

H3 Experiments with miniature heat exchangers for dilution refrigerators

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This paper describes experiments done on three types of miniature heat exchanger for dilution refrigerators. These have liquid volumes on the order of 0.1 cm^3 . The first is a continuous exchanger incorporating Cu(Cr) powder, the second is a continuous exchanger incorporating sub-micron silver powder, and the third is a discrete exchanger incorporating $1.8 \mu\text{m}$ copper powder. The first two were not successful and the reasons are not entirely clear. The third exchanger performed about as calculated and permits an order of magnitude reduction in liquid volumes from that normally used.

1 Introduction

The dominant heat capacity in most dilution refrigerators is that of the liquid He^3 in the system. Cool-down and equilibrium times are then proportional to the liquid volume in the refrigerator. Faster cool-down times are often desirable simply to speed the data-taking process, whereas in some experiments, such as that of spin-frozen polarized proton targets [1], rapid cool-down is required to make the experiment feasible. To reach a temperature of 10 mK, the total liquid volume in a set of heat exchangers has usually been about 1 cm^3 for a $20 \mu\text{mol/s}$ He^3 flow rate, provided the design has been optimized [2]. Such a universal size is due to the widespread use of -325 mesh pure copper powder in a set of discrete heat exchangers. Adsorption measurements [3] indicate the surface area of this -325 powder is equivalent to $7 \mu\text{m}$ diameter spheres.

Results of recent Kapitza resistance measurements on copper with 0.6 wt% chromium [4], $1.8 \mu\text{m}$ diameter copper powder [3], and $0.15 \mu\text{m}$ diameter silver powder [5] suggest that the use of these new materials in heat exchangers could improve heat transfer and thus permit much smaller liquid volumes to be used. The techniques of using these three materials in miniature heat exchangers with negligible viscous heating are described in this paper. Two of the heat exchangers were designed as the continuous type [6] to take advantage of the more efficient heat transfer in that type compared with a discrete heat exchanger.

2 Calculated behaviour

2.1 Surface area

All the heat exchangers were designed for an ultimate mixer temperature T_m of 10 mK with a He^3 flow rate \dot{n}_3 of

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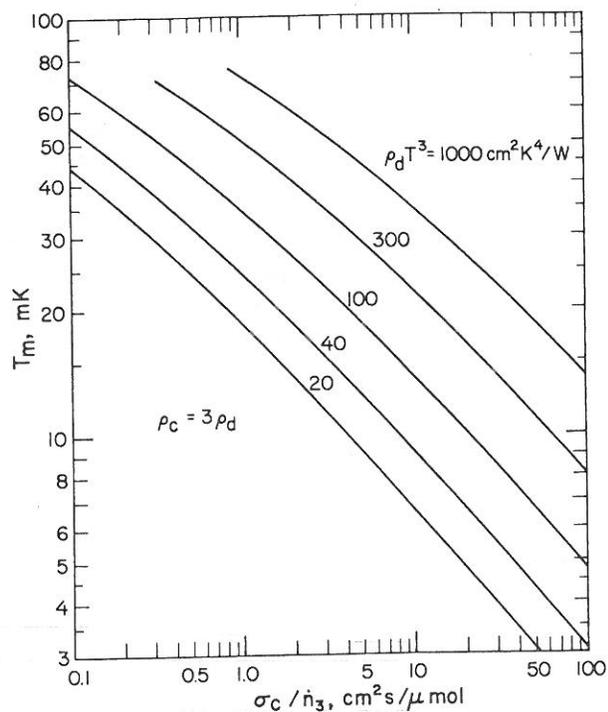


Fig.1 The calculated minimum mixer temperature as a function of the concentrated stream surface area divided by the He^3 molar flow rate for an ideal continuous heat exchanger. The various curves are for different Kapitza resistivities.

Table 1 Calculated surface areas and sponge volume for the concentrated side of three different heat exchangers.

Exchanger	Powder size (μm)	$\rho_d T^3$ ($\text{cm}^2 \text{K}^4 / \text{W}$)	σ_c / \dot{n}_3 ($\text{cm}^2 \text{s} / \mu\text{mol}$)	σ_c (cm^3)	$(V_{\text{sponge}})_c$ (cm^3)
Cu(Cr)-continuous	~ 10.0	20	4	80	0.033
Ag-continuous	0.15	~ 1000	200	4000	0.025
Cu-discrete	1.8	300	50	1000	0.075

$2 \times 10^{-5} \text{ mol s}^{-1}$. Figures for calculating heat exchanger surface areas have been presented [2, 6]. The calculations have been extended to include different values of ρ_c and ρ_d , the Kapitza resistivities in the concentrated and dilute sides, respectively. Figure 1 shows how the calculated ultimate mixer temperature varies with the surface area σ_c on the concentrated side of a continuous heat exchanger. This figure is valid for $\rho_c / \rho_d \approx 3$, which may be true for most materials, and for $\sigma_d / \sigma_c \approx 1.5-2.0$. Table 1 shows the calculated surface areas and volumes required on the concentrated side for powders packed to 40% of full density. These numbers are based on the measured $\rho_d T^3$ values

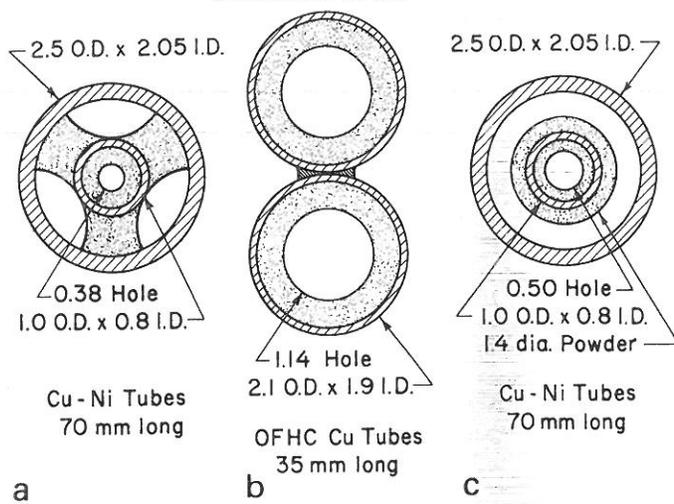


Fig.2 A cross-sectional view of the three different types of heat exchanger tested. Fig.2(a) is a continuous exchanger with Cu(Cr) powder, Fig.2(b) is a discrete exchanger with copper powder, and Fig.2(c) is a continuous exchanger with silver powder.

given in Table 1. For the one discrete heat exchanger the σ_c value was determined from Fig.5 of Ref.[2], which is for discrete heat exchangers of negligible heat conduction along the flow direction. The curve for a relative effectiveness of 60% was used and the resultant σ_c was scaled to take into account the different value for ρ_d for the 1.8 μ m copper powder. A relative effectiveness of 60% means that for a concentrated stream outlet temperature T_{co} of $T_m/0.36 \approx 28$ mK, the concentrated stream inlet temperature T_{ci} to the exchanger is 52 mK.

2.2 Viscous heating

The geometry used to achieve negligible viscous heating in the three different heat exchangers is shown in Fig.2. The parallel tube geometry of the discrete heat exchanger, Fig. 2(b), is very much like that used by Niinikoski [1] with coarser powders. Viscous heating in the exchanger causes the mixer temperature T_m (before viscous heating) to increase by ΔT_m , which can be calculated approximately from equations given previously [6, 8]. For a continuous heat exchanger the viscous temperature rise can be expressed approximately as

$$\left(\frac{\Delta T_m}{T_m}\right)_c = 0.07 \left(\frac{Z_c/4}{10^8 \text{ cm}^{-3}}\right) \left(\frac{\dot{n}_3}{10^{-5} \text{ mol.s}^{-1}}\right) \left(\frac{20 \text{ mK}}{T_{co}}\right)^4 \quad (1)$$

for the concentrated side and

$$\left(\frac{\Delta T_m}{T_m}\right)_d = 0.017 \times 0.54 \left(\frac{Z_d/4}{10^8 \text{ cm}^{-3}}\right) \left(\frac{\dot{n}_3}{10^{-5} \text{ mol.s}^{-1}}\right) \times \left(\frac{20 \text{ mK}}{T_m}\right)^4 \quad (2)$$

for the dilute side. In these equations Z is the flow impedance through the total length of the exchanger and T_{co} is the concentrated stream temperature at the outlet of the exchanger, which is approximated by $T_m/0.36$. The calcu-

lated $\Delta T_m/T_m$ was less than 2% for the three different exchangers.

2.3 Axial thermal conductance

Effects of the axial thermal conductance were determined by calculating the parameter Y_j given in Ref.[2] and [6]. The axial liquid conductances in all three exchangers were calculated to be negligible as were the body conductances of the two continuous exchangers. The thermal conductivity coefficients, k/T , used for the three different powders were: Cu(Cr), $2 \times 10^{-3} \text{ W/cm K}^2$; Ag, $6 \times 10^{-3} \text{ W/cm K}^2$; Cu, $2 \times 10^{-2} \text{ W/cm K}^2$. The low value measured for the silver powder is due to its extremely fine powder size. Both the Cu(Cr) and Ag powders have low enough thermal conductivity to be used in a continuous exchanger.

3 Construction of heat exchangers

For all three heat exchangers it was necessary to pack the inside of a tube with powder and yet have a hole extending through the powder. To accomplish this the tube was pre-coated with a dusting of 1.8 μ m copper powder that was fired on at 850°C for 30 min in hydrogen. A music wire of the correct size was inserted in the tube to provide the flow channel through the powder. Because the silver powder shrinks slightly even with a sintering temperature of 150°C, a tapered nylon fishing line was used instead of the music wire to provide the centre hole in this exchanger. The nylon expands more during the sintering and forces the silver powder out against the tube walls. An appropriately sized tube, tapped lightly with a hammer, was used to pack the metal powders in the annulus between the wire and the tube. The wire was moved occasionally during the packing to prevent the powder from binding too tightly to it. The music wire could be removed only before sintering for the copper powder exchanger. If handled with care before sintering, the powder would stay in place. The music wire or nylon line was removed after sintering for the other two exchangers.

The two continuous exchangers, Cu(Cr) and Ag, require, in addition, that powder be packed on the outside of the 1 mm Cu-Ni tube. For this a split graphite mould was used. The sintering conditions in a hydrogen atmosphere for the three exchangers were as follows: Cu(Cr), 750°C for 1 h; Ag, 150°C for 30 min, Cu, 650°C for 30 min. Three grooves were milled in the outside powder of the Cu(Cr) exchanger as shown in Fig.2(a). This difficult procedure was abandoned in the construction of the silver exchanger, Fig.2(c). The two continuous exchangers were completed by inserting the powder-coated 1 mm Cu-Ni tube inside another Cu-Ni tube and soldering on end fittings. The Cu(Cr) exchanger was then held at 450°C for 5 hours to precipitation harden the

Table 2 Actual surface areas and liquid volumes of the three heat exchangers after construction.

Exchanger	σ_c (cm ³)	σ_d (cm ³)	Packing fraction	(V_l) _c (cm ³)	(V_l) _d (cm ³)
Cu(Cr)	86	318	45%	0.027	0.15
Ag	3600	7700	37%	0.030	0.16
Cu	1100	1100	60%	0.060	0.060

Cu(Cr) powder [4]. The copper exchanger was finished by hard soldering the two copper tubes together in hydrogen at 680°C. The actual surface areas and liquid volumes of the three exchangers are shown in Table 2.

4 Experimental results and discussion

4.1 Cu(Cr) continuous heat exchanger

This exchanger was bent into a semicircle and mounted so the liquid flows would be horizontal to eliminate convective instabilities. Two models of this exchanger were built. The first was incorporated into a new dilution refrigerator designed to take full advantage of the small size of the heat exchanger. The lowest continuous mixer temperature reached with this system was a disappointing 89 mK. A second Cu(Cr) exchanger was made and this bypassed all the heat exchangers in an existing reliable dilution refrigerator. The mixer reached a temperature of 79 mK continuously. This somewhat lower value could be due to typical variations in both the refrigerator and constructed exchangers. After placing a 32 cm long conventional Cu-Ni coaxial tube heat exchanger before the Cu(Cr) exchanger the mixer reached 32.5 mK.

In an effort to explain the poor performance, we measured the Kapitza resistance of the Cu(Cr) powder used in the two heat exchangers. The Cu(Cr) powder was made by grinding a piece of bulk Cu-0.6 wt.% Cr and taking the powder which passed through a screen with openings of 44 μm . The results gave a value for $\rho_d T^3$ of about 250 $\text{cm}^2 \text{K}^4/\text{W}$ for the powder compared with the value of 20 $\text{cm}^2 \text{K}^4/\text{W}$ used in the calculations and determined from measurements of bulk Cu(Cr). Experiments are being done to try to understand the discrepancy between the Kapitza resistance of powder and bulk Cu(Cr).

4.2 Silver continuous heat exchanger

This exchanger was also bent into a semicircle and mounted horizontally in the second dilution refrigerator. In the first test the 35 cm long Cu-Ni coaxial tube heat exchanger was placed before the silver exchanger. The mixer reached only 39.3 mK. Two conventional copper powder discrete exchangers were then added between the Cu-Ni and silver exchangers, and the mixer reached 16.3 mK. With one more conventional copper powder exchanger the mixer reached 14.3 mK. Without the silver exchanger the mixer reached 14.8 mK. At this time we have no explanation for the poor performance of this exchanger. The Kapitza resistance measurements [5] were actually done on the same powder in the dilute stream, but in a copper cell instead of Cu-Ni.

4.3 Copper discrete heat exchanger

This miniature discrete exchanger was added as the fourth discrete exchanger to the second dilution refrigerator which before had reached 14.8 mK. The mixer then reached 13.5 mK and 13.8 mK on two separate runs. With the third conventional exchanger removed from the system the mixer temperature ranged from 14.0 to 14.4 mK on three separate runs in the continuous mode and 9.8 mK one shot. Thus the new miniature heat exchanger performed slightly better

than a conventional discrete heat exchanger which had five times the concentrated liquid volume and 10 times the dilute liquid volume. Measured values for T_{ci} and T_{co} for the new heat exchanger were about 48 mK and 29.5 mK, respectively. The calculated behaviour gave $T_{ci} = 52$ mK and $T_{co} = 28$ mK. The small difference could easily be due to the fact that the dilute side surface area should have been nearly twice that of the concentrated side. The actual dilute side surface area instead was made equal to that of the concentrated side because of technical details.

5 Conclusions

The results of our experiments so far on miniature heat exchangers show that small discrete heat exchangers can be made with 1.8 μm copper powder. Even though the liquid volume was less than 0.1 cm^3 on each side, it behaved slightly better than a more conventional discrete heat exchanger with about ten times the liquid volume. Further, size reduction should be possible with a continuous heat exchanger using a fine powder. However, we have not been successful yet with the miniature continuous heat exchangers, but we hope that after further experiments we will have success.

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