Tests of the Stability of Chinese RhFe Resistance Thermometers at Low Temperatures

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Abstract Rhodium–iron resistance thermometers are recommended as precise thermometers at temperatures below 25 K. The thermometers were developed at the National Physical Laboratory and produced by H. Tinsley and Co Ltd almost 50 years ago. Later, they were made by other companies and institutes as well, but despite this, the availability of the thermometers decreased and a new source of supply was needed. Several years ago, the Technical Institute of Physics and Chemistry (TIPC), Chinese Academy of Sciences, developed its own technology for making wire of Rh-0.5 at% Fe alloy which was used in the production of new thermometers. These devices have been tested previously at INRIM (Italy) and later at INTiBS (Poland). INTiBS has carried out an investigation focused on the thermometers' stability after thermal cycling treatment. This paper presents the results of stability tests of about 30 thermometers produced by TIPC in two batches. The resistance of each thermometer was measured at temperatures of about 4.6 K and 7.2 K before and after 10, 30 and 50 thermal cycles from room temperature. The methods of measurement and the design of the cryostat used for the research are also presented.

Keywords Cryogenic thermometers · Resistance thermometers · Rhodium–iron thermometers · Thermometer stability

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1 Introduction

The history of rhodium–iron resistance thermometers (RIRTs) extends for almost 50 years [1]. Due to their anomalous resistance versus temperature characteristics, they can be used for accurate temperature measurement at low temperatures, where platinum resistance thermometers become unusable because of low sensitivity. RIRTs were developed by R. Rusby at National Physical Laboratory and produced by H. Tinsley and Co Ltd in the early 1970s [2,3]. Later, they were produced by other companies and institutes [4–6], but despite this the availability of the thermometers decreased and a new source of supply was needed. Several years ago, the Technical Institute of Physics and Chemistry (TIPC), Chinese Academy of Sciences, developed its own technology for making wire of Rh-0.5 at% Fe alloy which was used in the production of new thermometers [7,8]. The construction of the thermometers is the same as capsule-type standard platinum resistance thermometers. The properties of the RIRTs were initially studied at INRIM and then at INTiBS at the end of the last decade [9,10]. Recently, after improvement of the thermometer production technology, about 30 thermometers produced by TIPC in two batches were tested at INTiBS. Between 2012 and 2013, RIRTs with the numbers shown in Table 1 were tested. The next batch, with the numbers shown in Table 2, was tested during the period 2014–2016.

This study is focused on measurements of the thermometer's resistance after 50 thermal cycles between room temperature and liquid helium temperatures. The resistance of each thermometer was measured at about 4.6 K and 7.2 K before the thermal treatment and after 10, 30 and 50 thermal cycles.

2 Thermal Treatment

For the thermal cycling treatment, the thermometers were mounted in a sealed stainless steel container made of a tube with a diameter of 25 mm and a length of 130 cm. The container was pumped to about 0.1 Pa pressure and then filled with gaseous helium to ensure a good thermal exchange between thermometers and liquid helium. The container was dipped slowly into liquid helium to cool the thermometers to 4.2 K over about 5 min. Then, it was removed from the helium Dewar, and the thermometers warmed to room temperature. The procedure was repeated 10 times initially and then 20 times in each of two subsequent runs. After each treatment, the thermometers were inserted into the copper block of a cryostat for measurement.

3 Cryostat Construction

A liquid helium cryostat developed at INTiBS, shown in Fig. 1, was used for the investigation. It was equipped with an adjustable thermal switch, *a*, mounted between the helium container, LHe, and the copper block, *b*. The heat switch was based on the dependency of the thermal conductivity of gaseous helium on pressure.

Nine thermometers from TIPC and the reference rhodium–iron thermometer B178 from Tinsley, which participated in the EURAMET.T-K1 key comparison [8], were mounted in the isothermal copper block, *b*. The copper block was surrounded by

Table 1 Results of RIRTs stability for the first batch of thermometers

	$T \approx 4, 36 \text{ K}$			$T \approx 7, 19 \text{ K}$		
	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2
<i>No. 8007</i>						
After 10 cycles	-0.093	0.055	0.00031	0.042	0.067	0.00018
After 30 cycles	-0.053	0.052		0.024	0.068	
After 50 cycles	-0.071	0.052		-0.071	0.067	
<i>No. 20102</i>						
After 10 cycles	-0.022	0.053	0.00033	0.013	0.065	0.00019
After 30 cycles	0.023	0.053		-0.036	0.065	
After 50 cycles	-0.048	0.051		-0.038	0.065	
<i>No. 20107</i>						
After 10 cycles	-0.016	0.069	0.00037	-3.7E-05	9.1E-05	0.00022
After 30 cycles	0.004	0.067		5.6E-05	8.9E-05	
After 50 cycles	-0.030	0.070		4.1E-05	9.0E-05	
<i>No. 20108</i>						
After 10 cycles	0.453	0.089	0.00099	-0.412	0.048	0.00039
After 30 cycles	2.373	0.081		3.442	0.048	
After 50 cycles	2.317	0.073		1.802	0.050	
<i>No. 20109</i>						
After 10 cycles	-0.077	0.054	0.00027	-0.153	0.068	0.00015
After 30 cycles	-0.052	0.056		-0.152	0.074	
After 50 cycles	-0.144	0.054		-0.165	0.068	
<i>No. 200912</i>						
After 10 cycles	-0.119	0.054	0.00036	-0.012	0.068	0.00022

Table 1 continued

	$T \approx 4, 36 \text{ K}$			$T \approx 7, 19 \text{ K}$		
	$\Delta T, \text{ mK}$	$u(\Delta T), \text{ mK}$	Self-heating, Ω/mA^2	$\Delta T, \text{ mK}$	$u(\Delta T), \text{ mK}$	Self-heating, Ω/mA^2
After 30 cycles	-0.030	0.051		-0.019	0.067	
After 50 cycles	-0.131	0.051		0.009	0.069	
No. 200913						
After 10 cycles	-0.814	0.052	0.00029	-1.086	0.067	0.00017
After 30 cycles	-0.875	0.052		-1.024	0.067	
After 50 cycles	-0.891	0.052		-1.116	0.067	
No. 200918						
After 10 cycles	-0.052	0.055	0.00032	0.014	0.068	0.00017
After 30 cycles	-0.078	0.052		-0.071	0.068	
After 50 cycles	-0.084	0.052		-0.064	0.068	
No. 200920						
After 10 cycles	0.037	0.053	0.00030	0.078	0.068	0.00018
After 30 cycles	0.087	0.053		0.050	0.068	
After 50 cycles	-0.047	0.052		-0.012	0.097	
No. 201017						
After 10 cycles	-0.055	0.052	0.00032	-0.138	0.071	0.00017
After 30 cycles	-0.066	0.059		0.049	0.084	
After 50 cycles	-0.433	0.054		-0.143	0.071	
No. 201020						
After 10 cycles	-0.088	0.074	0.00032	-0.066	0.099	0.00019
After 30 cycles	0.049	0.084		-0.008	0.096	
After 50 cycles	-0.024	0.073		-0.012	0.097	

Table 2 Results of RIRTs stability for the second batch of thermometers

	$T \approx 4, 6\text{ K}$			$T \approx 7, 2\text{ K}$		
	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2
<i>No. LT001</i>						
After 10 cycles	0.122	0.046	0.00013	0.058	0.040	0.00007
After 30 cycles	0.055	0.052		0.021	0.053	
After 50 cycles	0.044	0.051		0.023	0.040	
<i>No. LT002</i>						
After 10 cycles	0.071	0.192	0.00013	0.081	0.052	0.00008
After 30 cycles	−0.061	0.041		0.037	0.040	
After 50 cycles	−0.067	0.043		0.017	0.041	
<i>No. LT005</i>						
After 10 cycles	−0.256	0.044	0.00011	0.188	0.211	0.00007
After 30 cycles	−0.108	0.043		−0.039	0.051	
After 50 cycles	−0.127	0.046		−0.037	0.051	
<i>No. LT009</i>						
After 10 cycles	0.090	0.092	0.00013	0.051	0.157	0.00007
After 30 cycles	−0.148	0.089		−0.025	0.118	
After 50 cycles	−0.203	0.068		−0.110	0.121	
<i>No. LT011</i>						
After 10 cycles	−0.018	0.039	0.00016	−0.003	0.176	0.00010
After 30 cycles	−0.129	0.040		−0.053	0.039	
After 50 cycles	−0.149	0.040		−0.069	0.039	

Table 2 continued

	$T \approx 4, 6\text{ K}$			$T \approx 7, 2\text{ K}$		
	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2
<i>No. LT013</i>						
After 10 cycles	0.093	0.065	0.00016	0.044	0.115	0.00010
After 30 cycles	0.008	0.075		−0.015	0.131	
After 50 cycles	−0.021	0.083		−0.092	0.140	
<i>No. LT015</i>						
After 10 cycles	0.241	0.047	0.00013	0.151	0.114	0.00007
After 30 cycles	−0.086	0.056		−0.004	0.039	
After 50 cycles	−0.108	0.050		−0.009	0.045	
<i>No. LT016</i>						
After 10 cycles	0.142	0.042	0.00016	0.045	0.040	0.00009
After 30 cycles	0.082	0.071		0.015	0.049	
After 50 cycles	0.032	0.041		0.005	0.059	
<i>No. LT017</i>						
After 10 cycles	0.195	0.065	0.00013	0.110	0.099	0.00007
After 30 cycles	−0.126	0.038		−0.128	0.054	
After 50 cycles	−0.138	0.038		−0.105	0.067	
<i>No. LT019</i>						
After 10 cycles	0.114	0.049	0.00011	0.063	0.048	0.00006
After 30 cycles	0.029	0.049		0.026	0.044	
After 50 cycles	0.044	0.053		0.033	0.050	

Table 2 continued

	$T \approx 4, 6\text{ K}$			$T \approx 7, 2\text{ K}$		
	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2	ΔT , mK	$u(\Delta T)$, mK	Self-heating, Ω/mA^2
<i>No. 20101</i>						
After 10 cycles	0.120	0.043	0.00025	0.115	0.102	0.00016
After 30 cycles	0.029	0.060		−0.024	0.050	
After 50 cycles	0.013	0.045		−0.009	0.050	
<i>No. 200909</i>						
After 10 cycles	0.125	0.059	0.00011	−0.007	0.098	0.00006
After 30 cycles	0.052	0.067		0.022	0.052	
After 50 cycles	0.016	0.061		−0.033	0.080	
<i>No. 201301</i>						
After 10 cycles	0.117	0.045	0.00015	0.048	0.095	0.00008
After 10 cycles	0.092	0.265		−0.019	0.096	
After 10 cycles	0.012	0.055		−0.005	0.102	

Fig. 1 Liquid helium cryostat construction (*a* heat switch, *b* copper block, *c* thermal shield, *d* carbon thermometer, *e* lead sample, *f* mutual induction coils)

a thermal shield, *c*. Thermal contact between the thermometers and the block was ensured with Apiezon grease. A photograph of the copper block is presented in Fig. 2.

For controlling the temperature of the copper block near 4.6 K, a model 370 Lake Shore Cryotronics temperature controller, with an Allen–Bradley carbon resistance thermometer, *d* (Fig. 1) was applied. The temperature of the block was stable within ± 0.1 mK during the thermometer tests.

At a temperature close to 7.2 K, some tests were carried out with a superconducting lead (Pb) transition (the lead sample, *e*, is shown in Fig. 1) used for the temperature stabilization. The lead sample was inside mutual inductance coils, *f*. The signal from the coil was measured via a lock-in amplifier and transmitted to the PID controller. Because the accuracy of the temperature stabilization using this method was the same as obtained with the Lake Shore Controller, the second simpler methods were used for most tests.

4 RIRT Resistance Measurements

The measuring system is shown in Fig. 3. A Measurements International Model MI 6015 bridge (with a measurement uncertainty of 0.02 parts per million) was used

Fig. 2 Copper block with RIRTs

for the resistance measurements. The resistance R_X of each tested thermometer was measured using the standard thermometer no B178, placed in the copper block, as the reference resistor. This method reduces the error due to the temperature drift of the copper block, because both R_X and the resistance R_T of thermometer B178 have similar temperature coefficients, so the ratio R_X/R_T is nearly independent of temperature [11]. The resistance R_T was measured twice—before and after measuring the resistance ratios R_X/R_T of all of the tested thermometers. From these measurements, the values of R_T and the R_X can be calculated and they apply at the mean temperature of thermometer B178.

The resistance R_T was measured relative to a $10\ \Omega$ Tinsley standard resistor placed in a thermal enclosure outside the cryostat. The ratio resistance bridge and the standard resistor were calibrated at the Central Office of Measures (the National Measurement Institute in Poland).

The resistance R_T and the ratios R_X/R_T were measured at two currents, $I_1 = 1\ \text{mA}$ and $I_2 = \sqrt{2}\ \text{mA}$, and extrapolated to $I_0 = 0$. All numerical values of the resistances were the average of 40 measurements.

5 Tests of Thermometers Stability

To analyze the thermometer stability, we assumed that in a narrow temperature range—within about 10 mK—the dependence of $R = f(T)$ for the tested thermometers was linear with sufficient accuracy:

$$R = aT + b.$$

The a and b coefficients for each thermometer were determined in the first run before the thermal treatments. The resistance R_X was measured at three temperatures differ-

Fig. 3 Measuring system

ing by a few mK, and the linear regression function was applied to calculate a and b coefficients (Fig. 4, solid line). After the thermal treatments, the resistance was measured at two temperatures and the relation $R_1 = f(T)$ was determined assuming that the linear relationship $R_1 = aT + b_1$ is parallel to $R = aT + b$. The thermometer's stability is expressed by the temperatures difference $T'' - T'$ determined for two points

Fig. 4 Method of estimation of RIRT's stability

with the same value of resistance R' (see Fig. 4) after 10, 30 and 50 thermal cycles. The $T'' - T'$ value was calculated from the equation:

$$T'' - T' = \frac{b - b_1}{a}.$$

6 Measurements Results

The results obtained for the stability of the RIRTs at the temperatures of about 4.36 K and 7.2 K for the first and 4.6 K and 7.2 K for the second batch of thermometers are presented in Tables 1 and 2, respectively. The values of the uncertainty and self-heating effect are given too. The uncertainty budget included the following components:

- standard error of regression (function $R = aT + b$),
- uncertainty of determination of the self-heating corrections,
- uncertainty of ratio bridge,
- standard resistor thermal drift,
- standard thermometer reproducibility.

The results for some selected thermometers are presented in Figs. 5, 6, 7, 8 and 9. The thermometer numbers 20107, 200912 and 20108 were sent to INTiBS in the first batch, and the thermometer number 20101 and LT011 in second one, respectively.

Fig. 5 Graph of resistance versus temperature for thermometer 20107

Fig. 6 Graph of resistance versus temperature for thermometer 200912

Fig. 7 Graph of resistance versus temperature for thermometer 20108

Fig. 8 Graph of resistance versus temperature for thermometer 20101

Fig. 9 Graph of resistance versus temperature for thermometer LT011

Fig. 10 Stability of second batch RIRTs in 4.6 K

Fig. 11 Stability of second batch RIRTs in 7.2 K

It can be noticed that the stability of thermometer 20108 is worse than the other four, and this is probably related to the larger self-heating, see Table 1. It seems likely that this is due to a small leak in the capsule.

The stability of the second batch of thermometers is shown in Figs. 10 and 11. The stability results of thermometers which showed large self-heating were unreliable, and they are not included in the figures.

7 Summary

The studies carried out at INTiBS showed that the majority of the tested RIRTs produced by TIPC are very stable—within 0.2 mK—after thermal cycling treatment between the room and liquid helium temperatures. After first 10 cooling runs the $T'' - T'$ value was larger, but subsequent cycles of thermal treatment stabilized the RIRTs within the range 0.2 mK compared with the indication before thermal treatment.

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References

1. W.L. Tew, R.L. Rusby, P. Lin, L. Lipiński, P.P.M. Steur, B.W.A. Ricketson, *Int. J. Thermophys.* **36**, 2036 (2015)
2. R.L. Rusby, in *Temperature, Its Measurements and Control in Science and Industry, Part 2*, ed. by H.H. Plumb (ISA, Pittsburgh, 1972), p. 865
3. R.L. Rusby, in *Temperature, Its Measurements and Control in Science and Industry, Part 1*, ed. by J.F. Schooley (AIP, New York, 1982), p. 829
4. L.M. Besley, P. Lin, *Meas. Sci. Technol.* **4**, 1357 (1993)
5. G.A. Kytin, S.F. Vorfolomeev, Y.A. Dedikov, L.N. Ermilova, D.N. Astrov, in *Proceedings of the 17th Meeting of the CCT, Document 89-7* (BIPM, Sevres, 1989)
6. O. Tamura, H. Sakurai, *Jpn. J. Appl. Phys.* **26**, L947 (1987)
7. P. Lin, *Acta Metrol. Sin.* **29**, 171 (2008)
8. P. Lin, X. Li, B. Gao, L. Yu, R. Huang, F. Li, Development of Chinese standard type of rhodium–iron resistance thermometers, in *Presented on XIII International Symposium on Temperature and Thermal measurements in Industry and Science TEMPMEKO 2016, Zakopane* (2016)
9. F. Pavese, A. Szmyrka-Grzebyk, L. Lipiński, H. Manuszkiewicz, P. Qiu, J.T. Zhang, P. Lin, X.W. Li, *Int. J. Thermophys.* **29**, 51 (2008)
10. L. Lipiński, A. Szmyrka-Grzebyk, P. Lin, X.W. Li, H. Manuszkiewicz, D. Jancewicz, A. Grykalowska, P.P.M. Steur, F. Pavese, *Int. J. Thermophys.* **31**, 1696 (2010)
11. V. Batagelj, J. Bojkovski, *Int. J. Thermophys.* **32**, 1409–1417 (2011)