INVESTIGATION ON THE STABILITY OF 0.6-Ohm HTPRTs AT KRISS

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Introduction

The instability of HTPRTs at high temperatures is one of the most important sources of the irreproducibility of the ITS-90 over the range from 660 to 960 °C. The reason for the instability of an HTPRT is the change in the structure of platinum – growth of big grains, creation of vacancies and dislocations in the lattice, redistribution of vacancies and impurities. At the temperatures above 600 °C these processes go much faster than at low temperatures.

In the previous research on HTPRTs some methods were suggested to improve the stability of the thermometers: special annealing and slow cooling technique for maintaining the equilibrium concentration of vacancies, methods of protection platinum from contamination by using platinum and graphite tubes. However, the instability of HTPRTs is still observed very often, especially when an HTPRT is being used in different annealing and fixed point furnaces.

One of explanations for the effect of changing the conditions of use on the instability of HTPRT resistance at high temperatures may be linked to the change of the electrical potential that appears on the thermometer leads with respect to ground. The value of the potential strongly depends on the furnace, most probably because of the different leakage paths between the thermometer and ground. In papers [1, 2] it was shown that the resistance of an HTPRT can increase by the equivalent of several mK after annealing in the furnace with the leads of the thermometer connected to ground. It was also shown that application of a positive bias between the leads and ground can result in decreasing the resistance.

By this time the physics of this interesting and important phenomenon is not fully understood. In this short study, performed at KRISS, the further data, supported the effect of the voltage on the HTPRT stability were obtained. In order to develop recommendations and special methods for improving the stability of HTPRTs the investigation should be continued.

2. Initial stabilization of the HTPRTs at VNIIM.

The initial stabilization of the HTPRTs was performed at VNIIM after manufacturing the thermometers. The thermometers were annealed in the furnace at 1100 °C for about 100 hours. The furnace contained a ceramic tube and quarts protection tubes. The voltage measured between the thermometer leads and ground during the anneal was about +1.5 V. The graphs showing the change of the resistance at the TPW are presented in Fig.1. All the thermometers exhibited a decrease in the resistance. The stability of R(TPW) for the last 20 h at 1100 °C was better than the equivalent of 0.5 mK.

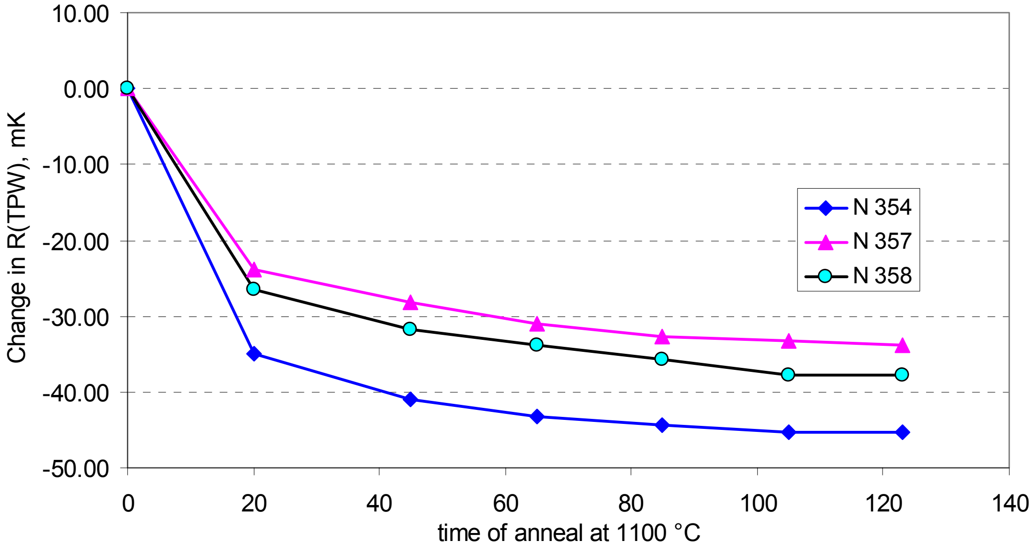

Fig. 1 Initial stabilization of the HTPRTs at VNIIM

3. Experiments in the annealing furnace at KRISS.

The annealing furnace at KRISS contains a ceramic tube and special quarts tubes inside which there are graphite and platinum cylinders are located for protection of the HTPRT sensing element from possible contamination from the ceramic. After the strain removing anneal at 675 °C, the HTPRTs were submitted to the investigation annealing cycles at 975 °C. The first measurements of the voltage between the thermometer leads and ground when the thermometers are in the annealing furnace, gave the values of about + 4 V to + 6 V for different thermometers. However, the first and the second anneals resulted in an increase in the TPW resistance by about the equivalent of 1.5 to 5 mK. (Fig.2). It was discovered later that during the anneal the leads of the thermometers lay on the grounded metal case of the neighboring furnace. Thus, the thermometers were unintentionally kept in the high temperature furnace with zero potential on the leads. This, most probably, was the reason for the observed increase in the resistance. Next two cycles were performed with the leads insulated from ground. It resulted in a decrease of the slope of the curves. Two thermometers approached a good stability. The third thermometer #358 continued increasing, but with a smaller rate.

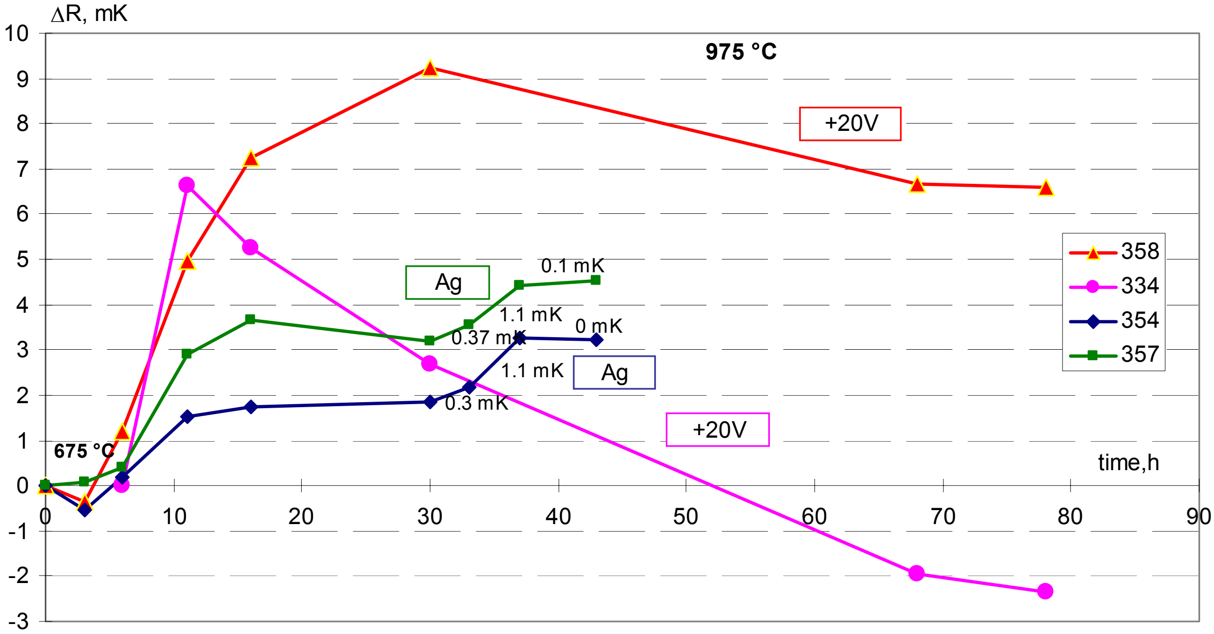

Fig. 2 Investigation of the stability of the HTPRTs at KRISS.

At that time one more HTPRT was involved into the study. HTPRT #334 was previously used for a long time at the Ag fixed point, and its quarts sheath was seriously devitrificated. The thermometer showed fast increasing in the R(TPW) after each measurement at Ag. The first 5-hour anneal at 975 °C resulted in the change of the R(TPW) resistance by the equivalent of + 6.6 mK. The next anneals were performed with the bias voltage of +20 V applied to the leads of HTPRT #334 with respect to ground, and, as seen from Fig.2, the resistance of the thermometer went down immediately after the application of the voltage. After 30 h of anneal the voltage of +20 V was also applied to the leads of HTPRT #358, which previously exhibited continues increase in the resistance. The behavior of the R(TPW) of the thermometer changed from increase to decrease It should be noticed that the rate of the decrease in the HTPRTs resistance became very small after about 50 h from the beginning of the anneal with the positive voltage on the leads.

4. Experiments at the fixed point of Ag at KRISS

Two HTPRT #354, #357 were calibrated at the Ag fixed point. Before insertion into the Ag cell the thermometers were slowly heated in an annealing furnace to 975 °C, then they were transferred to the cell. After the measurements in the Ag cell were completed, the thermometers were transferred to the annealing furnace again, annealed there at 975 °C for 2-3 h, and cooled for 7 h to 420 °C, then removed from the furnace. The first calibration at the Ag point resulted in an increase in R(TPW) of about 0.3 mK, the second calibration gave a greater increase of about 1.1 mK. The measurements of the voltage between the thermometer leads and ground, when the HTPRT was connected to the F-900 bridge, showed the value of about + 0.037 V, which is close to zero. It was suggested that the next calibration should be followed by an anneal and slow cooling of the HTPRTs with a small positive potential on the leads with respect to ground in order to reverse the change in the resistance occurring probably at the ground potential during the measurements at the Ag point. The voltage during the anneal was set to + 6 V. In Fig.2 one can see that the thermometers showed a very good stability in R(TPW).

5. An experiment at the Ag fixed point cell.

An interesting experiment was carried out during the measurements at the Ag cell. The thermometer # 357 was disconnected from the bridge, then it was kept at a positive potential of about +20 V on the leads with respect to ground for 0.5 h. After that the source of voltage was removed and the measurements at the Ag point were continued. Fig.3 shows the process of changing the resistance after removing the voltage. The drop of the resistance, corresponding to 30 mK, was observed and subsequently the resistance was slowly returning to its initial value for about 45 min. The results allow us to make a hypothesis that the positive voltage can influence the distribution of impurities and vacancies at a high temperature. The anneal with a positive potential on the leads can help to remove the excessive defects more effectively and even probably somewhat improve the electrical purity of platinum.

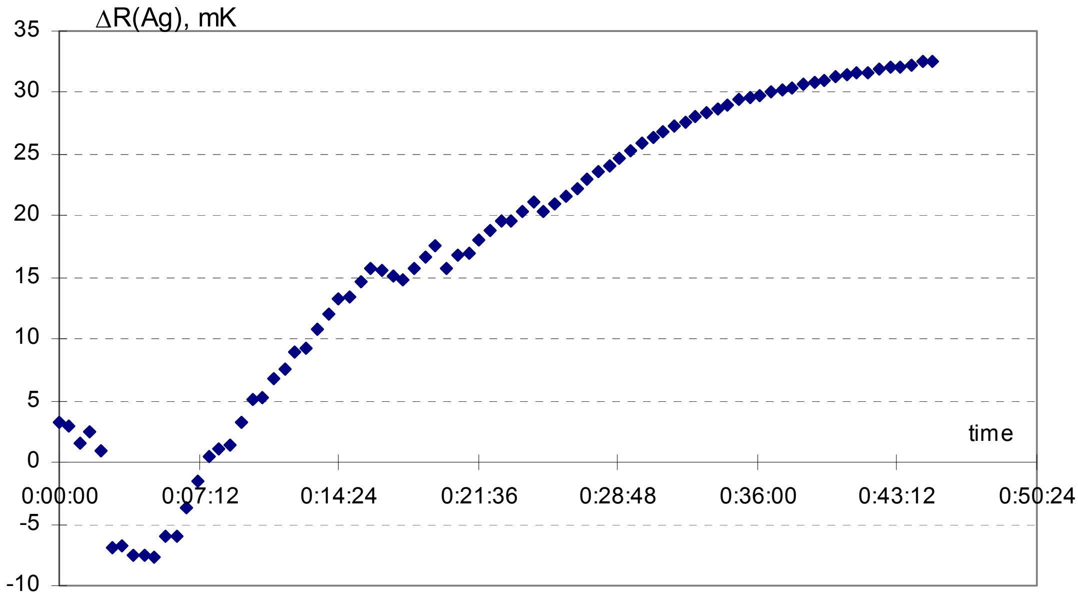

Fig. 3 The change in the R(Ag) after application of +20V between the leads and ground at the Ag fixed point cell.

6. Conclusions and suggestions for the future study.

1. The experiment at KRISS supported the previous observations on the effect of voltage between the thermometer leads and ground on the stability of the HTPRT.
2. From the experimental results it can be suggested that the thermometer must never be kept at a high temperature with its leads grounded.
3. After the measurements at the Ag fixed point were completed the thermometer should be annealed in the furnace with a positive potential on the leads, or a bias voltage should be applied to the leads with respect to ground in order to restore the equilibrium distribution of the defects.
4. The further study is necessary, which should help to develop methods of achieving a good stability for HTPRTs. The steps of the study should be as following:
   - Investigation of the effect of the voltage on the HTPRTs of different purity.
   - Study the process of changing the electrical purity of a contaminated thermometer under a positive voltage applied to its leads.
   - Estimation of the voltage value , which can be the most effective for the resistance stabilization.
   - Estimation of the time required for annealing the thermometer after

measurement at Ag point to obtain a very stable resistance value.

- The most important thing is to try to discover the real physical nature of the phenomenon. For this purpose it is required to carry out the chemical analysis of the platinum wire after application of the positive and negative voltages.

REFERENCES

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