A SUPERCONDUCTIVE DEVICE TO PROVIDE REFERENCE TEMPERATURES BELOW 0.5 K

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Résumé.- On a développé un dispositif contenant cinq matériaux supraconducteurs W, Be, Ir, $AuAl_2$ et $AuIn_2$ donnant respectivement les températures de références suivantes de 0,015 K, 0,024 K, 0,0975 K, 0,159 K, et 0,204 K. La reproductibilité de trois prototypes contenant chacun cinq matériaux supraconducteurs se situe entre 0,1 et 0,4 mK pour chacun de ces matériaux, sauf pour le Be qui est de 1 mK.

Abstract.- We have developed a device containing five superconductive materials W, Be, Ir, $AuAl_2$ and $AuIn_2$ which provides reference temperatures at 0.015 K, 0.024 K, 0.0975 K, 0.159 K and 0.204 K respectively. The reproducibility of three devices each containing these five superconductors was found to vary from 0.1 to 0.4 mK except for Be where it was 1 mK.

1. INTRODUCTION.- ^3He-^4He dilution refrigerators, offering very stable temperature operation from a few millikelvins to 0.6 K for basically indefinite periods of time, have been of great benefit to cryophysicists. One development at the NBS based on use of these refrigerators has been an accurate intercomparison of absolute thermometers which requires very stable temperatures over a period of several hours. Such tedious measurements of temperature are best suited to the establishment of a scale to be maintained at a national standards laboratory. Means must therefore be found to accurately transfer such a scale to other laboratories at which temperature measurement is a necessary but not the primary concern. To this end, we have been developing a superconductive fixed point device in which the reproducible superconductive transition temperatures, T_c, provide five fiducial temperatures which are measured on this temperature scale. This article will describe experiments completed on three protoype devices.

2. EXPERIMENTS AND DATA.- Figure 1 shows the arrangement of the coils and samples for a typical unit. Two holes are drilled through a round OHFC copper block, the other end of which has been machined to form a 6-32 screw. Into each of the two holes is inserted a bundle of 300 bare AWG 40 copper wires. Each bundle is welded in place, and the surface facing the 6-32 screw is then remachined flat. A serial number is stamped into the side of the unit near the screw. The whole assembly is electroplated with pure gold to an approximate thickness of 0.5 µm.

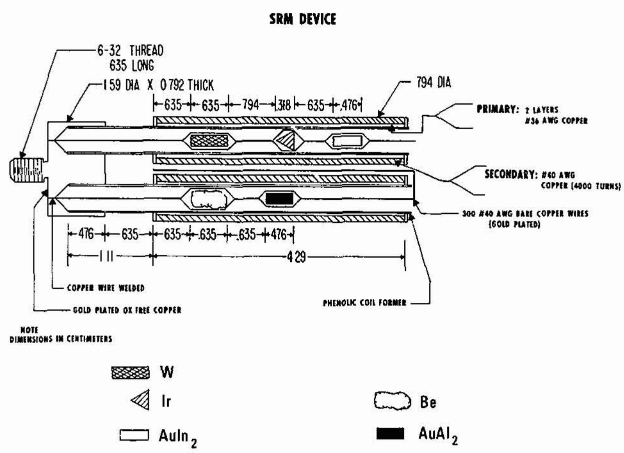

Fig. 1 : Fixed point device which provides five reference temperatures below 0.5 K. Samples of W, Be, Ir, $AuAl_2$ and $AuIn_2$ are bundled in copper wires which also enclose two sets of coils. The device is terminated in a 6-32 screw for easy attachment to an apparatus.

Samples of W, Be, $AuIn_2$, $AuAl_2$ /1/ and Ir are each bound at the positions shown in the figure with GE 7031 varnish and cotton thread. Two sets of copper coils (a primary and secondary) mounted on thin phenolic tubes are slipped into the two holes in the copper block and enclose the samples. The spare gold-plated copper wire is then folded back over the coils and lashed down with more GE 7031 varnish and thread. As a consequence of this design, the coils and samples should be in thermal equilibrium even at the lowest transition temperature ; no indication to the contrary has been observed.

Three units, constructed in the manner

described above, were cycled from room temperature to 0.011 K four times to test the reproducibility of the T_c values of each sample and to determine the variation in T_c from sample to sample.

The results are summarized in table I.

Table I

Measurement of the Superconductive Transitions of the Three Fixed Point Devices

Material	Serial Number	T_c Run 1 (mK)	T_c Run 2 (mK)	T_c Run 3 (mK)	T_c Run 4 (mK)	$\bar{T}$ (mK)	$\Delta\bar{T}$ (mK)	Range (mK)	Width, W (mK)	Hysteresis (mK)
W	1	15.3	15.4	15.5	15.5	15.4	0.1	0.2	0.15	0.03–0.08
W	2	15.3	15.1	15.4	15.5	15.3	0.2	0.4	0.2	0.05
W	3	–	–	15.4	15.0	15.2	0.3	0.4	0.2	0.5
Be	1	20.7	–	22.7	21.9	21.8	1.0	2.0	0.06	0.7– 1.4
Be	2	23.3	–	23.4	22.7	23.2	0.38	0.7	0.06	0.2– 1.2
Be	3	22.4	–	24.7	23.2	23.5	1.2	2.3	0.05	2.5–4.5
Ir	1	97.56	97.61	97.49	97.40	97.52	0.09	0.21	0.4	<0.1
Ir	2	97.36	97.36	97.18	97.13	97.26	0.12	0.23	0.4	<0.1
Ir	3	97.68	97.84	97.77	97.58	97.72	0.11	0.26	0.7	<0.1
$AuAl_2$	1	159.04	158.87	159.01	159.18	159.03	0.13	0.31	0.4	<0.1
$AuAl_2$	2	158.87	158.77	158.93	159.10	158.92	0.14	0.33	0.8	<0.1
$AuAl_2$	3	159.10	158.87	159.10	159.21	159.07	0.14	0.34	0.09	<0.1
$AuIn_2$	1	204.0	203.96	204.08	204.16	204.05	0.09	0.20	0.3	<0.1
$AuIn_2$	2	203.9	203.86	203.94	204.06	203.94	0.08	0.20	0.2	<0.1
$AuIn_2$	3	204.06	203.86	204.0	204.14	204.02	0.12	0.28	0.2	<0.1

By way of explanation of the entries, the first row identifies four T_c measurements for the tungsten sample in the prototype unit with serial number 1 ; the average T_c is 15.4 mK and the one-sigma deviation of these four measurements is 0.1 mK. The amount of hysteresis for this sample due to supercooling varied among runs from a minimum of 0.03 mK to a maximum of 0.08 mK, which is due to the variability in the cancellation of the ambient magnetic field. Note that a few entries are blank ; in a few cases the hysteresis was so large that the sample did not enter the superconducting state even when the refrigerator was allowed to cool to its bottoming temperature (~ 12 mK). Supercooling effects can be eliminated by spot welding or soldering a small amount of a higher T_c material to the sample /2/ ; experiments are in progress to test the suitability of this technique for the W and Be samples.

Setting aside the results for Be for the moment, we see from table I that the maximum spread in the T_c values for a given sample was 0.4 mK and that one standard deviation is half of that. On the basis of other measurements, we believe that this variation is due to variations in ambient magnetic field and is not due to actual changes in T_c of the samples. The wide variation in the T_c measurements of the Be samples is quite a different matter ; the differences in T_c values for different samples can be laid to lack of uniformity due to the preparation technique, but the variation in T_c values of the same sample is problematic and needs further study.

3. CONCLUSION.- Four thermal cycles of three prototype superconducting fixed point devices each containing five materials have shown that four of the materials provide temperature reference temperatures with a reproductibility of 0.2 to 0.4 mK. By far the most significant inconvenience in the use of the unit is the tendency of particular samples to supercool in small magnetic fields. Experiments are in progress to test the efficacy of attaching higher-T_c materials to the W and Be specimens

which are most susceptible to this effect. To date only the material Be has yielded an unsatisfactory variation in T_c ; further experiments are needed to determine its utility as a temperature fixed point. The results on the suppression of supercooling and improvement of the Be transitions are not yet in, but we are optimistic that units can be produced which will ultimately give these five fixed temperatures with a reproducibility of a few tenths of a millikelvin.

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