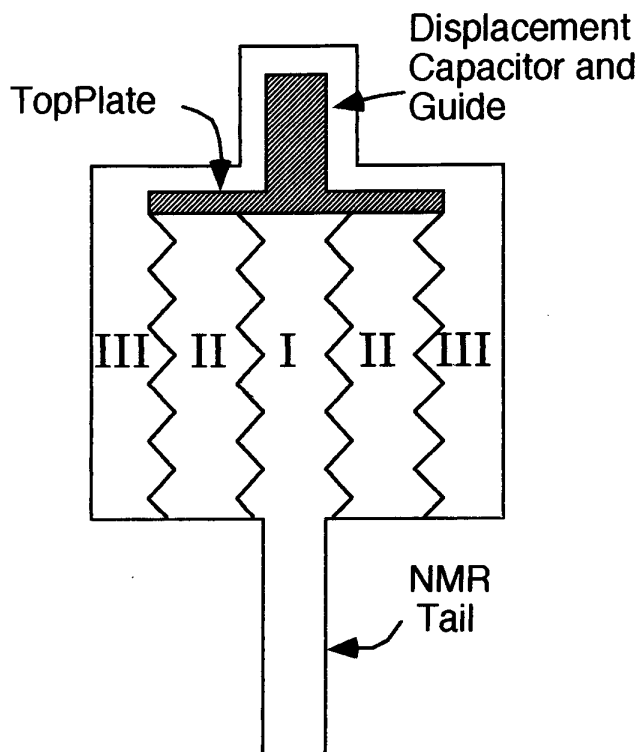

Figure 6. Schematic representation of the cell of Jim Sites.

THE CORRUCCINI-OSHEROFF COOLING CELL

Our next cooling attempts were made with a cell which was designed to investigate some unusual spin diffusion phenomena predicted by Leggett and Rice.[14] The cell is illustrated in Figure 7. We used the cold liquid ^{3}He in a Pomeranchuk cell to cool a separate chamber of either liquid ^{3}He or dilute liquid mixtures of ^{3}He in ^{4}He.[15,16] Doug worked on the development of the cooling technique while Linton Corruccini worked on the design of the chamber for NMR measurements. Following our experience with Jim Site's cell, Osheroff decided to try compressing the ^{3}He with a bellows which expands. The idea was to avoid crushing solid ^{3}He within contracting bellows. The ^{3}He compression cell was filled with liquid at the melting pressure and ^{4}He pressure was applied to the upper chamber. The differences in the melting pressures of the two isotopes were taken into account by having the diameter of the ^{4}He bellows larger than that for the ^{3}He by a ratio of 3.5:1. The ^{3}He could be completely solidified without raising the ^{4}He pressure above 10 bar.
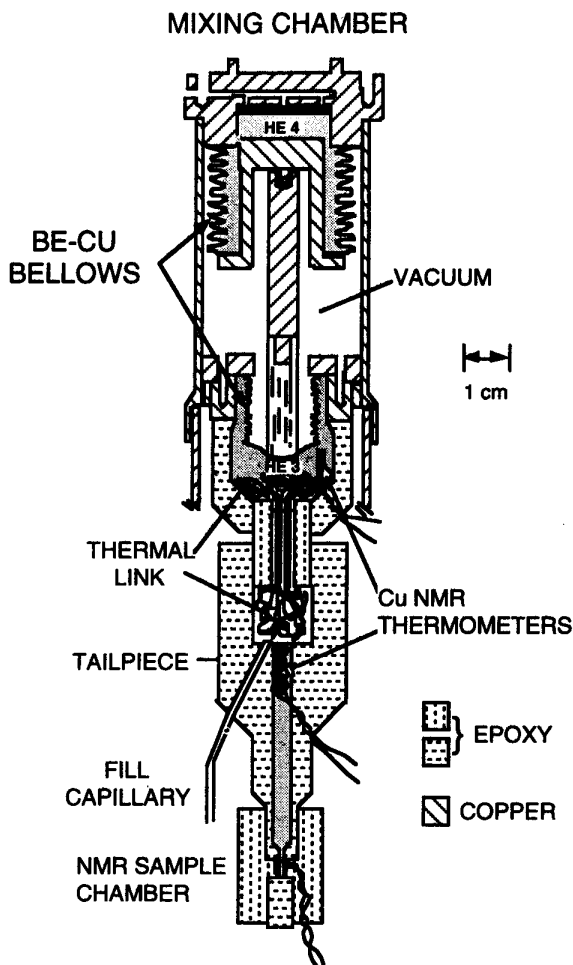

Figure 7. The Corruccini-Osheroff cooling cell.

A bundle of small copper wires made a thermal connection between the ^{3}He compression region and the NMR sample region. The temperatures of the compression region and sample region were measured with copper NMR signals.

The apparatus worked very well indeed, as did the Leggett-Rice theory. It was an easy experiment. Before compression, the cell was first cooled to 25 mK with a dilution refrigerator. We succeeded in cooling the sample region to temperatures as cold as 4 mK. We typically spent 8 to 12 hours in compressing the liquid ^{3}He in order to maintain thermal equilibrium and maximize the cooling efficiency between the cooling cell and sample cell. The chamber would remain at the lowest temperature for up to 4 hours, then slowly warm up to 10 mK over a period of 5 hours. Further warming was achieved by partial decompression of the cooling cell. The minimum temperature recorded by the copper NMR thermometer in the cooling cell was 3 mK. The cell was a precursor for the one used in the discovery of superfluid ^{3}He. Since it was not intended for studies of the ^{3}He there was minimal instrumentation in the compression cell.

OSHEROFF'S COMPRESSION CELL

The compressional cooling cell used by Osheroff[17] during the course of the experiments on melting ^{3}He was a variation of the one we used in Corruccini's spin diffusion measurements. The epoxy bottom of the cell could be readily replaced and in the course of six months no less than five different epoxy tail sections were used. The cell illustrated in Figure 8 is the version published with the results purporting to measure the phase transition in solid ^{3}He. It had two very important design changes from the previous cells. The first was that we changed the metal NMR thermometer from copper to platinum. The thermal equilibrium time for ^{195}Pt nuclei is a factor of 30 shorter than that of copper. It is far less susceptible to small eddy current heating effects than copper. It eventually read temperatures well under 2 mK.

The second change was probably more important. It was the inclusion of a gauge to measure the pressure of the melting ^{3}He. A thin metal diaphragm on the bottom of the cell deflected as the pressure changed. The amount of the deflection was measured capacitively. One plate was attached to the center of the diaphragm while the other was fixed to a mounting arrangement on the epoxy tail section. The design is one which had been invented by Straty and Adams[18] and became widely used at Cornell and elsewhere. The melting pressure is a unique function of temperature. At higher temperatures the vapor pressure of ^{3}He and ^{4}He gases in equilibrium with liquid helium are routinely used to calibrate other thermometers. Adams had previously suggested that the ^{3}He melting pressure be used as a temperature standard.[19] During the interval in late November 1971 in which Doug Osheroff was 'practicing' the use of the apparatus the pressure measurements gave information about changes in temperature of the apparatus.

Dave Lee and Doug Osheroff have described many of the details about

those early measurements in their Nobel Prize lectures. The well known 'pressure versus time' curve is reproduced in Figure 9. The experiment was conducted with a constant rate of compression of the ^{3}He bellows, that is with a constant cooling rate. The pressure scale can be interpreted as a measurement of temperature change. The temperature scale on the right was our best guess at the thermodynamic temperature and was based upon measurements of the ^{195}Pt magnetic susceptibility. The pressure measurements are relative to the maximum melting pressure of ^{3}He.

The points labelled A and A' are transitions of liquid ^{3}He from the normal liquid phase to the superfluid A phase and then back again. The cooling or warming rate changes at these points because of the change in heat capacity of the liquid ^{3}He. dT = (1/C)•dQ, where T is the temperature, C is the heat capacity, and Q is the heat input. For a constant rate of heating dQ/dt, the rate of temperature change becomes (dT/dt) = (1/C)•(dQ/dt). A sudden in-
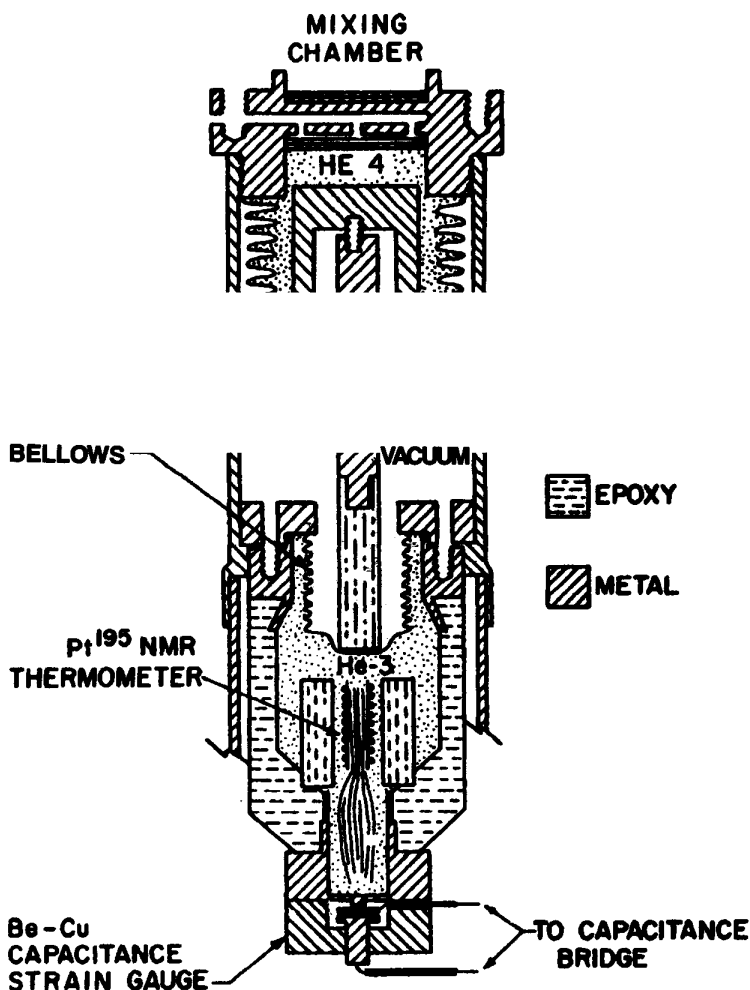

Figure 8. Doug Osheroff's Pomeranchuk Cell.

crease in heat capacity will cause the rate of cooling at A to decrease. At the time, we mistakenly identified the heat capacity change with the long sought nuclear magnetic transition in the solid phase. Points B and B′ are related to another thermal event. At point B there must be an evolution of latent heat because there is a sudden but small decrease in the temperature in the cell. The pressure at which the B type event took place varied, generally depending upon the cooling rate. We attributed this, correctly, to a super cooling. Point B′ is the equilibrium transition, we now know, from the superfluid B phase to the superfluid A phase. The temperature change pauses briefly as the B phase absorbs extra heat to pass through a first order phase change (like the melting of ice, or the freezing of liquid ^{3}He). Points C and D in Figure 9 correspond to the maximum melting pressure achieved and the time at which a slow decompression was begun.
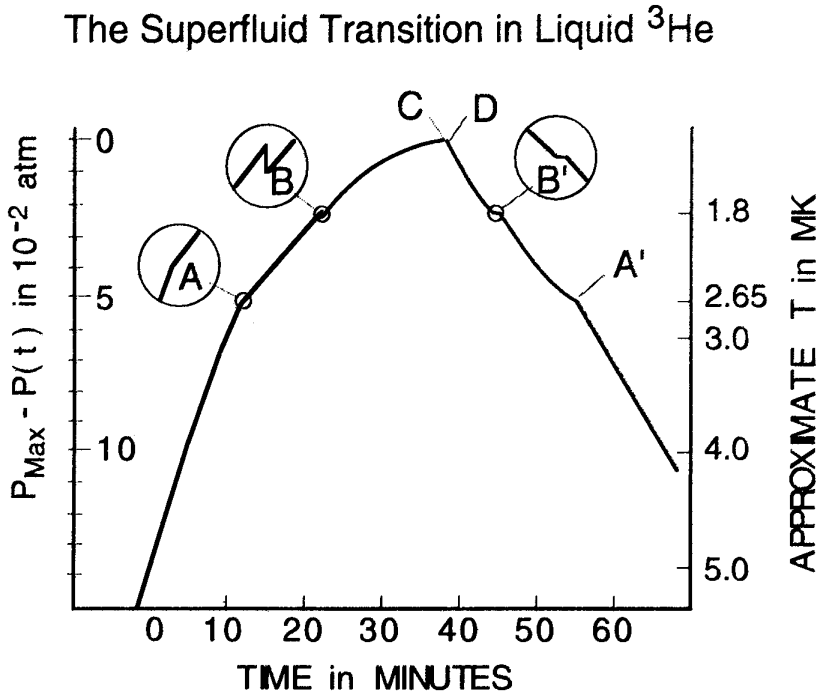
The Superfluid Transition in Liquid ^{3}He

Figure 9. Measurement of the change in ^{3}He melting pressure with time with a constant cooling rate in an Osheroff's Pomeranchuk cell.

This measurement in Figure 9 contained an embarrassing amount of contradictory detail. Since nothing was previously known about the nuclear ordering process we supposed that points B and B′ marked the transition to a second magnetic phase. Still, the total pressure change between point A and the maximum melting pressure was surprisingly large. In SI units the pressure difference is 0.00527 MPa. In this connection we made the following observation, "In order to obtain sufficient pressure change from 2.7 mK to 0 mK through integration of the Clausius-Clapeyron equation,

$$\Delta P = \int \frac{(S_{solid} - S_{liquid})dT}{(V_{solid} - V_{liquid})} \approx \int \frac{S_{solid}}{\Delta V} dT, \qquad [2]$$

to agree with the value presented above, one is forced to hold the solid entropy nearly constant over a broad temperature region below the 2.7 mK transition temperature. This possible behavior of the solid entropy is, in fact, also suggested by the nearly constant slope of P(t) between A and B in Figure 9. We know of no physical system which furnishes a precedent for the entropy behavior we postulate here."[20] Using our 'approximate' temperature scale would require the solid entropy to remain at the value R•log$_e$2 over the temperature interval between 2.7 mK and 1.5 mK!

Our misgivings about the interpretation of the data were well founded. In our subsequent paper about the NMR properties of the ^{3}He in the compression cell we finally got it right.[21] The change in heat capacity signaled at point A corresponded with a change in the properties of liquid ^{3}He and point B (or B') marked the phase boundary to a liquid phase with even different behavior.

Less than a year after the report of the A and B transitions in ^{3}He, the group in Helsinki[22] used Pomeranchuk cooling to study the pressurized liquid phase in the cell to show that the viscosity of liquid ^{3}He decreases by a factor of 1000 in the new phases. The viscosity measurements were made with a vibrating wire immersed within the liquid.

FINALLY, THE REAL PHASE TRANSITION IN SOLID ^{3}HE

After Anufriev, Jim Sites, and the Wheatley group had shown us that the compressional cooling method was an effective way to cool melting helium, we decided to begin a second set of measurements using a cell designed primarily for the optimization of studies of solid ^{3}He.[23] The compression cell designed by Bill Halperin is shown in Figure 10. The design thought was that by having a lens-shaped compression region there would be a minimum of heating related to crushing solid ^{3}He during compression. The cell contained both a pressure gauge for the ^{3}He and a method for measuring the absolute volume changes, a second set of capacitor plates attached to the moving diaphragm. We also had a provision for measuring the changes in the magnetization of the ^{3}He. The basic operation of the cell was the same as the others I have described. ^{3}He at the melting pressure was trapped in the lower region while the upper was filled with liquid ^{4}He. The assembly was precooled to 25 mK and compression was achieved by forcing liquid ^{4}He in the upper region.

In the sequence of measurements with this cell, we measured the entropy of solid ^{3}He down to temperatures below the phase transition. In addition we were able to measure the heat capacity of liquid ^{3}He through the superfluid transition, the latent heat of the transition between the A and B phases, and to determine a 'first principles' temperature scale.[23–25] Through
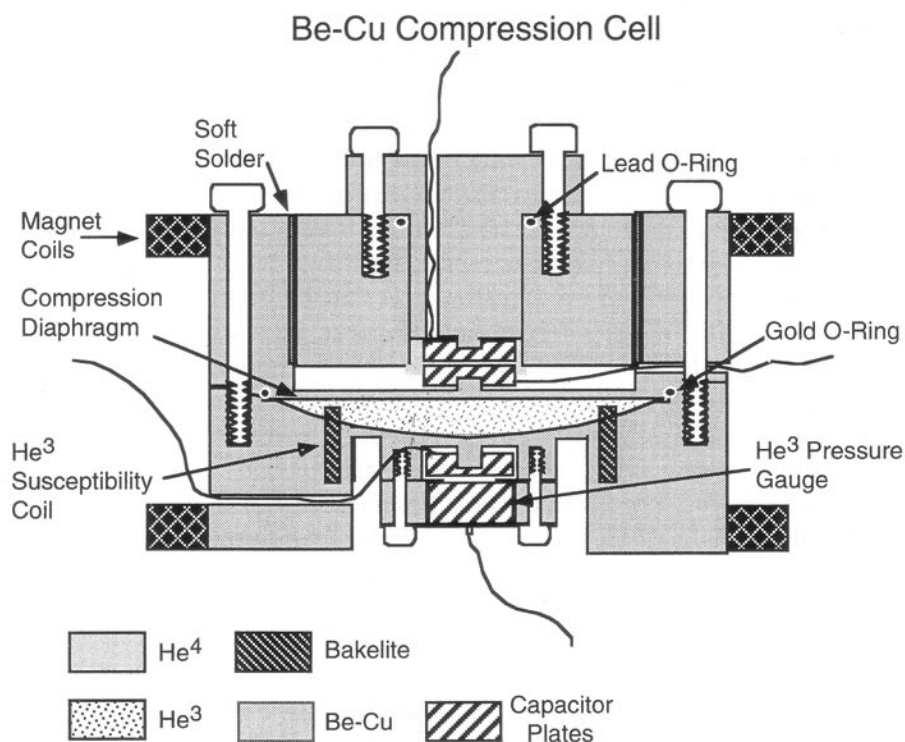
Be-Cu Compression Cell

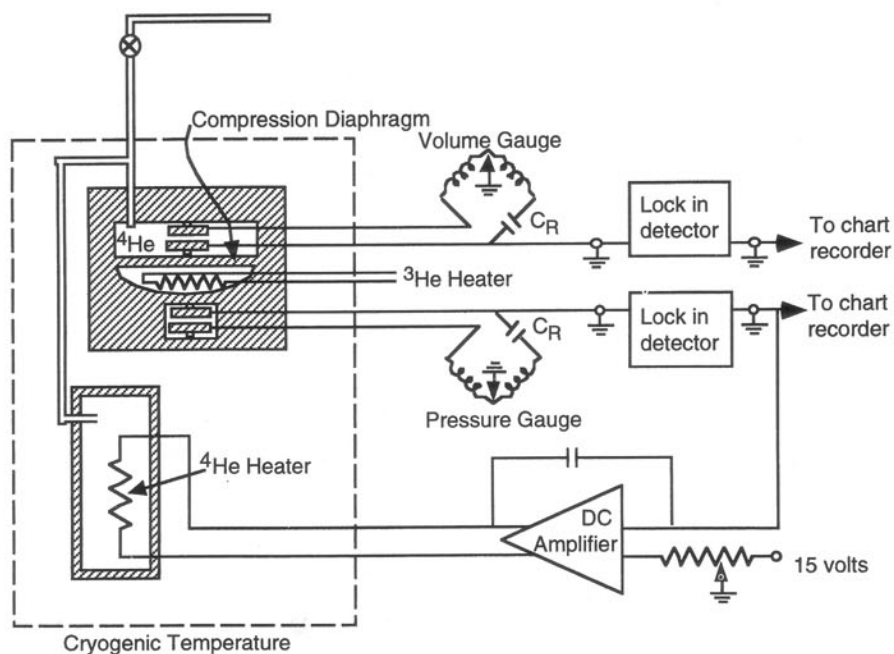Figure 10. The Pomeranchuk Cell of Bill Halperin.

Figure 11. Servo Loop for Controlling Compression Rate.

experience, we grew to understand the different time constants for thermal equilibrium of the liquid and solid phases. A separate container for liquid ^{4}He was located in the cryostat. Heat applied to the ^{4}He in that vessel would rapidly change the ^{3}He volume in the compression cell.

Heat pulses and 'cool pulses' could be applied by means of short bursts of decompression or compression of the diaphragm. Heating was also accomplished by passing a calibrated current through a heater wire in the ^{3}He compression cell. The melting pressure (and hence temperature) could be maintained at a constant value by passing the error signal from the pressure gauge back through the DC amplifier to the ^{4}He heater. An example of such a measurement is shown in Figure 12. A signal from the pressure bridge has been sent to the ^{4}He heater to maintain a constant ^{3}He pressure. At t = 0 a short heat pulse of 32.26 ergs was applied to the ^{3}He heater. In response, the servo control system briefly accelerated the rate of ^{3}He compression. The volume change associated with the heat pulse was 2.56×10^{-4} cm^3 as additional liquid was suddenly converted into solid ^{3}He. The output of the pressure bridge is also shown, the pressure has been converted to temperature units. During the measurement the maximum temperature excursion of the cell was less than 5 µK.
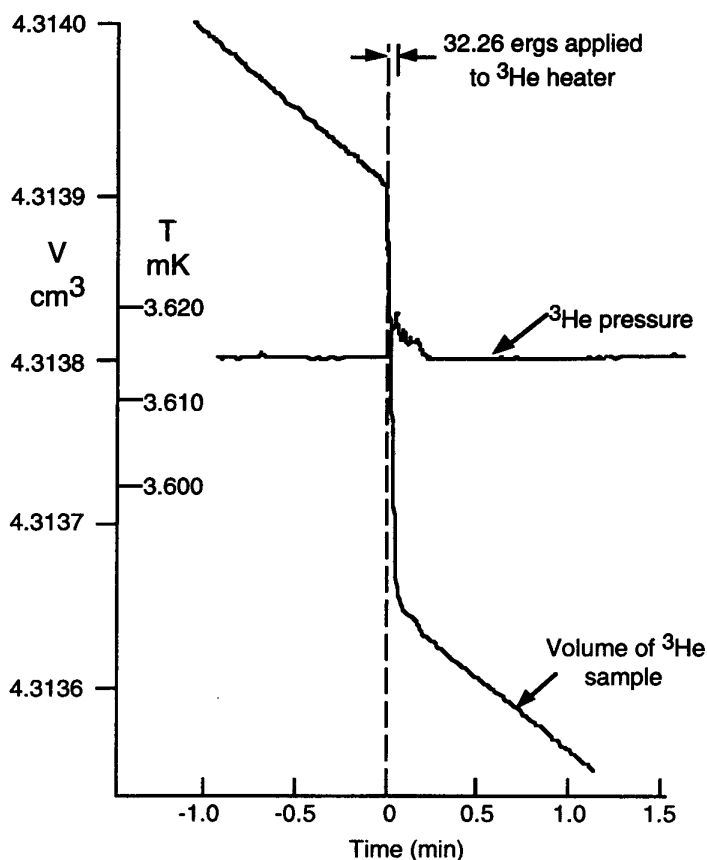

Figure 12. Measurement of volume change following a heat pulse with the compression cell constrained at constant pressure.

The Clausius-Clapeyron equation can be invoked, once again, with the data obtained in Figure 12. If we multiply both sides of [1] by the temperature, T, we obtain, $T \cdot \dfrac{dP}{dT}\Big)_{melting} = \dfrac{T \cdot \Delta S}{\Delta V} = \dfrac{\Delta Q}{\Delta V}$. The quantity on the right is the ratio of the heat input to the volume change. The measurement was repeated from temperatures near 25 mK down to the temperature of the maximum melting pressure to generate a table of values of P versus T(dP/dT).

With regard to the elusive phase transition of solid ^{3}He, measurements like that illustrated in Figure 12 and other non-equilibrium measurements with pulsed volume changes were used to generate the data shown in Figure 13.[24] At a pressure near the maximum melting pressure, the value of T(dP/dT) decreases rapidly, corresponding to an entropy decrease of more than half of the spin entropy. The experiment was the first quantitative identification of the point of magnetic order.

The entropy versus temperature curve shown in the inset curve in Figure 13 was obtained through integration of the P versus T(dP/dT) data. A relative temperature scale is given by $\dfrac{T}{T_{solid}} = \exp \displaystyle\int_{P_{solid}}^{P} \left[T \dfrac{dP}{dT} \right]^{-1} dP$. A single fixed point in temperature is sufficient to generate the complete low temperature melting curve from the data. The fixed point we used was that of the
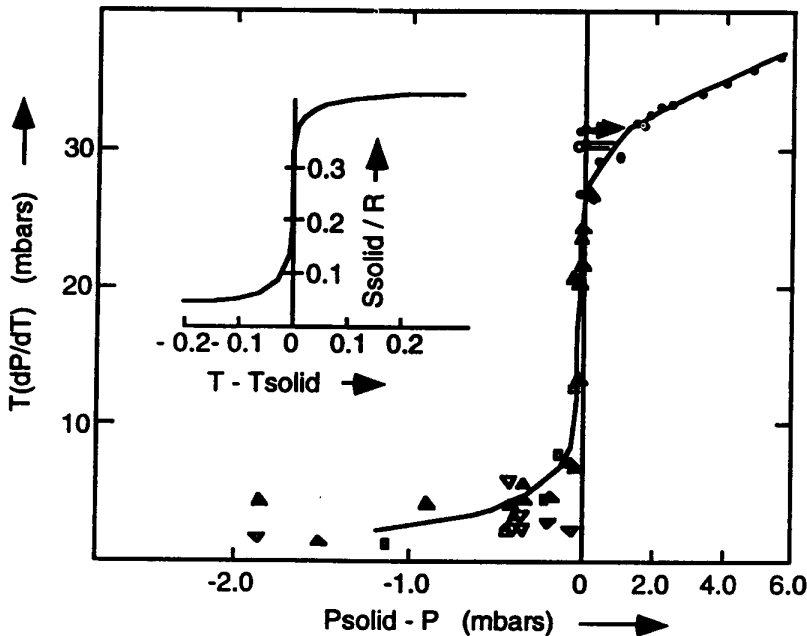

Figure 13. The entropy decrease at the nuclear magnetic phase transition of solid ^{3}He. The entropy versus temperature data were calculated from the measurements of T(dT/dP) versus pressure.

high temperature solid entropy, $S_{solid} \rightarrow R \cdot \log_e 2$. [The Debye temperature for solid helium is of order 30 K so that the phonon contribution is negligible at 30 mK.].

THE POMERANCHUK EFFECT AND MELTING CURVE IN 1996

The Pomeranchuk effect is no longer the preferred cooling technique for studying either liquid or solid ^{3}He. It is too restrictive because it permits measurements of ^{3}He only at the melting pressure. Moreover, an even more important reason is that the method of nuclear demagnetization is far more efficient and permits cooling of ^{3}He to temperatures in the range of 10 µK.[26]

The ^{3}He melting plays the same role in thermometry as it did in the days of our discovery of the superfluid transitions. There are four easily measured and reproduced fixed points on the melting curve: the minimum in the melting pressure; the superfluid A transition; the superfluid B transition; and the magnetic ordering transition of solid ^{3}He. Melting curve thermometers[27] have become the temperature standard for very low temperature work. In the days since Halperin's integration of T(dP/dT) along the melting curve there have been many independent measurements of the melting curve fixed points.[6,28] The most recent[28] give: $T_{minimum}$ = 0.31517 mK; T_A = 2.41 mK; T_B = 1.87 mK; and T_{solid} = 0.88 mK. The associated pressures are also know with great precision so that the strain gauge can also be calibrated with the fixed points. It is amusing to realize that many modern low temperature physicists routinely reproduce the data of Figure 9 as a standard temperature calibration of their apparatus.

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