**Journal of Low Temperature Physics manuscript No.**
(will be inserted by the editor)

# Adiabatic melting experiment on helium mixtures: status and prospects

**A. Sebedash**[1,2] · **S. Boldarev**[2] · **T. Riekki**[1] · **J. Tuoriniemi**[1]

Received: date / Accepted: date

**Abstract** We describe the improvements made to our earlier experiment, aiming to cool saturated helium mixtures at the melting pressure to ultra-low temperatures in the microkelvin regime. Cooling is produced by dissolving pure $^3$He in the superfluid state to pure $^4$He being released from the solid phase within the mixture of isotopes at the melting pressure. The limiting factor for the performance was considered to be the inevitable coupling of the liquid mixture with the surroundings at higher temperatures, such as through the filling line and the sintered surfaces needed for the precooling phase. These issues could be largely eliminated by the new design of the experiment. Results of testing the new components at low temperatures are presented and discussed.

**Keywords** Helium mixtures · Superfluidity · Dilution cooling · Solid helium

## 1 Introduction

Diluting pure liquid $^3$He into $^4$He is an efficient method of producing low temperatures in the millikelvin regime, and it is routinely used in dilution refrigerators widespread and commercially available [1]. The lowest temperature achieved in a continuously operating dilution refrigerator is about 2 mK [2], and the practical limit to this is set by the difficulty of transferring heat from the incoming warmer stream of $^3$He into the cooled opposite flow within the mixture phase. Also, the high viscosity of the Fermi fluids poses a limit to the rate at which $^3$He can be circulated in such devices. A small momentary enhancement of performance can be achieved by operating the dilution fridge in the single-cycle mode.

1: Department of Applied Physics, Aalto University,
P.O. Box 15100, FI-00076 Aalto, Finland

2: P. L. Kapitza Institute for Physical Problems RAS, ul. Kosygina 2,
Moscow 119334, Russia

Recently, a new mode of operation utilizing the dilution process for cooling helium mixtures to very low temperatures was proposed [3, 4, 5] and the operating principle of this was also demonstrated to work in practice [6]. Here an experimental chamber is filled with helium mixture containing $^3$He beyond the saturation limit ($> 8.1\%$ [7]) and pressurized so as to produce solid helium in the cell at about 25.7 bar [8], while keeping it at millikelvin range of temperatures by means of an ordinary dilution refrigerator. Under such circumstances, the produced solid phase contains $^4$He only, whereas $^3$He is expelled from the solidifying mixture into the pure liquid phase [9, 10]. Growing or melting the solid phase by adding or removing $^4$He in the cell can be accomplished through a so-called superleak line [3], which has to be led from the low temperature cell all the way up to the temperature of about 1.5 K, or else the line would be blocked by the solid pure $^4$He.

The benefit of this is that the isotopes can be separated right in the low temperature cell, and if the pure $^3$He phase is subsequently precooled to the superfluid phase, there is practically no entropy, *viz*. heat capacity, in the system. This stage of the precooling can be accomplished by adiabatic nuclear demagnetization and necessitates at least reasonably good thermal contact from the liquid to the cell wall through moderate amount of sintered silver powder. Once the proper initial conditions are set, some $^4$He can be removed from the cell through the superleak line and $^3$He will dissolve into $^4$He being released from the melting solid, thus reducing the temperature by the process of mixing. Since there was almost no entropy in the system to begin with, under adiabatic conditions this will produce a mixture with very low entropy as well, meaning that the temperature must be greatly reduced. The cooling factor can exceed several hundreds with favorable starting conditions and reversibly executed melting and mixing process [5].

This protocol, which was followed in the early experiments [6], already introduced some problematic components, which may limit the performance of the method in practical realizations. First, the liquid in the cell has to be thermalized to below 1 mK with the cell wall through sintered silver powder to overcome the Kapitza resistance. At the melting phase this works against us as the liquid is then supposed to be colder than the cell wall and the cooling power available at the lowest temperatures is not that great. Second, the low temperature cell is connected to substantially higher temperatures through the superleak line, which is prone to transmit undesired disturbances from the high temperature parts of the cryostat down to the cell. As the superleak is filled with pure superfluid $^4$He, it cannot conduct heat as such, but it may transmit pressure noise as the fourth sound with practically no attenuation at all. The driving force for such disturbances may be the temperature variations in the dewar or the 1 K pot [11], or an instability of the location of the lambda point in the filling line between the pot and the 4 K plate in the vacuum jacket [12]. These were indeed identified as points of concern in the early tests of this cooling scheme [6, 13].

To quantify the setting for the attempted experiments, the cooling power at molar rate $\dot{n}_3$ is given by the entropy difference:

$$\dot{Q} = \dot{n}_3 \cdot T \cdot (S_{3,d} - S_{3,c}) = 100.5 \cdot \dot{n}_3 \cdot T^2. \quad (1)$$

At $T = 100\ \mu$K and with flow rate of $^3$He, $\dot{n}_3 = 100\ \mu$mol/s from the concentrated to the dilute phase, labelled by the subscripts $c$ and $d$ above, the cooling power is equal

to $\dot{Q} = 100$ pW and decreases as $T^2$ with temperature. Therefore parasitic power sources even at pW-levels may be detrimental to the desired performance. In this paper we describe the measures taken to provide as perfect an isolation as possible of the experimental chamber from any conceivable external sources of energy during the final cooling phase of the liquid mixture.

## 2 Bellows system

The density of the solid $^4$He phase is about 10% bigger in comparison to the liquid, which means that growth of solid phase needs supply of additional liquid into the cell. An ordinary filling line would become blocked by solid at a temperature of about 1 K. Using a special superleak line (SL) made of a tube tightly packed with fine powder, one can transport liquid into the cell from across 1.5 K to millikelvin temperatures [3]. Only the superfluid component can freely flow through the superleak. The normal component is stuck in the powder pores. The thermal conductivity along the superleak is negligible but there is another mechanism for energy transfer through it — so called fourth sound can propagate in tightly packed powders.

To protect the sample from heating through the superleak by pressure noise, we have built a mechanical system, which prevents direct propagation of acoustic noise. The new filling line arrangement is represented in Fig. 1. The line from room temperature terminates into the upper part of a hydraulic press, which transfers the flow through changing volumes into a separate superleak line connected to the low temperature cell. The upper volume of the press is filled with pure $^4$He at a temperature higher than the superfluid transition, so that the possible oscillations due to an unstable location of the $\lambda$-point are avoided completely. The bottom volume, filled with saturated helium mixture, is anchored to the mixing chamber, so that again the $\lambda$-point is not encountered anywhere within the liquid domain. Both chambers consist of bellows as flexible elements permitting to change volumes by applying fluid pressure through the appropriate filling line.

A shaft connects the two bellows making it possible to transfer a movement from one to another. To keep the movement of the shaft translational, two linear ball bearings limit its lateral displacement. The bearings and other contacting parts of the shaft are anchored to the still plate (0.7 K). Even if movement of the shaft produces frictional heating, this power loads only the still chamber, where such high heat loads can be tolerated. Both chambers are fixed to a strengthened frame which serves to sustain internal pressure of the liquid.

The system described enables good thermal protection of the low parts of dilution refrigerator from heat load coming from the upper bellows (2.2 K) and upper part of the frame, which is anchored to the bottom of the still ($\sim$ 0.7 K). The numbers at the right of Fig. 1 represent the calculated heat flows along the elements of the frame in accordance with appropriate temperature differences and thermal conductivities. The displacement of the shaft was monitored using an inductance coil fixed to the frame, while a rod of magnetic material attached to the shaft plate would change its inductance proportional to the relative distance between them.

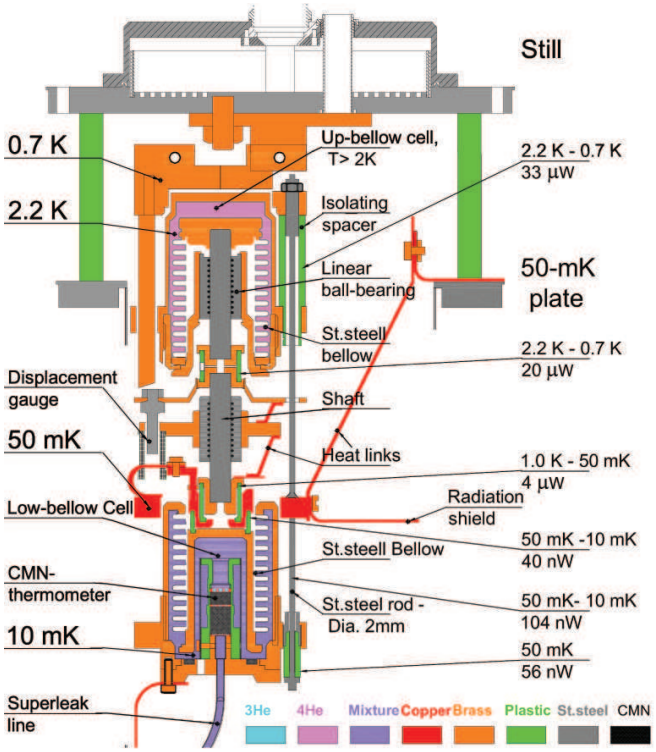


**Fig. 1** Hydraulic press with bellows. (Color figure online.)

We checked the performance of the bellows system under proper operating conditions at low temperatures and high pressures. A record of the mixing chamber temperature during full movement of the bellows is plotted in Fig. 2. During volume change of 6 $cm^3$ at flow rate of 80 $\mu$mol/s temperature of the mixing chamber remained practically constant. The rate of flow had been increased towards the end of the run with no significant change in temperature. There was a helium crystal in the bottom bellow, hence the pressure (25.3 bar) remained constant all the time. We also verified that heat input to the top bellow did not influence temperature of the mixing chamber; power up to 8 mW was applied with no impairment in performance of dilution refrigerator.

## 3 Thermal Gate.

One more advantage over former set-up [6] concerns the interior structure of the main experimental cell. The large amount of ultra-fine silver powder sintered in previous design onto upper wall of the cell and used to pre-cool the pure $^3$He phase deep into the superfluid state ($\sim$ 0.5 mK), has an opposite effect on thermal isolation of the liquid in the cell during melting process. One mechanism of energy flow to the

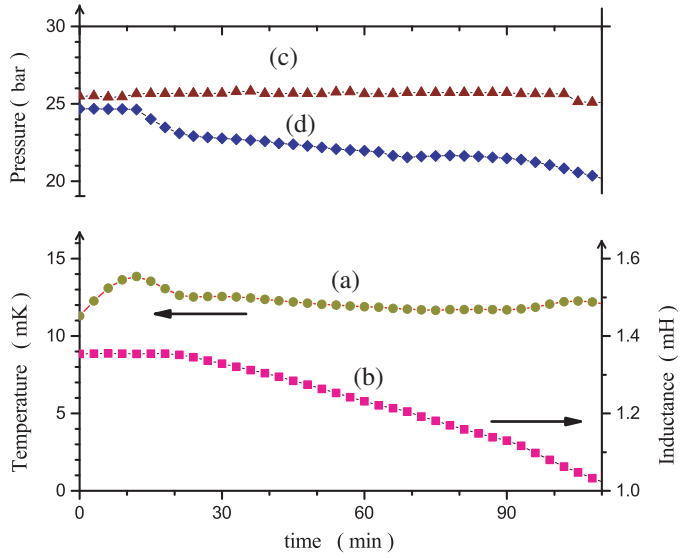


**Fig. 2** (a) Mixing chamber temperature vs time. (b) Displacement of the shaft. (c) Low-bellows pressure. (d) Top-bellows pressure. (Color figure online.)

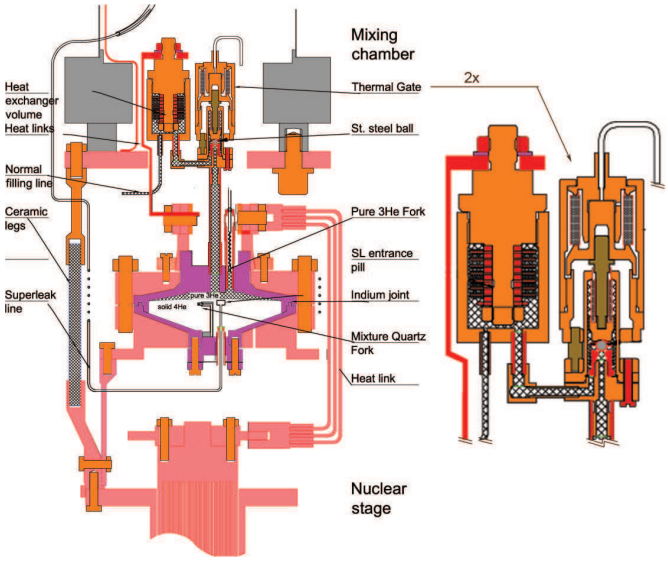


**Fig. 3** Cell and Thermal Gate arrangement. (Color figure online.)

liquid is evident: at melting the liquid should become colder than the container and the direction of heat flow then inverts. Another potential source of heat is associated with $^3$He movement in the pores within sintered Ag powder [14]. A line of attack on the problem is the spatial separation of the main heat exchanger and the mixture, with controllable heat conductivity in between.

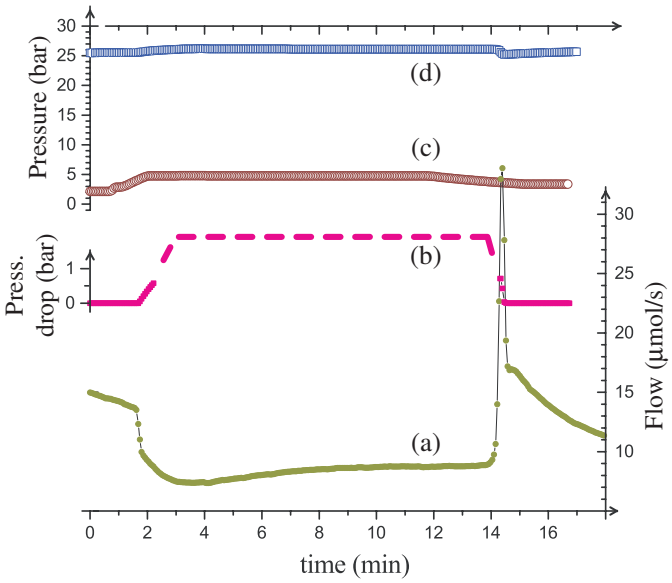


**Fig. 4** Gate operation cycle. (a) Flow. (b) Pressure drop on the gate. (c) Control pressure. (d) Cell pressure. (Color figure online.)

The new pre-cooling system (at the right of Fig. 3), which we call as Thermal Gate, consists of closely-spaced heat exchanger (left unit) and hydraulic valve (to its right) driven with pressurized liquid helium. The relative position of the thermal gate and the main cell is shown at the left of Fig. 3, along with construction in the vicinity of the cell. The heat exchanger is a copper completed shell equipped with sintered Ag sponges (of about 8 $m^2$ surface area) and through flow passage. In function, this passage is a constituent of the normal cell-filling line at preliminary phases of an experiment until top part of the line becomes blocked. Further, during period of $^4$He crystal growth and $^3$He pre-cooling, it plays the role of a heat link between the heat exchanger and the liquid in the main cell. The closing of the gate at the beginning of "adiabatic" melting reduces heat transfer through this linkage to a minimum.

The construction of the gate is akin to the one of above-described bellows system. Two low-sized hermitically sealed SS bellows are fitted into common framework. Free to move ends of both bellows are rigidly fixed to the heat-insulating rod of Vespel SP1; the same material is used as heat insulation between the brass framework and the copper bottom flange. The top bellow serves as a hydraulic actuator for a needle valve, embedded inside the bottom structure. The needle is a SS ball in form, fixed from the saddle side by a spring. The saddle constitutes the carefully smoothed conical recess around an axial hole (2 mm ID) in the copper bottom flange.

We saw no need the thermal gate would be superfluid tight, but it has to limit substantially the normal flow of $^3$He through it. On this reason, we confined the impedance testing of the system to liquid nitrogen temperature only (Fig. 4). Steady-state flow rate through the closed valve at pressure difference across it of about 2 bar has been measured as $\dot{n}_4 = 8$ $\mu$mol/s, to give hydraulic impedance value $Z$ equal to

$10^{13}$ cm$^{-3}$. Rough estimation of the heat flow under the operating conditions through the ring-shaped gap of equal impedance suggests that the heat flow at temperature around 0.5 mK will lie within the range of picoWatt.

## 4 Conclusions.

It is very challenging to cool helium mixtures to below 100 $\mu$K. The modifications to our earlier set-up described here aim to improve isolation of the experimental cell from external energy sources. The task is made complicated by the fact that the superfluid component of the mixture very effectively couples the low temperature space with the high temperature environment [6, 13]. It can transfer energy along filling lines by different sorts of sound modes. Then, at low temperature normal sound can transform into second sound by reflection from surfaces [15]. The cold hydraulic press was designed to prevent the propagation of sound through the filling line. We have verified that thermal load for the mixing chamber by thermal conductivity along the frame structure elements of the press is acceptable. Also, the presented results confirm that during movement of the shaft frictional heating in the bearings is tolerable.

Presence of the sintered powder in the cell in contact with helium mixture has been considered as one more potential source of heating [14]. Our new scheme solves this problem by placing the sintered heat exchanger into a separate volume, and by being able to adjust the thermal coupling of the sample with the heat exchanger by closing the connecting channel mechanically by a valve.

**Acknowledgements** Contributions by A. Salmela and J. Rysti, and discussions with M. Paalanen and J. Saunders are appreciated. This work was supported by the EU 7th Framework Programme (FP7/2007-2013, Grant No. 228464 Microkelvin) and by the Academy of Finland through its LTQ CoE grant no. 250280. This research was undertaken at the OtaNano - Low Temperature Laboratory of Aalto University.

## References

1. F. Pobell, *Matter and Methods at Low Temperatures* (Springer, Berlin, 2007)
2. D. J. Cousins et al., J. Low Temp. Phys. **114**, 547-570 (1999)
3. A. Sebedash, JETP Lett. **65**, 277 (1997)
4. A. Sebedash, Physica B **284-288**, 325 (2000)
5. J. Tuoriniemi et al., J. Low Temp. Phys. **129**, 531-545 (2002)
6. A. Sebedash et al., J. Low Temp. Phys. **148**, 725-729 (2007)
7. E. M. Pentti et al., Phys. Rev. B **78**, 064509 (2008)
8. J. Rysti et al., J. Low Temp. Phys. **175**, 738-754 (2014)
9. D. O. Edwards, S. Balibar, Phys. Rev. B **39**, 4083-4097 (1989)
10. C. Pantalei, X. Rojas, D. Edwards, H. Maris, S. Balibar, JLTP **159**, 452 (2010)
11. G. Lawes, G. Zassenhaus, S. Koch, E. Smith, J. Reppy, J. Parpia, RSI **69**, (1998)
12. V. Arp, Heat Transport through helium II, Cryogenics **10**, 96-105 (1970)
13. A. Sebedash et al., J. Low Temp. Phys. **150**, 181-186 (2008)
14. R. König, A Betat, L. Roobol, A. Voncken, F. Pobell, JLTP **101**, 107 (1995)

15. L. A. Melnikovsky, J. Low Temp. Phys., **150**, 174-180 (2008)