SRD1000 with improved reference points for thermometry below 1K

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Reference samples of improved quality were prepared and tested for a new series of the Superconductive Reference Devices SRD1000 for thermometry below 1K. In the past years prototypes of the device were evaluated by the PTB in Berlin, the NPL in London, BNM-INM in Paris and the CNRS-CRTBT in Grenoble. The evaluation proved that the SRD1000 is a convenient and reliable means of transferring the Provisional Low Temperature Scale 2000 (PLTS-2000). For the new series the quality of the transitions of Cd at 520 mK and Zn at 850 mK was significantly improved by preparing new crystals and applying better surface etching and attachment procedures to the samples. We report on the results of the characterization of the reference samples.

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1. INTRODUCTION

The Provisional Low Temperature Scale PLTS-2000[1] below 1K has been defined as equation for the melting pressure of ^{3}He. Superconductive Reference Devices SRD1000 were developed to provide a direct and practical means for transferring the scale to the ultra low temperature research community[2]. The SRD1000 cryogenic sensor contains 10 superconductive samples of reference materials. The transition of the superconducting to normal state of each sample can by monitored by dedicated room-temperature electronics supplied with the sensor. The midpoints of these transitions provide 10 reference temperatures between 15 mK and 1200 mK, see Table 1.

In the past years we produced prototypes of the cryogenic sensors and measurements electronics. They were we evaluated by several European institutes for metrology[3,4]. The width of a transition significantly contributes to the uncertainty of determining the transition temperature. For a new series of the SRD1000 we developed improved methods for preparation and attachment of the reference samples in the sensors in order to reduce the transition widths. We present the results of the characterization of the new Cd and Zn samples.

2. SAMPLE PREPARATION AND CHARACTERIZATION

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-sulphate 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 samples were attached to the micro-coil detectors of a SRD1000 test sensor which was mounted to the mixing chamber in a dilution refrigerator to characterize the superconductive transitions. During some initial tests, we found that applying varnish on the surface of the samples for attaching results in unwanted broadening of the superconductive transitions. Probably the varnish causes significant mechanical stress in the samples when cooling them to ultra-low temperatures. Better results were achieved when we attached the samples using a vacuum grease and a silver spring, which at the same time acts as a thermal link between the sample and the sensor body. In the prototype devices produced for the evaluation[4] we omitted the surface-etching step for Cd and Zn and used some varnish to attach the samples.

Figure 1 shows the superconductive transition of a Cd sample in prototype SRD004 and Figure 2 a transition of a sample prepared following the new procedure. The output voltage of the SRD1000 detection electronics is given as a function of temperature, normalized to 0 % for the superconducting state of the material and 100 % for its normal state. The difference between the temperatures to reach respectively the 90 % and 10 % level is defined as the width W_C of the transition. The W_C of the newly prepared sample is about 5 times smaller than that of the prototype. Figures 3 and 4 compare the transitions of Zn samples. The W_C of the current sample appears to be about 4 times smaller than that of the prototype.

Also for the other reference materials the sample preparation and attachments procedures are being improved while developing a new series of the devices. Table 1 overviews the expected transition widths of the new series compared to those of the prototypes.

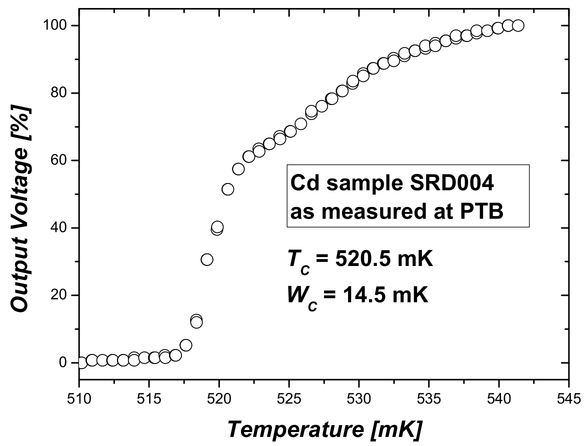

Fig. 1. Superconductive transition of cadmium in prototype SRD004, as measured by PTB[3].

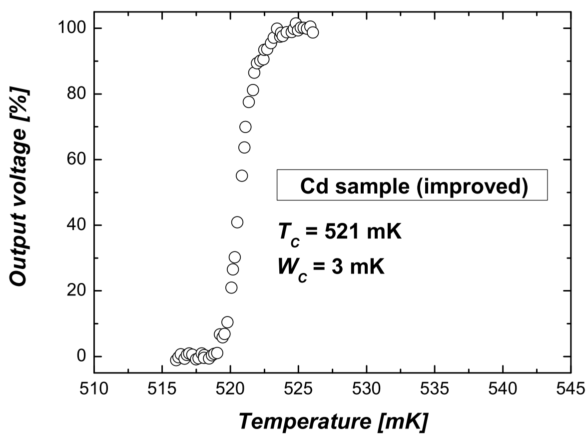

Fig. 2. Superconductive transition of a cadmium sample of improved quality.

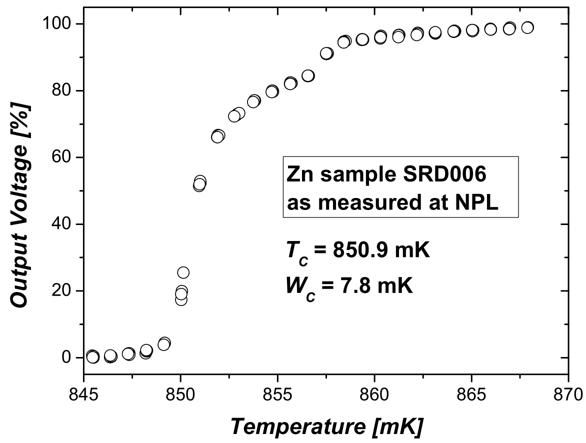

Fig. 3. Superconductive transition of zinc in prototype SRD006, as measured by NPL[3].

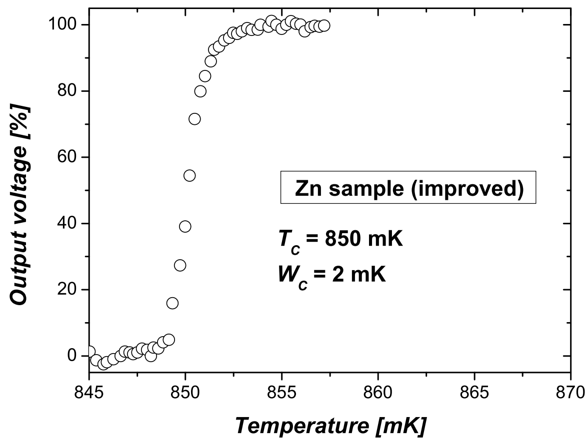

Fig. 4. Superconductive transition of a zinc sample of improved quality.

Improved superconductive reference device

reference material	nominal T_C [mK]	W_C prototypes [mK]	W_C improved series [mK]
W	15	< 0.2	< 0.2
Be	22	< 0.3	< 0.3
$Ir_{80}Rh_{20}$	30	0.7 - 1.2	0.3 - 1
$Ir_{92}Rh_{08}$	65	0.7 - 1	0.3 - 1
Ir	98	0.3 - 1	0.3 - 1
$AuAl_2$	145	0.4 - 0.7	0.3 - 0.6
$AuIn_2$	208	0.5 - 3	0.5 - 1
Cd	520	12 - 15	2 - 4
Zn	850	5 - 16	2 - 3
Al	1180	2 - 4	2 - 4

Table 1. Reference temperatures T_C and widths W_C of the superconductive transitions of the SRD1000 prototypes and that of a new series with improved sample quality.

3. CONCLUSIONS

Applying new preparation and attachments techniques for SRD1000 reference samples has resulted in improved superconductive transitions compared to those of a first series of prototypes. For the Cd and Zn reference points the transition width is reduced by a factor of 4 or more. This enables a more accurate determination of the reference temperature supported by these materials. The improvements will be implemented in a new series of the SRD1000. More information can be found at the SRD1000 web page[5].

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