SRD1000: A NEW SUPERCONDUCTING REFERENCE DEVICE FOR THERMOMETRY BELOW 1000 mK

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ABSTRACT

As part of a European project for the development and realization of a temperature scale below 0.65 K, research has been performed to realize a new superconducting reference device for thermometry below 1000 mK, the SRD1000. This device uses the superconducting transition temperatures of various materials to establish fixed points on the temperature scale. The research consisted of selecting, producing and testing suitable sample materials to be used for the new sensor. As compared to the former SRM 768 device (NBS, U.S.A.), which is not any longer available, the poisonous Be has been omitted and IrRh alloys are introduced to obtain additional fixed points between 15 and 100 mK. At the high temperature end Zn and Al are used to provide an overlap with ITS90. Apart from the research on materials a new detection system for the superconducting transitions was developed and tested, consisting of a planar micro coil system and dedicated electronics. In 1999 we will construct two prototype devices containing the materials W, IrRh alloys, Ir, AuAl2, AuIn2, Cd, Zn and Al. These devices will be tested and calibrated against a 3He melting curve thermometer in the temperature range of 15 mK to 1000 mK. Results of preliminary measurements are presented.

1. INTRODUCTION

At present a European project is carried out on a temperature scale below 0.65 K, the lowest point of the current International Temperature Scale ITS-90. This will result in a proposition for an international temperature scale between 1 mK and 1 K. The prime scale carrier will be the equilibrium melting pressure of 3He. Anticipating the new scale, research has been carried out on a superconducting reference device for transferring the scale in a convenient way via a secondary thermometer to its users. This device uses the normal to superconducting transition temperatures of various materials to establish fixed points along the temperature scale. The goal of the present work is to construct and characterize two prototype devices, each containing at least 9 reference materials. The superconducting transitions are detected with a planar micro coil system and dedicated electronics. Three partners contribute in this research: The NMi Van Swinden Laboratorium (NMi), Hightech Development Leiden (HDL), and the Kamerlingh Onnes Laboratorium (KOL). The calibration of the prototypes will be performed against the NMi realization of the future ultra low temperature scale, as described in ref. [1].

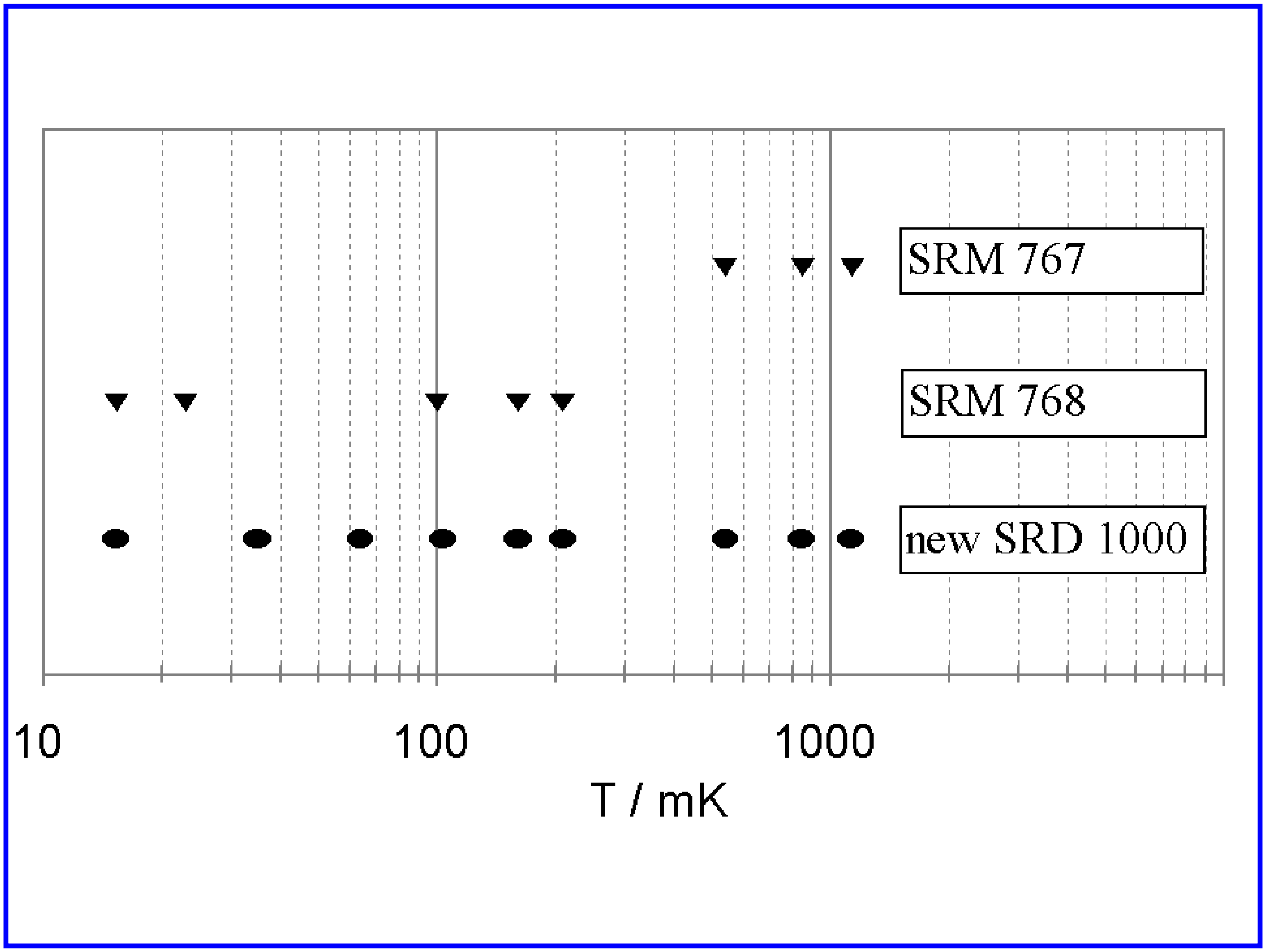
2. THE NEW SUPERCONDUCTING REFERENCE DEVICE

Because of the positive experience with the former SRM 767 [2] and SRM 768 [3] devices, we tried to benefit as much as possible from the experience obtained by NIST, the former NBS, in selecting reference materials for the new device. It was decided that at least the same materials in the temperature interval 15 mK to 1.13 K would be used, except for the poisonous beryllium with normal to superconducting transition temperature Tc 23 mK. Additionally research was carried out to find two or three suitable reference materials to fill in the gap between the tungsten (Tc 15.3 mK) and iridium (Tc 110 mK) transition temperatures. Although there are no elements known to have a Tc between 25 mK and 100 mK, e.g. IrRh alloys show a Tc that depends upon the composition. Above 500 mK, three materials have been selected, of which zinc and aluminum provide an overlap with the ITS-90. Because the upper limit of the melting curve thermometer is 1 K, the aluminum point (Tc 1.13 K) will be calibrated against a RhFe thermometer. Table 1 shows the reference materials selected for use in the new fixed point device. The distribution of fixed points of the former SRM 767 and SRM 768 and of the new SRD1000 over the temperature scale is shown in fig. 1.

Table 1: Selected reference materials for the SRD1000

Material	W	IrRh alloys	Ir	AuAl$_2$	AuIn$_2$	Cd	Zn	Al
T_c / mK	15.34	20 - 100	110	161	204	540	850	1134

Figure 1: The SRD1000 fixed points compared to the SRM 767 and SRM 768



3. THE USE OF SUPERCONDUCTING TRANSITIONS AS THERMOMETERIC FIXED POINTS.

Because there is no latent heat involved in the superconducting phase transition, the temperature has to be controlled externally to realize the fixed point. The difficulty however is that the transition always occurs over a finite temperature interval. This transition width is an important parameter and determines how accurately the fixed point, defined as the temperature halfway the transition, can be realized.
Another important parameter is the reproducibility of the transition. Experience with the SRM 768 has shown that with high quality materials reproducibility of the order of a few tenths of a mK can be reached.
The superconducting transition is also dependant on the magnetic field present. Typical values for this field dependance dTc/dB are 0.1 mK/mT. So even the earths magnetic field of about 50 mT can shift the transitions by as much as 5 mK. To reduce this effect to a maximum acceptable 0.05 mK the earths field has to be shielded by at least a factor of 100.

4. THE SRD1000 SENSOR

The technique used to detect the transitions is based on the familiar principle of exclusion of magnetic flux in the superconducting state. One of the new features of the SRD1000 is that the transition is detected by planar micro coil system, in stead of by conventional coils. The planar coil system is realized by a thin film niobium structure on a silicon substrate. As compared to conventional coils the geometry of planar coils provides a much higher coil-sample material filling factor. To reduce the magnetic field present at the location of the materials, magnetic shielding is required. The sensor is contained in a cylinder made from Cryoperm 10 [4]. After a special heat treatment the material has a magnetic permeability of over 10000 at 4 K resulting in a calculated magnetic field attenuation of over 100. The low residual magnetic field is trapped during cool down using a superconducting niobium cylinder located inside the Cryoperm 10 shield.

5. REFERENCE MATERIALS

Although most of the selected materials have already been used for thermometric fixed points before, considerable effort was required to obtain reliable and reproducible results. The reason is that there are a number of difficulties involved in the production of the materials:

- The impurity content of the materials is very important. For some materials shifts of Tc as high as 1 mK per ppm impurity have been found. The shift is expected to be even bigger for magnetic impurities.
- For some materials the crystal structure is important. For example AuIn2 and AuAl2 need to be single crystal to obtain sharp and well-defined transitions.
- Internal stresses can also influence the transition width and reproducibility. Therefore some materials need to be annealed before inclusion in the sensor.

The dependence of Tc upon material parameters like impurity, crystal structure and stress make it impossible to prepare samples that show identical transition temperatures within the accuracy of the measurement, even when each specimen is prepared from the same bulk material. Therefore each device will have to be calibrated individually.

6. NEW MATERIALS

In the search for new fixed points between 25 mK and 100 mK two types of materials were investigated. First alloys of molybdenum and niobium were investigated, based on earlier work by R.A. Hein *et al.* [5]. Unfortunately all our sample materials showed transitions not suitable for the realization of fixed points. Subsequently alloys of iridium and rhodium were investigated, using the results of Mota *et al.* [6]. He found transition temperatures between 5 mK and 100 mK and widths of about 1 mK for such alloys, which makes them very promising for fixed points in the desired temperature interval.

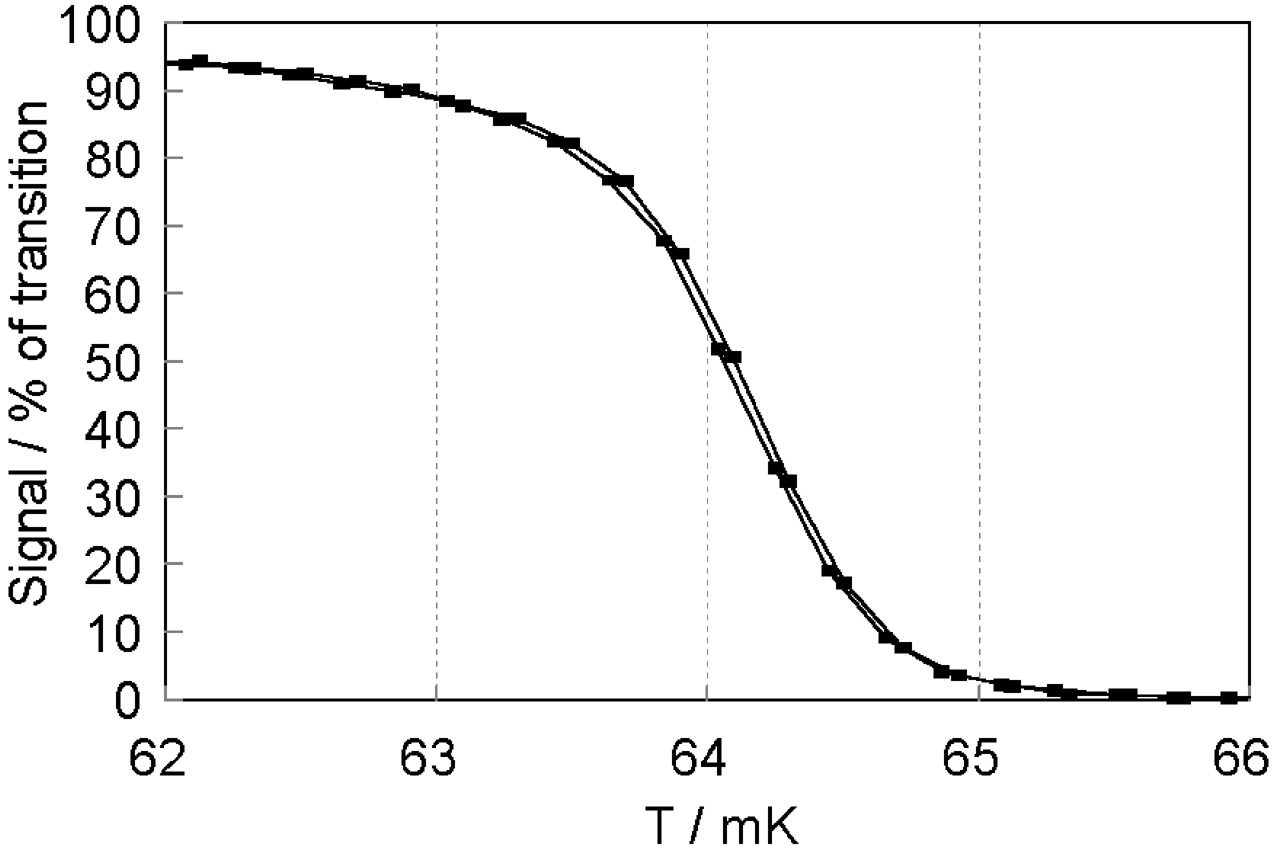
7. RESULTS

About 40 samples of various materials have been prepared. The normal to superconducting transitions were analyzed to determine the transition temperature Tc and the transition width. Tc is defined as the temperature halfway the transition and the width is defined as the temperature interval in which the central 80% of the transition occurs. Of each sample material the best results obtained up to now are presented in table 2. To show the suitability of the new IrRh fixed points, a full transition of Ir92Rh08 is shown in fig. 2. It is estimated that the transition temperature can be determined within a few tenths of a mK. The transition was measured using a prototype of the SRD sensor combined with dedicated electronics.

Table 2: Results

material	**T_c / mK**	**Width / mK**
W	*transition not yet observed with the present prototype*	
$Ir_{80}Rh_{20}$	35	1
$Ir_{92}Rh_{08}$	64	2
Ir	104	1.5
$AuAl_2$	161	0.3
$AuIn_2$	204	1.9
Cd	540	7
Zn	850	4
Al	1134	10

Figure 2: The normal to superconducting transition of $Ir_{92}Rh_{08}$ at about 64 mK



8. CONCLUSIONS

Research has been carried out to realize a superconducting reference device for transferring the temperature scale between 10 mK and 1 K in a convenient way to its users. At least 9 materials were selected to be used as superconducting fixed points. Compared to the SRM 768 device, the new device offers at least two new reference points at about 35 mK and 64 mK in stead of the beryllium point at about 23 mK. Furthermore Cd, Zn and Al are present to support the high temperature end of the scale. For all selected materials (W, IrRh alloys, Ir, AuAl2, AuIn2, Cd, Zn and Al) except tungsten high quality transitions have been obtained. Still additional work will be performed to improve and characterize additional prototypes.

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