STATUS REPORT ON THE DEVELOPMENT OF A SUPERCONDUCTING REFERENCE DEVICE FOR PRECISION THERMOMETRY BELOW 1 K

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ABSTRACT

A Superconducting Reference Device (SRD1000), including 10 to 12 reference points in the temperature range 10 mK - 1 K and dedicated measurement electronics, is being developed to provide a direct and accurate traceability to the new Provisional Low Temperature Scale (PLTS-2000). In this paper we describe the preparation and the analysis of bulk samples of Ir_xRh_{100-x} alloys (showing transition temperatures T_c between 15 mK and 100 mK), single crystals of $AuIn_2$ (T_c = 160 mK), $AuAl_2$ (T_c = 208 mK) and Zn (T_c = 850 mK). The use of Mo/Au and Ti/Au bilayers as reference materials is proposed. Results of low temperature acceptance tests on best samples obtained are presented. The acceptance tests were performed in order to select the reference materials that are to be included in future pilot series SRD devices.

1. INTRODUCTION

A strong need for accurate temperature measurements in the millikelvin range has been felt in recent years from academic and industrial research laboratories. As a result of the commercialization of ^{3}He-^{4}He dilution refrigerators, temperatures between 1 mK and 1 K are easily accessible to many users while traceable thermometry of such temperatures is generally limited to national metrology institutes. Last year a provisional extension of the ITS-90 [1] down to 0.9 mK, based on the ^{3}He melting pressure curve, was officially adopted (PLTS-2000, [2]). The practical realization of this new scale implies the use of a ^{3}He Melting Curve Thermometer (MCT, [3]), which is time consuming and very expensive to set up.

The National Institute of Standards and Technology (NIST, USA) has produced two different superconducting fixed-point devices (SRM 767 [4] and SRM 768 [5]). These devices are no longer available. Metrological investigations on high-purity superconductors [6] showed that highly reproducible superconducting reference points for precision thermometry can be realized with high-purity metals by applying appropriate methods for sample preparation and characterization.

Within an EU project Ultra-Low Temperature Scale Dissemination, a Dutch consortium of research institutes and industries is developing a new superconducting reference device (SRD1000) to provide a direct and accurate traceability to the PLTS-2000. The SRD1000 will include 10 to 12 reference points in the temperature range 10 mK – 1 K and dedicated measurement electronics. This new device combines the past experience in NIST reference samples with recent results on new materials, leading to the following selection of reference materials for the SRD1000:

- W (T_c = 15 mK), Ir (T_c = 100 mK), $AuAl_2$ (T_c = 160 mK) and $AuIn_2$ (T_c = 208 mK), as in the SRM 768;

- Ir_xRh_{100-x} alloys (x = 92, 80 and 73) prepared by Leiden Instituut voor Onderzoek in de Natuurkunde (LION), which have tunable transition temperatures between 20 and 100 mK depending on the relative concentration of the two components [7];
- Mo/Au and Ti/Au bilayers [8] to be developed by University of Twente (UT), which provide adjustable transition temperatures between 100 and 600 mK by changing the thicknesses of the two layers;
- Cd (T_c = 520 mK), Zn (T_c = 850 mK) and Al (T_c = 1180 mK), as in SRM 767.

The detection of the superconducting phase transitions is based on the traditional magnetic-induction technique but a novel planar microcoil system allows the miniaturisation of the device. Dedicated external electronics was developed by Hightech Development Leiden (HDL) for monitoring the superconducting transitions. In this paper we report the preparation techniques of the samples adopted by LION and UT, and the results of the low temperature acceptance tests performed by Nederlands Meetinstituut (NMi) for selection of the reference materials to be included in a future pilot series of SRD1000 devices.

2. SAMPLES PREPARATION AND ANALYSIS

At LION two batches of polycrystalline samples of Ir_xRh_{100-x} (x = 92, 80 and 73) were synthesized by arc-melting the constituent elements in the desired ratio on a water-cooled hearth under high-purity argon atmosphere. Iridium and Rhodium can form a homogeneous alloy at all ratios, with a melting temperature between 2719 K (for Ir) and 2236 K (for Rh). The solid has the face-centered cubic structure. To ensure homogeneity, the alloy buttons were remelted several times. One batch was prepared by homogeneously mixing the powders of Ir and Rh before melting together. In the second batch, the Ir and the Rh powder were first melted separately into shot, before melting the alloys together in the desired ratio. Both batches of arc melted samples were annealed in the arc furnace during several hours at an unknown but rather high temperature (about 1500 °C). Analysis with electron-probe microanalysis (EPMA) has shown that the alloys have the desired stoichiometry and are indeed homogeneous, within the 2% resolution.

A single crystal of $AuAl_2$ was prepared by using a modified Czochralski tri-arc technique [9]. $AuIn_2$ single crystal was grown from the melt in vacuum by the recrystallization method from a polycrystalline compound, in a vertical furnace with a temperature gradient. The crystallographic structure and the lattice parameters of these single cristals were confirmed with powder X-ray diffraction. The quality of the single crystals was inspected by means of X-ray backscattering in the Laue geometry. The reflections were recorded on photographic film and compared with the expected crystal structure and orientation. Microprobe analysis showed that the stoichiometry was good and no metal was phase-separated out. Also Zn crystals, prepared by recrystallization from the melt from high-purity shot, were shown by X-ray analysis to be perfect single crystals.

University of Twente (UT) is developing bilayers consisting of a superconductor and a normal metal. Because of the proximity effect, the transition temperature of such bilayers can be tuned in the temperature range between 100 and 600 mK by varying the relative thicknesses of the two layers. All possible material combinations were studied that can provide transitions in the mentioned temperature range. Mo/Au and Ti/Au bilayers [8] were selected on basis of the long-term stability and the sharpness of the transition. At present several Mo/Au and Ti/Au bilayers are being fabricated using the Pulsed Laser Deposition (PLD) technique. The temperature of the substrate is one of the crucial parameters and the final temperature will be chosen such that a monocrystalline layer is obtained [10]. Based on the proximity model the transition temperature has been calculated as a function of the relative thickness of the layers for several materials combinations. The obtained results are used for choosing the appropriate layer thicknesses according to the desired transition temperature.

3. EXPERIMENTAL SET-UP

Several runs were performed at NMi ultra-low temperature facility to provide acceptance tests on the samples prepared at LION to be selected as reference materials for a future pilot series of SRD devices.

A gold-plated OHFC copper comparator block was attached to the mixing chamber stage of a ^{3}He-^{4}He dilution refrigerator (Oxford Instruments, Kelvinox400). A MCT cell, a SRM 768 NIST device, up to three SRD test devices (depending on the run), a manganine wire heater and a RuO_2 thick film resistor thermometer were mounted on the comparator block. An isothermal radiation shield surrounded the comparator block. The temperature of the comparator block was controlled by a PID temperature controller that used the RuO_2 thermometer as a sensor and the manganine wire heater as an actuator [11].

The initial filling pressure of ^{3}He in the MCT cell was about 3.54 MPa and it allowed us to operate the MCT thermometer from the minimum temperature up to 800 mK. The minimum temperature reached was 15 mK. As a consequence we could not observe the transitions of the W samples.

The superconducting transitions of the samples included in the SRM 768 and in the SRD test devices were monitored using a dedicated mutual inductance detection system developed by HDL.

4. RESULTS

Table 1: Overview of the proposed PLTS-2000 reference temperatures and best results obtained thus far for the superconducting transitions of various metal samples.

Proposed reference temperature [mK]	**Selected metal sample**	**Sample specification**	**Observed T_c [mK]**	**Observed W [mK]**	**Reproducibility T_c [%]**
1180	Al	4N7 Al foil [1)]	1164	8.0	0.09
850	Zn	melt 6N Zn shot [2)]	850.3	3.0	0.05
520	Cd	5N Cd foil [1)]	529.3	3.5	0.08
250 – 300	MoAu bilayer	to be made	-	-	-
208	$AuIn_2$	melt 5N4 Au, 6N In powder [1)]	208	0.4	0.03
160	$AuAl_2$	melt 5N4 Au, 6N Al powder [1)]	161.3	0.2	0.02
100	Ir	melt 4N5 Ir powder [1)]	99.8	0.5	0.06
60	$Ir_{92}Rh_{08}$	melt 4N5 Ir, 4N Rh powder [1)]	61.3	0.5	0.1
35	$Ir_{80}Rh_{20}$	melt 4N5 Ir, 4N Rh powder [1)]	34.0	0.9	0.3
22	$Ir_{73}Rh_{27}$	melt 4N5 Ir, 4N Rh powder [1)]	[4)]	[4)]	[4)]
15	W	3N5 foil [1)] or 4N 1-xtal [3)]	[4)]	[4)]	[4)]

[1)]Alfa Aesar, Germany, [2)] Cominco American, [3)] Goodfellow, UK, [4)] the transition was not observed down to about 15 mK.

Table 1 gives an overview of the reference points a materials selected for the SRD1000. Samples of various qualities and shapes were prepared. Superconducting transition temperatures T_c and width of the transitions W were measured. The best results obtained thus far are summarised in the table.

The reproducibiity of a transition is estimated to be 12.5% of the transition width. For the transitions above 50 mK this reproducibility is less than 0.1 % of the temperature.

The Figures 1 and 2 show examples of the transitions of respectively $AuIn_2$, $AuAl_2$, Ir and $Ir_{80}Rh_{20}$.

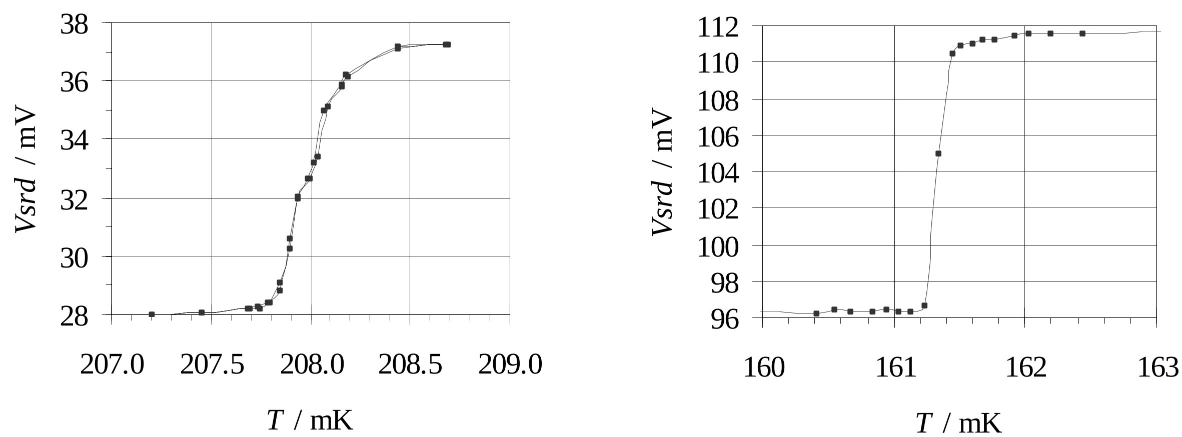

Figure 1. Transitions of respectively $AuIn_2$ (T_c = 208.0 mK and W = 0.4 mK) and $AuAl_2$ (T_c = 161.3 mK and W = 0.2 mK).

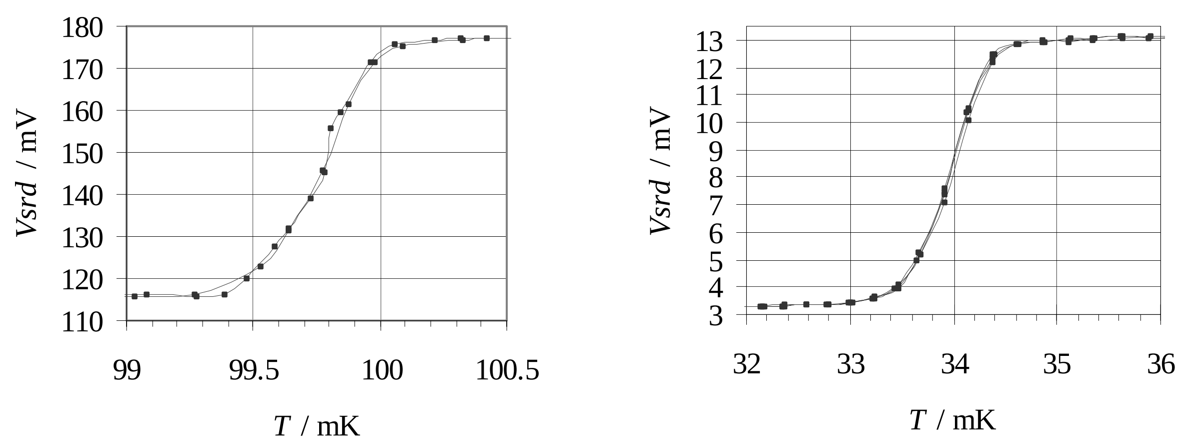

Figure 2. Transitions of respectively Ir (T_c = 99.8 mK and W = 0.2 mK) and $Ir_{80}Rh_{20}$ (T_c = 34.0 mK and W = 0.9 mK).

5. CONCLUSIONS AND FUTURE DEVELOPMENTS

Measurements on samples of $Ir_{92}Rh_{08}$, Ir, $AuIn_2$, $AuAl_2$, Cd, Zn and Al have shown that the base materials selected and preparation techniques developed allow us to realize reference temperatures of sufficient reproducibility to support the PLTS-2000 above 50 mK. Work still continues to produce and test bilayers of Mo/Au and Ti/Au. For the proposed materials providing reference temperatures below 50 mK preparation techniques are being improved and new samples will be tested. Finally a pilot series of SRD1000 devices and measurement electronics will be developed and distributed to various European metrological institutes and industrial partners, which will evaluate this series to determine its suitability as transfer standard for the PLTS-2000.

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