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The New Cornell Copper Demagnetization Stage

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The persistent failure of a previous welded copper demagnetization stage to reach temperatures significantly below 100 microkelvin caused us to replace the original structure. The new stage has been machined from a single ingot of moderately high purity (4 9's) polycrystalline copper, with a much more rigid cross-section. Heat treatment increased the RRR of the copper to 4000. The cryostat is now capable of maintaining temperatures below 50 microkelvin for over a week. However there appear to be gradients between the thermometer and the copper nuclei in the main field, which may be a factor of 5 or more colder. Much of the improved performance comes from the identification and removal of an existing heat leak. The vibrational heating is dramatically smaller than that of the old stage, inferred from the magnetic field dependence of the heat leak.

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1. INTRODUCTION

As a motivation for the change which has been made in our demagnetization stage, we first discuss very briefly the construction and performance of the previous assembly, and describe which potential weaknesses we wished to cure. Next we describe the machining and heat treatment of the new stage in somewhat greater detail. The heat treatment in particular has some interesting aspects. Finally we address the current improved performance levels and the question of what is currently limiting the minimum temperature.

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2. OLD STAGE

The former demagnetization stage was formed of 18 bars of high purity copper (5-9's), initially in the form of 10 mm rods, but rolled into roughly rectangular pieces about 3 mm thick. These were then heat treated to achieve a RRR of around 1000, and then e-beam welded into a top plate of high purity copper 150 mm diameter and 13 mm thick, cut from a bar indicated by the supplier as having a RRR of 600. After some initial problems with vibrational heating, the assembly of bars was stiffened by adding a few drops of epoxy at several levels to bond the bars together into a more rigid array. During the initial stages of demagnetization, down to about 300 μK, the temperature as measured by the Pt NMR thermometer evolved in a nearly reversible fashion, though with about a 10% drop in H/T. However, the system became highly irreversible below this temperature, and the temperature as measured by the Pt thermometer on the top plate would only go down slightly below 100 μK, although nuclear temperatures measured on the copper directly in the demagnetization region (using the demagnetization field to provide the H_0) were much lower. Although this temperature range was adequate for many experiments, we eventually decided an improvement was needed. We were concerned with several details of our earlier construction as potential bottlenecks in the performance. The e-beam weld was rather uneven looking, and we had been worried that if one bar were loosely attached thermally, it could present a virtual heat leak. The inability to anneal the top plate, because it was too big for our tube furnaces, gave a potential thermal resistance and a possible Kondo specific heat contribution if there were non-oxidized magnetic impurities in low concentration. The low rigidity of the assembly made it excessively prone to vibrational heating. All these factors led us to a monolithic fabrication by machining from a single bar of copper small enough to fit into one of our annealing furnaces.

3. NEW STAGE FABRICATION

The new stage was machined from a single bar of 'cryogenic grade oxygen-free' copper[1] 100 mm in diameter and 800 mm length. Slabs were cut off 4 sides over most of the length, leaving 13 mm on one end at the full diameter, and then the rest of the length was turned to a 40 mm diameter. To reduce eddy-current heating, slots were cut over the length of the bar, leaving an array of fins as shown in figure 1.

These were cut in four groups of 5 slots at a time using ganged slitting saws. The machining required considerable experimentation with test pieces, but was eventually quite reliable as long as all fixturing was well secured, the cut depth was limited to approximately 5 mm (we used .75 mm thick saws),

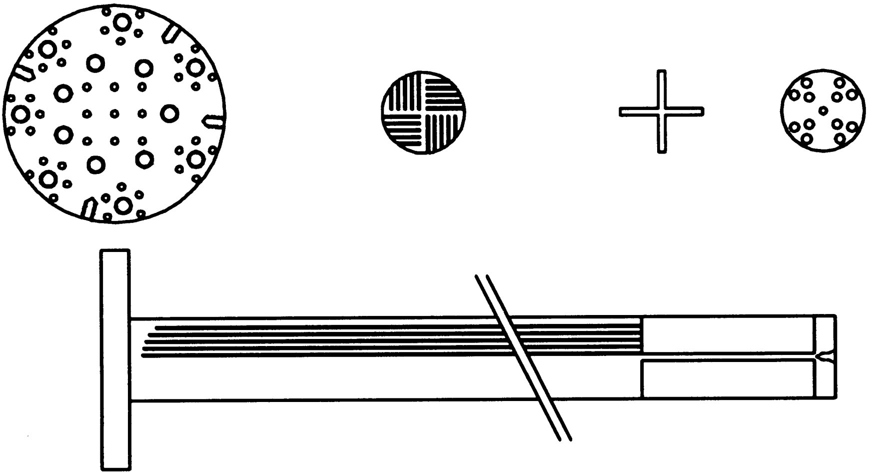

Fig. 1. Side view and several sections of the new copper stage.

and the cutters were run in a "climbing" direction of rotation under continuous spray lubrication. The surface speed of the cutters was maintained at a usual value for copper, but the feed rate was kept very low. It was found prudent to replace the cutters after each set of slots. On the top flange, the main experimental area, two different types of mounting arrangement were machined, for compatibility with our pre-existing sample and thermometry mounts–tapered holes of roughly 7 mm diameter, intended to accept mating tapered (Morse #0) rods, to be tightened by a nut and washer below the plate, and arrays of threaded M3 holes, with which flat plates could be mounted onto the plate. Many of these mounting holes were placed near the outer perimeter of the 100 mm plate, as the actual experimental area extends to 160 mm diameter. Four copper support posts were machined to extend from the perimeter of the new stage to the 160 mm diameter of the support structure, and were attached by M5 copper screws to the top plate. The support structure consisted of 4 square brass posts (8mm) extending most of the distance (160 mm) down from their mounting on the sides of the mixing chamber, with the last 40 mm being comprised of 8mm Vespel SP22[2] rod to provide thermal isolation. Segmented brass rings were attached to the brass posts at two levels to provide a substantial degree of stiffening to this structure. These rings may be removed to give more convenient access during assembly of experiments.

At the bottom end of the stage, provision was made for clamping either an additional experiment, or a thermal link to a second demagnetization stage. Our demagnetization magnet has compensation coils to reduce the fields dramatically at both ends of the bundle, so that eddy currents should not be a problem from this section which has a solid cross-section.

Connection to the heat switch was through 10 mm copper rods, the end of one clamped to the heat exchanger in the mixing chamber, and the end of the other fastened to one of the tapered holes in the stage plate. The heat switch itself consisted of 4 tin wires of high purity, with a RRR greater than 10000, 1.25 mm diameter and 5 mm free length, soldered into the copper bars at the height of the mixing chamber. Although the heat switch was a considerable distance from the stage, and in the compensated region of the demagnetization magnet, a Nb shield was placed around its magnet to reduce any possibility of interactions from fringe fields.

Of the total 116 moles (7.4 kg) of copper in the stage, an effective 77 moles (4.9 kg) is in the high field region (the maximum rated field of the magnet is 9 Tesla at the center, dropping to 5 mT at the end plates at the same 74.5A current). Because of the design of our magnet system, the copper rod is somewhat longer and thinner than for the cryostats in some other labs. As the electronic heat conduction from the region of nuclear cooling to the region of experimentation is proportional to the area/length ratio and to the electrical conductivity, it is important to try to compensate for the slightly unfavorable geometrical factor by achieving a very high conductivity for the copper in our stage. The manufacturer provided a recipe for a low-temperature (500°C for 1 hour) anneal guaranteed to give a RRR of 500 for this grade of copper, but this seemed somewhat marginal for our purposes. We did a much higher temperature anneal in a small partial pressure of air, a popular technique in the low temperature community.[3] The optimal conditions seem to depend somewhat on the exact nature of the magnetic purities in a particular sample of copper, and we made measurements on a number of test pieces. Although the best results were obtained at temperatures of about 1050°C for periods of a few hours at a pressure of $6x10^{-5}$ mbar, operating at a temperature this close to melting made us worry about the quality of temperature control in the vacuum furnace. Also, the rate of evaporation became quite significant during the period of heat treatment. Thus we eventually decided to use a 950°C anneal for 6 days at that same pressure. The air was continually admitted through an impedance furnished by a length of small bore capillary, chosen to match the pumping speed of the pumping system (limited mainly by the flexible hose connection to a small turbo pump. This eventually resulted in a RRR measurement of 4000 for three test samples which were placed in the tube furnace at the same time as the actual demagnetization stage (because of the geometry of the stage, its resistance was too small for us to measure accurately). When it came time to do the annealing of the stage, we had to move to a larger furnace than we had used for the trial pieces, and to arrange a support to keep the long copper bar from sagging during the annealing process. The initial support structure was made from graphite, and turned out to be completely unsatisfactory–test pieces yielded a RRR value of only 200. This may have been either because of the oxygen in the admitted air having reacted with the graphite and not being available to oxidize metallic impurities in the copper, or may have been actually caused by carbon dissolving into the metal and causing some sort of scattering, as the results were actually substantially worse than an anneal in vacuum at the same temperature. An additional test with a stainless steel fixture, covered with a thin layer of silica cloth to avoid direct contact with the metal, was also unsuccessful–apparently significant amounts of chromium were distilling out of the stainless steel and being taken up by the copper. Finally an alumina silicate ceramic,[4] machinable before firing in air to 1100°C, was used , and this proved to be very satisfactory. (Slabs of the ceramic material approximately 15mm thick by 100mm wide and 300 mm long had a shallow groove machined to match the curvature of the long 40mm copper rod, and formed a tray resting on the quartz tube walls to support the copper stage centered in the furnace. A smaller piece of the ceramic supported the larger diameter plate at the top. The copper rested directly on the ceramic during the annealing process.) The copper support pieces were annealed at the same time as the main bar. The bar for the heat link rod was annealed separately using the same recipe, but only for 72 hours, and test samples run concurrently gave a RRR of 3500.

4. NEW STAGE PERFORMANCE

For a first assessment of performance, the new stage was cooled down with only the minimal instrumentation of a Pt NMR thermometer, a thick-film ruthenium oxide resistance thermometer for observing pre-cool behavior, and a heater. We were disappointed to discover that on the first cooldown, the lowest temperature performance was rather similar to that of the old stage–a heat leak of about 4 nW, and a bottom temperature of slightly below 100μK. In other respects there was great improvement. The time-dependent heat leak present after the initial cooldown vanished considerably faster (within about two weeks). On the old stage we had been confronted with a rather considerable field-dependent heat leak which was very noticeable in warm-up rates, being as much as 100 nW at fields around 1T; on the new stage this additive heat leak was too small to observe. At this point we realized that the Nb outer conductors for four superconducting coaxial cables extending from the 1K pot down to the stage were a potential source of heat leak if the thermalization at intermediate temperature levels were not adequate. These were removed, and on the next cooldown we achieved a minimum temperature of approximately 45 μK. There was essentially perfect reversibility down to about 100 μK, followed by a rapid loss of contact with the cold nuclei as demagnetization continued. It seems that there is still a considerable thermal bottleneck somewhere between the cold copper nuclei and the electrons cooling the Pt NMR thermometer mounted on the stage plate, as the temperature can be sustained below 50 μK for a week (during which time considerable inputs of electrical heating were applied to bring things up to a temperature where the thermometer was again in some closer approximation to equilibrium with the copper in the field). An additional unidentified heat leak of nearly 1 nW persists, but computationally this would appear unlikely to provide a 40 μK temperature gradient. There is some concern that we have a small residual heat leak directly into the Pt thermometer, perhaps because of the fact that the H_1 coil has been wound directly onto the Pt wires. We have currently replaced this coil with a separated coil (at a considerable cost in filling factor), and hope to test this in the near future.

ACKNOWLEDGMENTS

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