Study of Heat Leaks to Copper Nuclear Demagnetization Stage

Hisashi Nakagawa [1], Hideo Yano, Osamu Ishikawa, Tohru Hata

Graduate School of Science, Osaka City University, Osaka 558-8585, Japan

Abstract

Heat leaks to a copper nuclear stage at ultra-low temperatures have been investigated. We have constructed a massive copper nuclear stage and performed its cooling with negligible amount of heat release sources such as plastics and epoxies. With this stage we have managed to cool a Pt-NMR thermometer down to 45 μK at the bottom of the stage. The whole heat leaks were found to be 1nW at a final field of 10 mT on the 10 th day after cooldown to 4.2 K and remain fairly constant during 2 months. The small and time-independent heat leaks enable us to obtain the lowest temperature quite soon and maintain temperatures below 100 μK for several weeks.

Key words: copper nuclear demagnetization stage; time-dependent heat leak;

Nuclear adiabatic demagnetization has been utilized for refrigerating materials such as superfluid ^{3}He in submillikelvin regions. Recently, two types of the nuclear stage have been used: a stage machined from a massive copper block[1] and a stage consisted of the copper plates[2]. In these nuclear refrigerators time dependent heat leak has been observed. This heat leak requires more than 1 month to decrease to about 1 nW after cooldown to 1 K. In ultra-low temperature studies it is very important to reduce heat leaks to a nuclear refrigerator and an experimental space, because the achievable lowest temperature and cooling-time of the nuclear refrigerator depend directly on heat leaks. The sources of time dependent heat leak have been found in materials such as epoxies, grease and plastics[3]. Also the ortho-para conversion of H_2 present in some metals has been considerd as a time dependent heat leak; the ortho-para conversion of 1 ppm H_2 in 1 kg copper produces a heat leak of about 5 nW[3].

In the present paper we report heat leaks to a massive copper nuclear stage which we have constructed for the study of quantized vortices and impurity effect in superfluid ^{3}He below 200 μK and the reserch for superfluidity in ^{3}He-^{4}He mixture below 100 μK. For reduction of heat leaks we used no heat release material such as graphite instead of plastics, and investigated the characteristics of the nuclear stage.

Our nuclear stage has been machined from an oxgen free copper (OFC) rod with purity 99.99 % (Hitachi Cable Ltd.). We slited the copper rod with a metalsaw for the reduction of an eddy current heating and it has fifty-two plates of 2 mm thick parallel to its axis. The total amount of copper is 158.6 moles (10 kg) and in a field of 8 T is effective 90.6 moles. The residual resistivity ratio has been increased up to 4000 after heat treatment for 144 hours in vacuum of 1×10^{-3} torr at 950 °C. The nuclear stage is mounted under the mixing chamber of a home made dilution refrigerator through a Pb heat switch. Temperature at the bottom of the stage has been measured with a Pt-NMR thermometer supported with a copper rod inside a Nb shield. A ^{3}He melting curve thermometer is also mounted on the bottom of the stage for temperature calibration. A heater for the measurements of heat leaks and heat capacity is on the top of the stage.

We only used small amount of materials; Kapton tape ($\sim$0.15 g), stycast ($\sim$0.3 g), Be-Cu ($\sim$4.7 g), cotton thread ($\sim$0.3 g) and graphite with 99.9% purity ($\sim$19 g) . For instance, the estimated heat release of 0.3 g stycast is about 6 pW after one week cooldown

[1] E-mail: hisakyu@sci.osaka-cu ac.jp

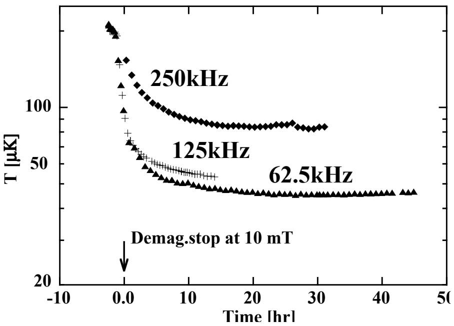

Fig. 1. Temperature measured with the Pt-NMR thermometer as a function of time after demagnetizations to a final field of 10 mT. The measurements were performed at three NMR frequencies of 250, 125 and 62.5 kHz.

to 1 K[3]. Supports between the stage and the mixing chamber are graphite rods. And we used screws machined from OFC rod.

The nuclear stage in a magnetic field of 8 T has been precooled with the dilution refrigerator. After 4 days precooing the nuclear stage has been demagnetized to 100 mT with a sweep rate of 10 mT/min and then to a final field of 10 mT with 1 mT/min. We have performed temperature measurements at three different frequencies of Pt-NMR thermometer by the demagnetizations for each frequency. Figure 1 shows the relaxation of temperature for each NMR frequency at the final field. We found that the lowest temperature is decreasing with frequency. At the frequency of 62.5 kHz we obtained the lowest temperature of 45 μK at about 26 hours after demagnetization.

Heat capacities and heat leaks have been also measured at several magnetic fields. The measured heat capacities between 60 μK and 4 mK are consistent with the calculated values, which implies that the nuclear stage has no extra heat capacity. The heat leaks at the final field are shown in Fig.2 as a function of time after cooldown to 4.2 K. The whole heat leak was found to be 1 nW after only 10 days and remain fairly constant during 2 months. We also show a heat leak at 0 mT to the nuclear stage of the Bayreuth group[1]. They suggested that time dependent heat leak is caused by the ortho-para conversion of H_2 in the nuclear stage. In our stage, however, the time dependence could not be observed at10 mT. Consequently, the heat leak due to the ortho-para conversion is negligible small in our stage. This result also implies that we can eliminate the time dependent heat leaks by removing plastics and epoxies from the nuclear refrigerator. At high magnetic fields we have observed field dependent heat leaks, which should be produced by relative movement between a magnet and the stage. At the magnetic field of 100 mT the heat leak is also shown in Fig. 2. The time dependence was observed at 100 mT, but this origin has not been identified yet.

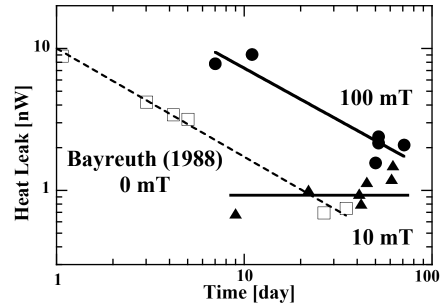

Fig. 2. Heat leaks as a function of time at 100 mT (solid squre) and 10 mT (solid triangle) after cooldown to 4.2 K. Heat leak data (open square) at 0 mT in Bayreuth group[1] are also shown. The solid line and broken line are for guide to the eyes.

The NMR frequency dependence of the minimum temperature implies a temperature difference between the Pt-NMR thermometer and the stage. From the entropy loss measurements by the method of demagnetizing and magnetizing the stage, the minimum temperature of the nuclear spins of the stage is estimated to be 20 μK at the final field. The temperature difference should be caused by heat leaks into the thermometer. Supposing the thermal resistance between the thermometer and the Cu foot[4], we estimated the heat leak to be about 40 pW. The heat input by an r.f. pulse for Pt-NMR is estimated to be 1.6 nJ applied every 3000 seconds. Thus, heating due to the temperature measurement is negligible. Magnetic field for Pt-NMR should also be considered. Eddy current heating in the thermometer and a metal support is induced by an ac component of the field and /or a mechanical oscillation. As shown in Fig. 1, this effect is observed between 250 and 125 kHz and becomes small when we decrease the frequency to 62.5 kHz. Therefore, we can not explain this large temperature difference from only the r.f. pulse and the eddy current heating, and should consider the other unidentified origins of the heat leak. A silver support might be utilized rather than a copper support for reduction of the temperature difference[1].

In summary, by decreasing the sources of the time dependent heat release such as plastics, we could considerably reduce the heat leaks into the nuclear stage. Due to the small and time-independent heat leak, we can obtain the lowest temperature quite soon and maintain temperatures below 100 μK for several weeks.

References

[1] K. Gloos, *et. al*, J.Low Temp. Phys.**73** (1988) 101

[2] P.Skyba,*et. al*, Cryogenics **37** (1997) 293

[3] F.Pobell, Matter and Methods at Low Temperatures, Spinger-Verlag (1992)

[4] T.Mamiya,*et. al*, Rev. Sci. Instrum. **58** 1428 (1988)