Int J Thermophys (2008) 29:51–56
DOI 10.1007/s10765-007-0335-3

Preliminary Results on New Prototypes of Precision Rh-0.5at%Fe Resistance Thermometers of Chinese Production

F. Pavese · A. Szmyrka-Grzebyk · L. Lipinski · H. Manuszkiewicz · Ping Qiu · Jin Tao Zhang · Peng Lin · Xing Wei Li

Published online: 9 January 2008

Abstract Given the practical impossibility of obtaining new precision Rh-0.5at%Fe resistance thermometers in recent years, the possible re-starting of the production of such thermometers in Yunnan (China) was explored by Istituto Nazionale di Ricerca Metrologica (INRIM). Ten prototypes of the new production were made available in early 2006. The paper reports the preliminary data from the testing performed to date on these prototypes at National Institute of Metrology (NIM) and Instytut Niskich Temperatur i Badan Strukturalnych (INTiBS). Although a problem with the alloy composition was detected, the reproducibility results on thermal cycling are very encouraging. Resistance-temperature (R-T) characteristics below 30 K, though not identical with those of similar thermometers formerly available from Tinsley and VNIIFTRI, are still suitable for accurate metrology.

Keywords Resistance cryogenic thermometers · Rhodium–iron · Stability on thermal cycling

F. Pavese (✉)
Division of Thermodynamics, Istituto Nazionale di Ricerca Metrologica (INRIM), strada delle Cacce 73, 10135 Torino, Italy
e-mail: f.pavese@inrim.it; f.pavese@imgc.cnr.it

A. Szmyrka-Grzebyk · L. Lipinski · H. Manuszkiewicz
Instytut Niskich Temperatur i Badan Strukturalnych (INTiBS), Wroclaw, Poland

P. Qiu · J. T. Zhang · P. Lin
National Institute of Metrology (NIM), Beijing, China

X. W. Li
Yunnan Kunming Dafang Science and Technology Ltd., Kunming, China

1 Introduction

In view of the continuing worldwide shortage of accurate RhFe thermometers, which are necessary to maintain the ITS below the lower limit of the standard platinum resistance thermometers (SPRTs), co-operation between the Istituto Nazionale di Ricerca Metrologica (INRIM) and the National Institute of Metrology (NIM) allowed the revival of Chinese production discontinued about 10 years ago. The paper reports the first data obtained from a batch of 10 thermometers measured at NIM and the Instytut Niskich Temperatur i Badan Strukturalnych (INTiBS) for the R-T characteristics and the stability on thermal cycling.

2 Chinese Rh–Fe Thermometers

The thermometers were initially produced in the 1980s by a Yunnan factory and were tested for their resistance-temperature characteristics and their stability under thermal cycling by Besley and Lin Peng [1]. That initial model used the same nominal alloy (Rh-0.5at%Fe) as NPL, and as then produced by Tinsley and the Institute for Physical–Technical and Radiotechnical Measurements (VNIIFTRI) (both of which will be referred to in the paper as "commercial models").

Subsequently, the Chinese production was discontinued. However, the know how remained available in Yunnan, thanks to a small factory producing various types of thermometers. It was also possible to find a spool of the original wire ($d = 0.05$ mm) used in the previous production.

2.1 Specimens

Ten prototypes were produced, contained in a platinum capsule of the size typical of SPRTs. They were made with a room-temperature resistance of 50 Ω, similar to the Tinsley 'short' model, as a compromise between sensitivity and reduced wire length.

2.2 Procedures and Measurement Results at NIM

The 10 prototypes were initially measured by NIM at 4.2 K only and were subjected to 10 thermal cycles between room and liquid helium temperatures. The results of this first test are shown in Table 1. The uncertainty of the reproducibility measurements was about 1 mK (1 σ) in the apparent temperature differences, ΔT. One of the thermometers was found to be unstable, possibly due to a short between two turns of the coil, while the stability of the others was essentially within the measurement uncertainty. However, the resistance values at 4.2 K were found to be much higher than typical commercial thermometers of this nominal type, while the sensitivity, evaluated in a small interval around 4.2 K, was found to be lower.

Table 1 NIM early test results

Thermometer	Reference temperature (K)	Pre-cycling resistance (Ω)	Post-cycling resistance (Ω)	Resistance variation (mΩ)	ΔT (mK)	Sensitivity ($\Omega \cdot K^{-1}$)
200601	4.2623	10.54924	10.54923	−0.01	−0.06	0.154
200603[a]	4.2623	10.65238	10.63981	−12.77	−93	0.137
200604	4.2623	10.46646	10.46646	0.00	0.00	0.151
200606	4.2623	10.56412	10.56424	0.12	0.78	0.153
200612	4.2623	10.59819	10.59821	0.02	0.13	0.156
200614	4.2623	10.54049	10.54056	0.07	0.46	0.151
200616	4.4348	10.43516	10.43530	0.14	0.95	0.148
200618	4.4348	10.36995	10.37001	0.06	0.41	0.145
200619	4.4348	10.35691	10.35701	0.10	0.68	0.148
200620	4.4348	10.45832	10.45833	0.01	0.07	0.150

[a] Unstable

2.3 Procedures and Measurement Results at INTiBS

In order to validate these preliminary results, the thermometers were shipped to IMGC (now INRIM) and then measured by INTiBS.

As the first step in the investigation carried out at INTiBS, the resistance-temperature (R-T) characteristics of nine thermometers (#200601, 200604, 200606, 200612, 200614, 200616, 200618, 200619, and 200620) were measured in the low-temperature range from about 2.3 K to about 25 K. A cryostat suitable for measurements of thermometer characteristics under isothermal conditions was used. The tested thermometers and the standard Rh–Fe thermometer S/N B178 calibrated at NPL were placed in an isothermal copper block.

The temperature of the block was controlled with a LakeShore Cryotronics temperature controller Model 370 with an uncertainty of ±0.1 mK. A thermal shield surrounded the block. A Measurements International Model 6015 resistance bridge (uncertainty of measurements equal to 0.02 ppm) was used for the thermometer resistance measurements. The R-T characteristics in the temperature range between 2.3 K and 25 K were measured twice, once at the beginning of the test (first cooling cycle at INTiBS) and again at the end, after 80 cooling thermal cycles down to liquid helium temperature. The resistances of the thermometers were computed for $I = 0$ mA. In this way, the self-heating of the thermometers was also determined; it was found to be less than 1.5 mK at 2.3 K for a current of 1 mA.

For the thermal cycling, the thermometers were mounted on a plastic shaft placed inside a stainless-steel tube. The tube was filled with a small quantity of helium to increase the heat exchange. All thermometers were simultaneously cooled for about 10 min to the temperature of liquid nitrogen (about 77 K), where they were kept for a maximum of 20 min before being cooled to liquid helium temperature (4.2 K). After each cooling cycle, the thermometers were heated to room temperature. Twenty full

Table 2 INTiBS uncertainty budget

Uncertainty component	k	(mK)
Gradient and stability of temperature in the cryostat		0.2
Bridge accuracy		0.22
Bridge noise		0.1
Overheating of tested thermometer		0.2
Measurement on the RhFe standard thermometer		0.1
Uncertainty of the difference (ΔT) values: u	1	0.4
U	2	0.8
Calibration of the RhFe standard thermometer		1
Value of the standard resistor		0.33
Uncertainty on T values: u	1	1.1
U	2	2.2

cooling cycles were performed from room temperature to 4.2 K and back. Then, the thermometer resistances were measured at 4.2 K. The thermal cycles were repeated another 20 times down to liquid helium, and the thermometer resistances were again measured at two temperatures: 4.2 K and 4.7 K. Finally, the thermometers were thermal cycled down to liquid helium for another 40 cycles, making a total of 80 cycles. Then, the full calibration of the R-T characteristics was repeated.

The uncertainty budget for the INTiBS measurements is reported in Table 2 [2,3].

For all thermometers, the following model describing the R-T characteristics was used:

$$R_{\text{cal}}(T) = a + bT^{1/2} + cT + dT^{3/2} + eT^2 + fT^{5/2} + gT^3 + hT^{7/2} \tag{1}$$

For all thermometers and runs, the fitting residuals were between 0.4 mK and 0.6 mK ($1\,\sigma$), though some periodicity can be observed in the residuals since the number of experimental points is smaller than normally necessary to obtain random residuals with the Rh-0.5at%Fe alloy in that temperature range.

2.3.1 R-T Characteristics

Typical R-T characteristics for four RhFe thermometers are presented in Fig. 1 and the relative sensitivity in Fig. 2. The other thermometer characteristics lie between them.

The sensitivity is about 40% less, $0.16\,\Omega \cdot \mathrm{K}^{-1}$ at 4.2 K, compared to about 0.23 $\Omega \cdot \mathrm{K}^{-1}$ for a commercial thermometer with the same room-temperature resistance.

2.3.2 Stability to Thermal Cycling

In analyzing the stability on thermal cycling, one must bear in mind the dispersion due to measurement uncertainty. Obviously, not all the items in the uncertainty budget in

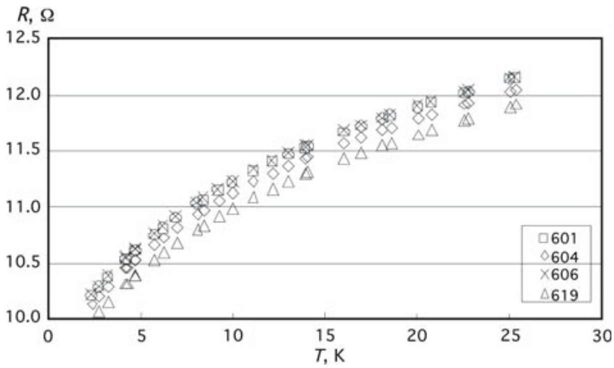

Fig. 1 R-T characteristics of the Chinese RhFe thermometer 50 Ω prototypes

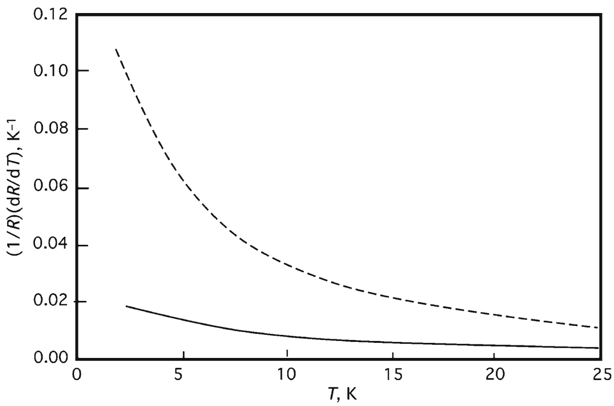

Fig. 2 Relative sensitivity $1/R(\mathrm{d}R/\mathrm{d}T)$ of the RhFe thermometer prototypes: solid line, present work; dotted line, commercial

Table 2 are relevant to reproducibility; should only the items listed in the first part of the table be considered, the typical uncertainty would be about 0.4 mK ($1\,\sigma$).

In Fig. 3, the best case (a) and the worst case (b) are reported. The two lines indicate the fitting of the initial and final calibration points (separately) over the whole temperature range with respect to the fitting function of Eq. 1 using all calibration points as the baseline.

3 Discussion and Conclusions

The results reported indicate that the fabrication of the thermometers is of very good quality and that these prototypes can represent a valid alternative to the models so far commercially available. However, the resistivity of the wire, being much higher than that of the wire used so far in commercial production, indicated that the alloy used for these prototypes was possibly of the wrong composition. An analytical assay found that the alloy contains too much iron, but the main reason for the dissimilarity

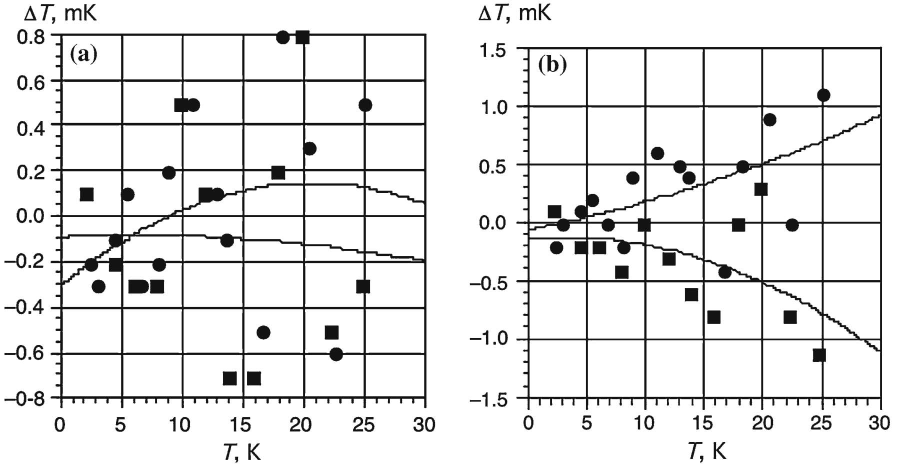

Fig. 3 Reproducibility of prototypes over 80 thermal cycles (in the range of 2.3−25 K): (**a**) #200612 best and (**b**) #200618 worst case. Dots are the residuals of the data of both initial and final calibrations, fitted together. See text for the meaning of the two lines

from the commercial thermometers was found to be an annealing procedure that almost certainly oxidized the iron, resulting in non-uniform concentration within the rhodium, an effect observed at NPL to result in the observed characteristics.[1] More work is in progress in this respect.

Acknowledgment This work was made possible by a partial grant obtained from the Ministry of Foreign Affairs of Italy in the frame of an Italy-China co-operation project.

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[1] We are grateful to NPL for the private communication.