SRD1000 Pilot Series For Transferring The PLTS-2000

W.A. Bosch^{+*}, J. Engert[#], J.J.M. van der Hark^{*}, X.Z. Liu^{*}, and R. Jochemsen^{*}

⁺HDL, P.O. Box 691, 2300 AR Leiden, The Netherlands

^{*}Kamerlingh Onnes Laboratory, LION, Leiden University, P.O. Box 9504, 2300 RA Leiden, The Netherlands [#]Physikalisch Technische Bundesanstalt, Abbestrasse 2-12, 10587 Berlin, Germany

Abstract. The SRD1000 device supports 9 reference temperatures on the PLTS-2000 using superconductive to normal transitions of various metals. The evaluation of prototypes by several European metrological institutes showed that the SRD1000 concept is convenient and reliable for transferring the scale. Currently a pilot production series is being developed with improved quality for the superconductive transitions of W, Be, Zn and Cd compared to the prototypes. To achieve this we apply better techniques for preparing the metals and add EMI filters to the electrical leads of the devices. We report on technological improvements and present results of calibrations of the devices on the PLTS-2000. For interpolation between the reference points the sensor will be provided with an internal magnetic thermometer.

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INTRODUCTION

The Provisional Low Temperature Scale PLTS-2000^[1] below 1K has been defined as the equation for the melting pressure of ³He. The SRD1000 Superconductive Reference Device was developed to provide a direct and practical means for transferring the scale to the ultra-low temperature research community^[2]. The cryogenic sensor contains samples of superconductive materials attached to a set of micro-coil detectors. The transitions of the superconducting to normal state of the samples can be monitored by dedicated room-temperature electronics supplied with the sensor. The transitions provide 10 stable reference temperatures between 15 mK and 1.2 K, 9 on the PLTS-2000 and 1 on the ITS-90. In the past years we produced prototypes of the sensors and detection electronics. They were evaluated by several European institutes for metrology^{[3],[4]}. The SRD1000 concept proved to be convenient and reliable for transferring the scale. In this paper we present the status of the development of a new pilot production series with improved sample quality compared to the prototypes. The stability and width of а superconductive transition significantly contributes to the uncertainty of determining the transition temperature. Measurements at LION showed that we succeeded to achieve better transitions for samples of W. Be, Cd and Zn for devices of the new series compared to the prototypes. Devices and detection

electronics are being calibrated against the PLTS-2000 at the metrology institute PTB to achieve optimal accuracy, reliability and traceability of the reference temperatures.

STATUS OF THE DEVELOPMENT

The characteristics of the reference points of the pilot production series are listed in Table 1. The table gives the measurement results of typical temperatures T_C and widths W_C of the superconductive transitions of the reference samples. Also an estimate is indicated for the relative uncertainty $U(T_C)/T_C$ of the determination of T_C related to the characteristics of the transition: $U(T_C)/T_C = 2 (0.1 W_C/T_C) \times 100 \%$.

TABLE 1. Characteristics of the temperature reference points of the SRD1000 pilot production series.

reference sample	T _C (typical) [mK]	W _C (typical) [mK]	$\frac{U(T_C)/T_C}{[\%]}$
W	15	< 0.2	< 0.26
Be	21	< 0.3	< 0.28
$Ir_{80}Rh_{20}$	30	< 0.5	< 0.34
Ir ₉₂ Rh ₀₈	65	< 0.5	< 0.16
Ir	98	< 0.5	< 0.10
AuAl ₂	145	< 0.5	< 0.06
AuIn ₂	208	< 1	< 0.10
Cd	520	< 4	< 0.16
Zn	850	< 3	< 0.08
Al	1180	< 4	< 0.06

Compared to the prototypes we achieved improved quality for the reference points, in particular for those of the samples of W (15 mK), Be (21 mK), Cd (520 mK) and Zn (850 mK). The samples of W of about 3 x 3 x 0.5 mm were spark cut from a 6N single crystal (Alfa Aesar), followed by surface polishing and acetone / ethanol ultrasonic cleaning. The Be samples of 3 x 3 x 0.1 mm were cut from 2N8 foil (Alfa Aesar) and ultrasonically cleaned. In order to reduce supercooling effects occurring in these samples, spot welds of small dots of Al were applied on part of the surface area to induce superconductivity in the materials via the proximity effect. While testing the W and Be superconductive transitions in a dilution refrigerator, we noticed that T_C in both materials depended on the level of electro-magnetic interference from outside the cryostat, generated e.g. by digitally controlled instruments. Our experiments revealed that this dependence was not a result of heating of the samples but a direct influence from the radiation on the superconductivity. The proximity layer induced by the Al dots may have been sensitive for the energy of the interference. After we shielded this area with a silver laver and added EMI filters in the electrical leads close to the devices, both the W and Be transitions appear to be stable and immune for common interference present in a laboratory environment. Single crystal rods of Cd and Zn were prepared by slowly melting 6N shot of the materials in a sealed vertical vacuum tube. Samples of about 3 x 3 x 0.5 mm were spark cut from the crystals and etched for several minutes in a copper-sulfate solution to remove the interaction area of the cutting process. Contamination and mechanical stress in this area may lead to broadening of the superconductive transition. Finally, the samples were ultrasonically cleaned with distilled water. The width of the transition W_C was reduced by a factor of 3 to 4 when we attached the samples not with GE-varnish but with vacuum grease and a silver spring, which at the same time acts as a thermal link between the sample and the sensor body.

Devices and related detection electronics are being calibrated at the PTB against the realization of the PLTS-2000. Figure 1 shows an example of the calibration parameters of the reference point of Ir₉₂Rh₀₈ at about 65 mK. The output voltage V^* of the electronics is given as a function of temperature T_{2000} along the scale. V^* is the output voltage change relative to V_{SC} that is observed when the sample is in the superconducting state. Our experiments have shown that the heights of the voltage steps of all transitions are independent of the experimental environment in which they are realized. This means that for reproducing a calibrated reference point one does not have to complete a full temperature sweep from the superconducting to normal state of the sample, but one can directly find the reference temperature at V_{C}^{*} obtained from the calibration. In the example of Figure 1, $T_{C} = 65.38$ mK at $V_{C}^{*} = 0.0212$ V.



FIGURE 1. Superconductive transition of a sample of $Ir_{92}Rh_{08}$ and its calibration parameters.

FUTURE AND CONCLUSIONS

We plan to extend the number of supporting points for transferring the PLTS-2000 with a Mo reference point at about 950 mK and a Ti point at about 250 mK. Furthermore the sensor design allows the inclusion of a magnetic thermometer to produce a signal for interpolation between reference points.

A pilot production series of SRD1000 devices and detection electronics is being developed with improved characteristics compared to a prototype series. The systems are calibrated at PTB to achieve the highest accuracy and reliability for transferring the PLTS-2000 to the ultra-low temperature community. More information can be found on the SRD1000 web page^[5].

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