7. Magnetic Thermometry

7.1 Magnetic Thermometer

Magnetic thermometry is based upon measurement of paramagnetic susceptibility. Useful papers concerned with the EPT-76 temperature range are Rusby and Swenson (1980); Cetas and Swenson (1972); van Rijn and Durieux (1972); Cetas (1976); Mangum and Bowers (1978). For an ideal paramagnet the zero-field susceptibility is related to temperature through the Curie law $\chi = C/T$ where C is the Curie constant. Although in magnetic thermometry one approximates to this by using dilute paramagnetic salts, it is generally necessary to take account of interactions and other effects and write $\chi = C/(T + \Delta + \gamma/T)$, where Δ includes first-order dipole-dipole and exchange couplings and also a shape factor, while γ is due primarily to crystal field splitting of the ground state and second-order interaction effects [Hudson (1972)]. The existence of interactions implies a lower limit for the use of any given salt, while the upper limit is set by diminishing sensitivity ($d\chi/dT$ is approximately equal to $-C/T^2$). The susceptibility measurement is usually made by the ac mutual inductance method in which the salt sample is situated in a set of coils whose mutual inductance M is balanced against a reference in a Hartshorn bridge or a variant thereof [Hudson (1972)] (SQUID techniques do not appear to have been applied much above 1 K). The bridge balance X is linearly related to M and hence to χ. The working equation for a magnetic thermometer becomes

$$X = A + B/(T + \Delta + \gamma/T) \qquad (7.1)$$

Unless Δ or γ is obtainable from theory, a minimum of four fixed points is needed to calibrate the thermometer.

Salt crystals must be grown carefully from ingredients of high purity. Most of the suitable salts are hydrates and almost all of these are efflorescent, tending to lose water-of-crystallization if kept at room temperature. This tendency is considerably diminished if the enclosing volume is small and is filled with an inert gas; but it is catastrophically increased if the enclosure is evacuated. Simple refrigeration, however, avoids most such problems. The properties of the most important salts have been widely discussed and tabulated [e.g. Hudson (1972)]. Cerous magnesium nitrate (CMN) is the closest approximation to an ideal paramagnet in common use: $\gamma = 0$ and for a sphere Δ is about 0.3 mK. It is, however, highly anisotropic and its usefulness is limited to temperatures below 3 K because of its low

Curie constant ($C_\perp$ is about 0.011 K in SI units referred to unit volume) and the existence of an excited state which becomes significantly populated at higher temperatures [Rusby and Swenson (1980)]. Another rare-earth salt, neodymium ethylsulphate (NES), is less anisotropic and about four times stronger than CMN. It has been used successfully between 2 K and 16 K [Mangum and Bowers (1978)] and there appears to be no bar to using it down to 0.5 K or lower [Hudson (1972)].

Chromic methylammonium alum (CMA) is a salt of intermediate strength (C being about 0.077 K) which has been used for the range 1 to 20 K [Cetas and Swenson (1972); Durieux et al. (1962)]. Manganous ammonium sulphate (MAS) [Cetas and Swenson (1972); Cetas (1976); Durieux et al. (1962)], and gadolinium sulphate (GS) [Cetas (1976); Durieux et al. (1962)] are classed as strong, with C about 0.26 K and 0.79 K respectively, and they are therefore more suitable if the range is to extend to 30 K. Another possibility is gadolinium metaphosphate (GP) [Mangum and Bowers (1978)], which has the practical advantage of being anhydrous, but for all of these strong salts Δ and γ would have to be determined in the experiment using at least four fixed points, and the lower limit for accurate use might not be below 2 K.

7. 2 Technical Aspects of Magnetic Thermometry

For the simple case of a primary solenoid of m turns per metre and a secondary of turns-area product nA wound on it, the mutual inductance M is $\mu\mu_0 mnA$. If the coils contain a salt with a geometrical filling factor f, then $\Delta = (1 + f\chi)$ and if $\chi = C/T$ the sensitivity dlnM/dT is approximately - fC/T^2. For f = 0.5 and C = 0.26 K as for MAS, one therefore requires a precision $\Delta M/M$ of 2 parts in 10^7 if 1 mK is to be resolved at 25 K.

With a primary current i_p of angular frequency ω the solenoidal field H is Mi_p and the voltage across the secondary is $i_p\omega M$ or $\mu\mu_0 HnA\omega$. The design of the thermometer and bridge must be such that this is large enough to achieve the desired sensitivity. Typically $\mu_0 H$ might be 0.1 to 0.3 mT without departing seriously from the zero field limit, and the frequency is usually between 30 and 300 Hz. Taking 0.2 mT and 100 Hz and a secondary of 500 turns on a radius of 1 cm, the voltage would be 0.02 volt, 2 parts in 10^7 of which is 4 nV. This is not unreasonable, but there is need for caution and the design should aim to provide greater sensitivity than is indicated by such calculations. While the product nA can be gauged in advance, the frequency and field may be limited by the effect of eddy currents (roughly proportional to $\omega^2 H$) induced in nearby metallic components. To combat them, glass and plastic materials should be used where possible, and the coil system should be as well separated from the thermometer block as is practicable. Good heat transfer may be achieved through varnished copper wires, but tests of their effectiveness should be undertaken. Various other precautions could include [Cetas and Swenson (1972)] using a quadrupolar configuration of secondary coils, and shielding the coil system with superconductive Pb sheeting. The latter reduces external interference and ensures that eddy currents are independent of metallic resistivities and hence are independent of temperature.

As regards the measurement technique, it is standard practice to include in the cryostat a set of empty coils identical to those surrounding the salt, with the secondaries connected in opposition. Then the net mutual inductance is small and relatively insensitive to extraneous magnetic and electrical interference. Suitable mutual inductance bridges with adequate sensitivity can be assembled using commercially-available measuring equipment.

An essential preliminary to magnetic thermometry is a check of the performance of the bridge and coil system in the absence of the salt. After the system is cooled from room temperature, the bridge balance should first be checked for stability at a constant temperature for a period of hours during which time any dependence on primary current, coolant liquid level, exchange gas pressure, room temperature, or disturbance to the cryostat should be tested. Then the bridge balance should be monitored as a function of block temperature in order to establish the background effects at all currents to be used. If these effects are not small, steps should be taken to reduce them or at least to ensure their reproducibility from one assembling of the coils to another.

Only when the background has been satisfactorily characterized should a salt be mounted and magnetic thermometry begin. Again, the stability of the measuring system should be monitored after initial cooling. Bridge balance and thermometer resistance may then be measured at many temperatures in the range, with enough time allowed at each temperature to reach equilibrium. It is advisable to make measurements at more than one value of the primary current and to check for drifts in the bridge balance by returning to the starting temperature at the end of a measurement session. It may help the analysis if there is some overlap between data taken in different sessions, particularly after an overnight interval or a transfer of liquid helium or nitrogen. After correction for bridge drifts and background effects, the unknown constants are evaluated from the measurements taken at the chosen reference temperatures, and values of T_{90} at intermediate temperatures are calculated.