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AMORPHOUS $Zr_{0.7}Pd_{0.3}$ AS A TEMPERATURE REFERENCE NEAR 2.5 K

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ABSTRACT

The superconductive transition temperature of the high solidification rate material $Zr_{0.7}Pd_{0.3}$ was used as a temperature reference to estimate the temperature deviation between the thermometer and the sample in the pan of a vacuum microbalance with 10^{-9} N sensitivity.

The latter system belongs to a set-up for measuring magnetic susceptibilities at low temperatures down to 2.2 K. The transition temperature of the amorphous $Zr_{0.7}Pd_{0.3}$ sample was calibrated in a separate helium bath in good contact with the thermometer. In this bath the lambda transition point of liquid helium offered an additional check on the thermometer.

INTRODUCTION

In the existing set-up (ref.1) for measuring static magnetic susceptibilities by the Faraday method the temperature in the region below 4.2 K is controlled by a flow of helium gas under reduced pressure. This gas cools the top of the sample room containing stagnant helium gas. The gas, at about 13 Pa, takes care of the heat exchange, homogenizing the temperature of the room in which the thermometer is located at the bottom. In the same room the sample is put on the pan that hangs down from the balance. The sample room is located in the dewar tail either between the pole shoes of an electromagnet with horizontal field or on the vertical axis of an iron-free solenoid.

A practical difficulty which is encountered with this experiment has to do with the temperature measurement. The thermosensor has to be mounted at about 2 cm away from the sample. Near the lower temperature limit a small temperature deviation might exist between the temperature of the thermometer and that of the sample.

The present paper deals with the measurement of this temperature difference.

DESCRIPTION OF THE CALIBRATING MATERIAL

There are few suitable temperature reference materials close to but above 2.2 K. A ribbon of amorphous $Zr_{0.7}Pd_{0.3}$ alloy with a cross-section of 1.3 mm by 35 µm was kindly made available to our institute by ORNL (USA). This substance is obtained by rapid quenching and has a superconducting transition near 2.5 K. Considerable scatter exists in the literature data on this transition point

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(ref.2). The data range from 2.53 K to 2.66 K. As it may depend on the detailed local structure each sample will have to be calibrated separately in an environment where the temperature may be controlled with great accuracy.

Four samples were used altogether: S1 and S2 served for preliminary tests while S3 is the sample used to perform the calibration. S4 is to be stored as a permanent temperature standard.

CALIBRATION OF THE TRANSITION TEMPERATURE

In a helium bath both the sample and the calibrated thermometer are fixed on a common copper cylinder (ϕ 34 x h 59 mm) ensuring good thermal contact but preserving electrical insulation. The transition temperature is determined resistometrically by the four-wire method at a current of 10 mA. The first series of runs were performed with the sample S1 which is a fairly long strip. The distance between the potential contacts was 79 mm out of a total length of 86 mm. At 4.2 K the resistance was 3.7 ohms.

By reducing the helium pressure above the bath the temperature is gradually lowered, followed by a warm-up period. This cycle was repeated several times in order to test the reproducibility. The resistance R, expressed in ohms per mm of length, is seen to drop to zero at a well-defined temperature (Fig. 1). It was shown that this temperature is not affected by the value of the current by lowering the current of 1 mA .

The thermometer was checked at the lambda transition point of helium which was reached in this series of measurements at a reading of 2.1780 ± 0.0006 K. The lambda point is easily observed because of the peak in the specific heat.

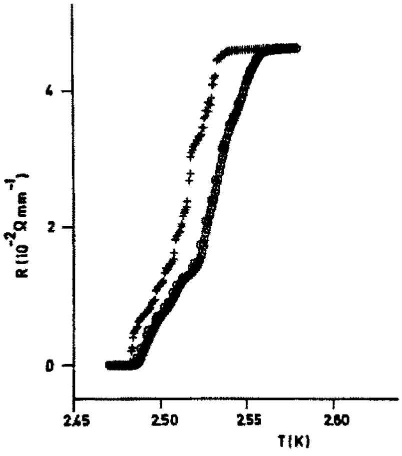

Fig. 1. Resistance per mm versus temperature in sample S1 at 10 mA (+) and 1 mA (o).

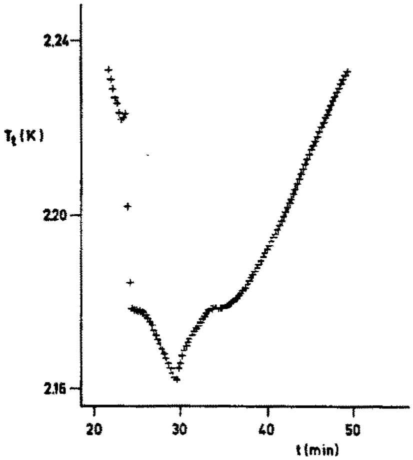

Fig. 2. Time dependence of the temperature near the lambda point of helium.

The cool-down and warm-up curves show a temporary stabilization of the temperature versus time at the onset of the transition (Fig. 2).

The superconductive critical temperature of the sample was T_c = 2.48 K.

Some structure is evident in the resistivity curve which points to local heterogeneities. The reproducibility was very good in all cases which proves that these effects are real.

These tests established the feasibility of the experiment. The intercalibration was then performed in a second series of runs with the sample S3 for which the measurement of the susceptibility has already been carried out (see next paragraph). The latter measurement has to precede the present one in time because the potential contacts cannot be removed without risk of damaging the material.

With a current of 10 mA the critical temperature was found to be T_c = 2.512 K .

Fig. 3 shows that in this curve there is less structure and also that the two samples are clearly different.

MICROBALANCE MEASUREMENTS

The vertical force per unit weight on a sample is (SI units):

$F = m\chi B \text{ grad } B/\mu_0 \quad N$.

The optimum position of the sample is near the maximum of the distribution curve of the magnetic field times the gradient. For the iron-free coil this was investigated in detail in a previous publication (ref.3).

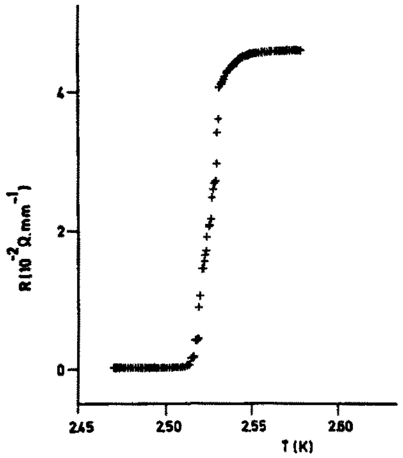

Fig. 3. Resistance per mm versus temperature in the intercalibrating sample S3 at 10 mA.

The determination of the transition temperature follows by applying the same technique. With the magnet current in chopped mode the temperature is slowly cycled through the superconductive transition point.

Preliminary tests were performed with the small sample S2 which is about 5 mm long and weighs 1.06 mg. They indicated the need for a larger amount of material. Nevertheless it was possible to measure the magnetic susceptibility and to check that the alloy is weakly paramagnetic. In the high field (2.06 T) of the large electromagnet at 47.1 $T^2.m^{-1}$ a value of $\chi = 1.19 \times 10^{-8}\ m^3.kg^{-1}$ was obtained which is almost independent of the temperature.

After this test the main measurement started with the sample S3 which was 24 mm long and weighed 7.29 mg. It was rolled into a small spiral which fitted inside a box of Au/Cu alloy. This box is routinely used to cover samples on the microbalance pan.

At 900 mA through the iron-free solenoid the magnetic induction is equal to 9 mT and the induction times the gradient amounts to $3.73 \times 10^{-8}\ T^2.m^{-1}$. Above the transition temperature the susceptibility of the sample, pan and cover box are too low to show up in the signal. Below the transition temperature the superconductor behaves as a diamagnetic material as persistent currents are induced when the magnetic field is switched on. Near the transition but below it the force is observed to be roughly proportional to the temperature difference. By extrapolation to zero force of the results obtained at 900 mA the resulting critical temperatures were finally derived for four runs (Fig. 4), leading to an average value and a quadratical error equal to

$T_{tc} = 2.5650 \pm 0.0023$ K .

as read on the thermometer.

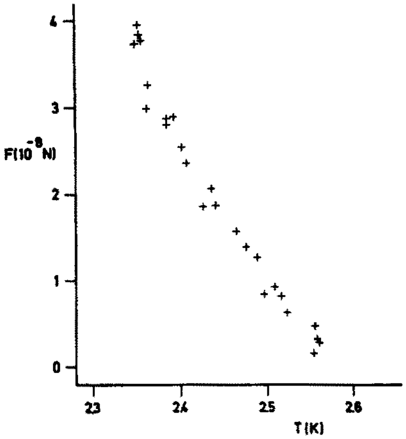

Fig. 4. Vertical force below superconductive transition temperature in S3.

A problem arises here as this temperature is a weak function of the applied magnetic field. The field cannot be reduced below a certain limit imposed by the sensitivity of the microbalance (10^{-9} N). The correction is rather small and was taken from literature (ref.4). The decrease of the critical temperature per unit of temperature is the inverse of

$dB/dT_c = - 2.4\ K^{-1}$.

The reading at zero field would therefore be

$T_{tc} = 2.5650 + 0.009/2.4 = 2.569$ K .

With the aim of creating a permanent temperature standard another sample S4 weighing 7.04 mg was cut out of the same ribbon. No potential contacts will be soldered to this sample. The critical temperature at 9 mT was found to be (Fig. 5) $T_{tc} = 2.56$ K .

TEMPERATURE DEVIATION

The measurements performed on the sample S3 established the link between the critical temperature determined in the helium bath $T_c = 2.512$ K and the reading of the thermometer near the microbalance (corrected for the shift caused by the magnetic field) $T_{tc} = 2.569$ K. The correction to be applied to the thermometer reading near 2.5 K is

$\Delta T = T_c - T_{tc} = - 0.053$ K .

CONCLUSION

The correction to the thermometer reading at 2.5 K is only 2 % but the fact that it has been determined in this experiment will add to the confidence with which the temperatures in the lower range can be established.

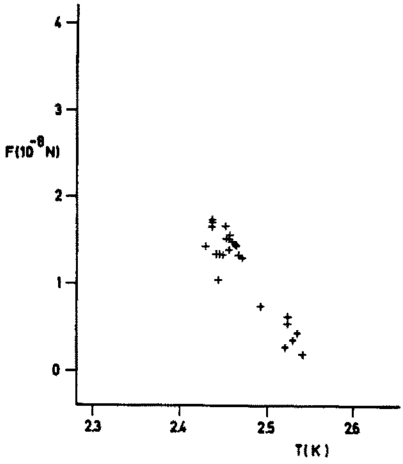

Fig. 5. Vertical force below superconductive transition temperature in S4.

A similar calibration existed up to now only with thallium, a very soft material, which oxidizes readily in the laboratory and which cannot be handled easily. The amorphous $Zr_{0.7}Pd_{0.3}$ alloy has been kept in the laboratory for about one year and no corrosion has been seen. The low signal strength however means that it can only be used with a very sensitive system.

REFERENCES

1 A. Van den Bosch and J. Vansummeren, Thermochimica Acta, 82 (1984) 51-62.
2 K. Kai, M. Ikebe, K. Suzuki and T. Nomoto, J. Less-Common Metals, 89 (1983) 229-235.
3 J. Vansummeren and A. Van den Bosch, Progr. in Vacuum Microbalance Techn., 2 (1973) 147-155.
4 S.J. Poon, Phys. Rev., B25 (1982) 1977-1980.