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ON SAMPLE POSITIONING AT LOW TEMPERATURES

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ABSTRACT

A method is presented to measure the position of a small solid sample which is suspended by thin copper wires, from a microbalance, in a dewar that has been described earlier [1]. The shrinkage of the wires at low temperatures is reproducible to within ± 0.15 mm.

INTRODUCTION

An apparatus for the measurement of static magnetic susceptibilities on small solid samples, following the Faraday method, has been described previously [2]. The striking part of the set-up is a vacuum microbalance with a sensitivity of about 10^{-4} dyn. Down from this balance hangs the sample holder, on thin wires, in a Dewar between the pole pieces of an electro-magnet. The sample temperature can take any value between 3 and 300 K. A data acquisition and processing system has been adapted to the apparatus [3]. The processing system, performing the calculations on a computer, allows the investigator to spend more time to the measurements. It results in a larger number of susceptibility values, yielding better statistics and a better precision on the measurements. To make the most of the increased precision a more frequent adjustment of the sample position is required.

EXPERIMENTAL

The sample position is defined, just by laying it down on the sample holder, a pan. This pan, however, is often brought out of its aimed position during the removal of very small samples from it. An accidental touch of the suspension bends the wires and consequently alters the position of the holder. The wires plastically deform as they are made of copper. Nevertheless, copper is still used because 1) its magnetic susceptibility is low which property keeps the background corrections small; and 2) its electric conductivity enables one to reduce parasitic electrostatic effects. Thick wires are too stiff. The 48 mg sample-pan is not heavy enough to pull the wires straight downwards and neither is it easy to strech them whilst hanging on the balance. Copper wires of a diameter 0.02 mm were breaking too often. The 0.07 mm compromise was strong enough to last now for more than six years. The wires are soft enough to be straightened by a slight pressure on the pan. The force results in a slight elongation of the wires. To bring the sample in its former position relatively to the electro-magnet the magnet must be lowered. To facilitate this adjustment the magnet has been mounted on an hydraulic system which enables one to move it up or down along the z-axis, to the level for which the bottom of the pan is just pointed by the cross of an optical viewer, fixed to the electro-magnet [2]. The adjustment of the height is carried out whilst the metallic Dewar is removed, so that the sample pan can be seen. After the adjustment the magnet is rolled off, the Dewar is mounted and the magnet is rolled back again in its measuring position. The reproducibility of the magnet setting ensures the sample to hang versus the magnet in nearly identical positions for all measurements at room temperature (RT). At the lower temperatures, however, a systematic deviation out of the RT work coordinates is revealed. It is due to the shrinking of the suspension wires at the cooling. As cooling always occurs in the Dewar, which is a metallic one, the position of the sample pan at low temperatures cannot be seen without additional instrumentation. To use instruments which were at hand in the laboratory, the balance has been taken as null detector at the positioning of a copper coil, outside the Dewar, versus a small iron sample, 9.47 mg, that lies on the balance pan.

The balance detects the z-component of the force which can be expressed by: $F_z = M.(dH/dz)$ (1) , wherein M is the magnetic dipole moment of the sample and dH/dz is the gradient of the magnetic field. For the measurement of the shrinkage of the suspension wires, the large electro-magnet is rolled off. The magnetic field and its gradient at the sample place then is generated by a small 250 turns-coil. The coil-system is drawn in Figure 1. The current is fed by a constant current-source and was 0.603 A in this experiment. In agreement with the expectation from the field gradient behaviour and equation (1), a coil position is found for which the z-component of the force is zero and about which position the force changes practically linearly with P. The proportionality constant is 2.5×10^{-2} dynes/mm. Because the precision on a single force measurement is about 2.5×10^{-4} dyn the resolution in the displacement detection along the z-axis, in the considered P region, is of the order of 0.01 mm.

RESULTS AND DISCUSSION

The experiments, for measuring the shrinkage of the suspension wires, have been started at room temperature. At this temperature the zero field gradient was measured to be at P = 3.1 mm. The precision on the initial value of P is only 0.1 mm. Nevertheless, when P was changed only by screwing up or down the coil-holder, the reproducibility of the zero setting was $\leq$ 0.02 mm. After locating the origin, the sample has been brought at different temperatures. In one of the runs the outer Dewar-vessel was filled with liquid nitrogen which then was pumped to decrease the temperature below 63 K. The inner vessel was filled with liquid helium at about normal pressure. The sample temperatures

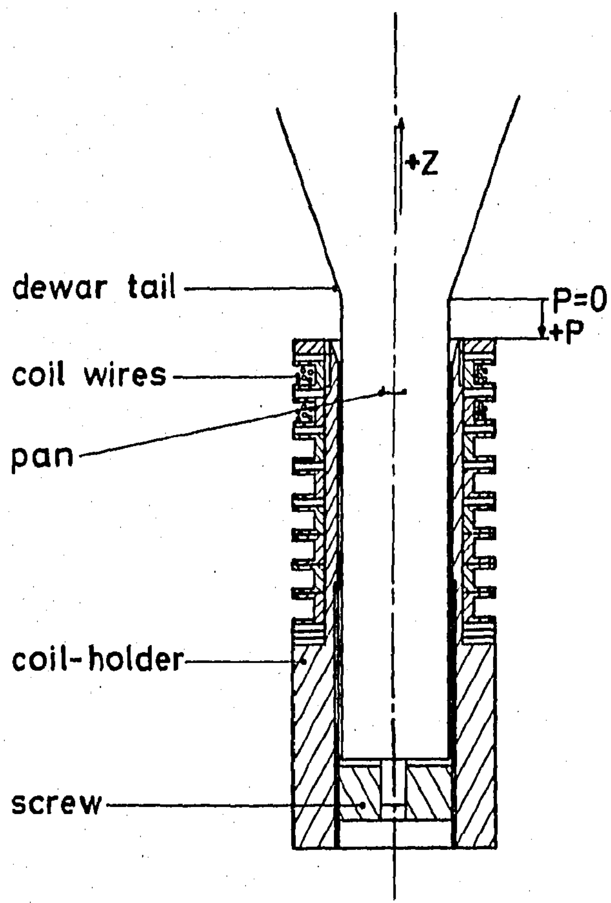

Fig. 1. The copper-coil/sample-pan situation.

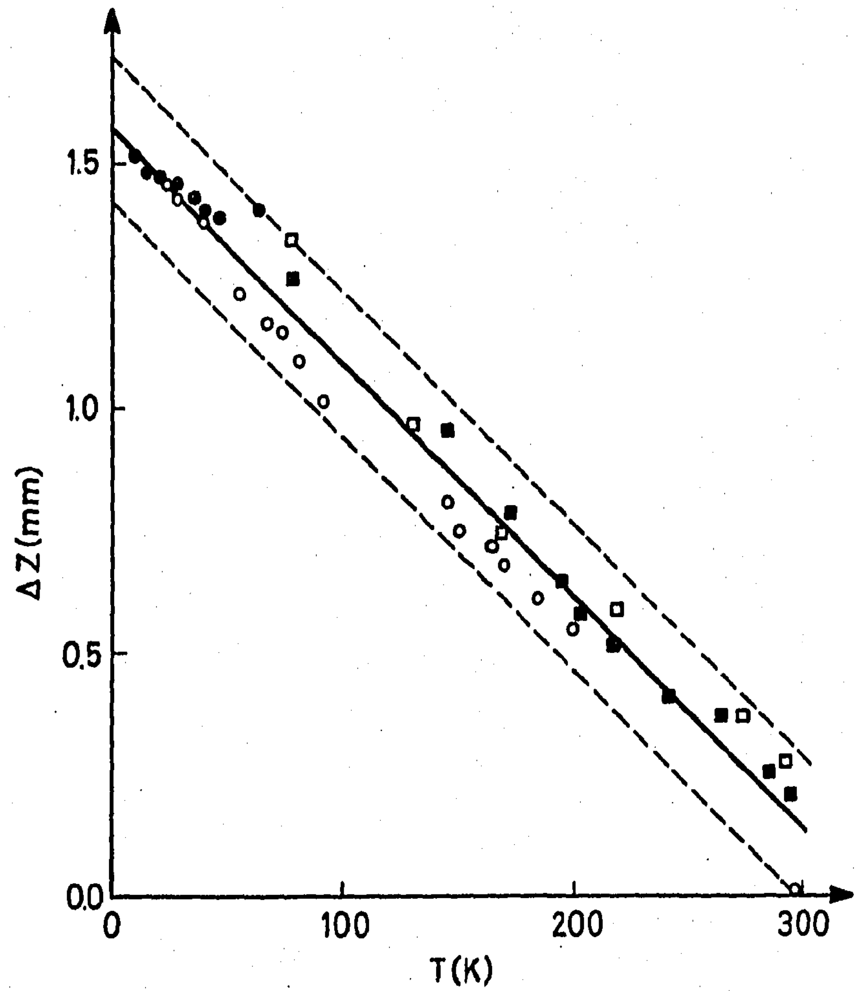

Fig. 2. The suspension wires shrinkage versus sample temperature.

were set by choosing the amount of evaporated helium gas to flow over the heat exchanger of the sample tube in the Dewar [1]. The measured changes in height of the sample position for this case are given in Figure 2 by the closed circles. The open circles are related to the measurements carried out during the warming up of the sample tube after all nitrogen and helium was boiled off from the Dewar vessels. The higher the temperature the slower the warming up so that the temperature region between 140 and 200 K had to be measured the day after the run started. Another run has been measured with liquid nitrogen in the inner vessel and heating the sample electrically with the power just needed for the aimed temperature. The results are given by the open squares. The closed squares are related to a similar experiment but for a fast heating in which the power used was about twice that needed to reach room temperature in the former run. In the latter run the power is cut off when the temperature was about the aimed one. Then ten minutes were waited before carrying out the measurements, this in order to allow the temperatures to become relatively in equilibrium.

The shrinkage value Δz can be expressed by a linear equation of the measured temperature, when deviations of ± 0.15 mm are tolerated.

CONCLUDING REMARKS

The shrinkage study, which lasted two weeks, learned that, when the balance is kept continuously in equilibrium and the susceptibility apparatus is not disturbed, the changes in height of the sample at low temperatures are reproducible to within ± 0.15 mm; even when the temperatures are reached by different cooling procedures.

The linear equation, which expresses the shrinkage, is introduced in the data processing computer programme and helps for choosing the right field and field-gradient values at the susceptibility calculations.

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