Experimental Study and Computer Modeling of the Triple Point of Argon System

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Abstract: The triple point of argon is a defined fixed point in ITS-90 for calibration of standard platinum resistance thermometers (SPRTs). A new triple point of argon system with multiple re-entrant wells was developed. This system was tested to evaluate the duration and quality of the argon triple-point temperature plateau. The testing results showed that the plateau can be as long as 100 hours with the temperature change less than 0.05 mK. The re-entrant wells' uniformity testing showed that the temperature is consistent among the multiple re-entrant wells. Uncertainty analysis shows that the combined uncertainty of the argon system is 0.25 mK (k = 2). In order to study the heat transfer process and the influence on the thermal equilibrium of the argon system during the realization of the triple-point of argon plateau, finite element analysis (FEA) modeling was carried out to simulate the thermal conductivity, convection, and radiation inside the argon system. The FEA simulation results are described and discussed in this paper. According to the simulations results, suggestions are provided in order to quickly reach thermal equilibrium and obtain a long, stable plateau in the triple point of argon system.

Keywords: fixed point, ITS-90, SPRT calibration, FEA simulation, temperature, triple point of argon

1. Introduction

The triple point of argon (T_{90}=83.8058 K) is one of the most commonly used fixed points for the calibration of standard platinum resistance thermometers (SPRTs) at low temperatures below 273.16 K. In the past twenty years, a few argon apparatus for realization of the triple point of argon were developed by other researchers [1-5]. In order to calibrate SPRTs at the triple point of argon efficiently and accurately, a new triple point of argon system based on the design of the argon triple point apparatus by NIST [1] with multiple re-entrant wells and accurate triple-point plateau temperature was recently developed at Fluke Corporation. This newly developed argon system was tested under different experimental conditions. The immersion profile of the re-entrant wells was measured and compared with the ITS-90 theoretical hydrostatic values. The temperature consistency among re-entrant wells was also checked. The experimental results are reported and the influence of different experimental parameters on the performance of the argon system is discussed in the paper.

In order to study the heat transfer process and the influence on the thermal equilibrium of the argon system, finite element analysis (FEA) modeling was carried out to simulate the complex heat transfer process that includes heat conductivity, convection, and radiation occurring in the argon system. Based on the simulation results, suggestions are provided in order to quickly reach the thermal equilibrium and obtain a long, stable plateau.

2. Introduction of the triple point of argon system

Figure 1 is the schematic drawing of the triple point of argon system. The main structure of this system includes a high-accuracy temperature controller with the resolution of 1.0 mK for the temperature setting point, a stainless steel liquid nitrogen Dewar vessel, two argon gas reservoir cylinders with the volume of 13.4 liters for each cylinder for storage of argon gas, a vacuum shield, a heater shield with a 12 watts flexible heater attached on the outside surface of the shield, and a central argon cell with four stainless steel re-entrant wells. The immersion depth of the re-entrant wells is 160 mm. The inside diameter of the re-entrant wells is 8.0 mm. High-purity argon (certified purity: 99.9999%) is used in the argon system. A total amount of 13.3 moles of argon is permanently sealed inside. The pressure of the argon system is 1.172×10^3 KPa at room temperature. A PRT sensor is attached at the inside surface of the heater shield. It is used with the flexible heater and the temperature controller together to control the temperature of the heater shield.

When inserting SPRTs into the re-entrant wells or when moving SPRTs during the calibration process, helium gas flows through the re-entrant wells to prevent SPRTs from getting stuck inside the re-entrant wells due to condensation and freezing of moisture in the air. The volume of the liquid nitrogen Dewar inside the triple point of argon system is 40 liters. The liquid nitrogen after filling can last up to 20 hours with the argon system operating at the triple-point plateau.

3. Experimental procedures and results

The first step for realization of the triple point of argon is to fill liquid nitrogen into the argon system one day in advance to freeze the argon system. It takes about 15 minutes to fully fill the liquid nitrogen Dewar. The argon gas inside the two argon gas cylinders will be condensed into the central argon cell overnight. The temperature can be read from the controller or the pre-inserted monitoring SPRTs. The second step is to raise the argon system temperature to a certain point (e.g. -190.0 °C) that is below, but close to, the triple point of argon (-189.3442 °C). The system is left at this temperature for about two hours to stabilize the system. The temperature is then raised to about 1.5 to 2.0 °C above the triple point of argon (-187.344 °C) to pre-melt the outer layer of the argon cell. The pre-melting time is about one hour. The fourth step is to drop the argon system temperature to a maintenance temperature that is about 20 to 60 mK higher than the triple point of argon. The argon system will remain at the triple point of argon plateau for a certain period (from a few hours up to a few days depending on the conditions) until the three-phase equilibrium at the triple point of argon is broken because of melting and vaporization of solid and liquid argon.

In this study, standard platinum resistance thermometers (Fluke SPRT models 5681 and 5683) were used to measure the triple point of argon melting plateaus. A resistance bridge (Measurements International DC Automated Thermometry Bridge, model 6010T, accuracy ±0.05 ppm) was used to measure the resistances of the SPRTs. A Tinsley 10 ohm AC/DC standard resistor was used with the bridge. A triple point of water cell (Fluke model 5901) with a maintenance bath (Fluke model 7312) provided the temperature comparison standard.

In order to investigate the influence of the maintenance temperature on the triple-point plateau of the argon system, a few experiments were carried out at different maintenance temperatures. Figure 2 shows the tested plateau with the maintenance temperature 20 mK above the triple point of argon. It can be seen that the plateau duration is over 100 hours with the temperature change less than 0.05 mK.

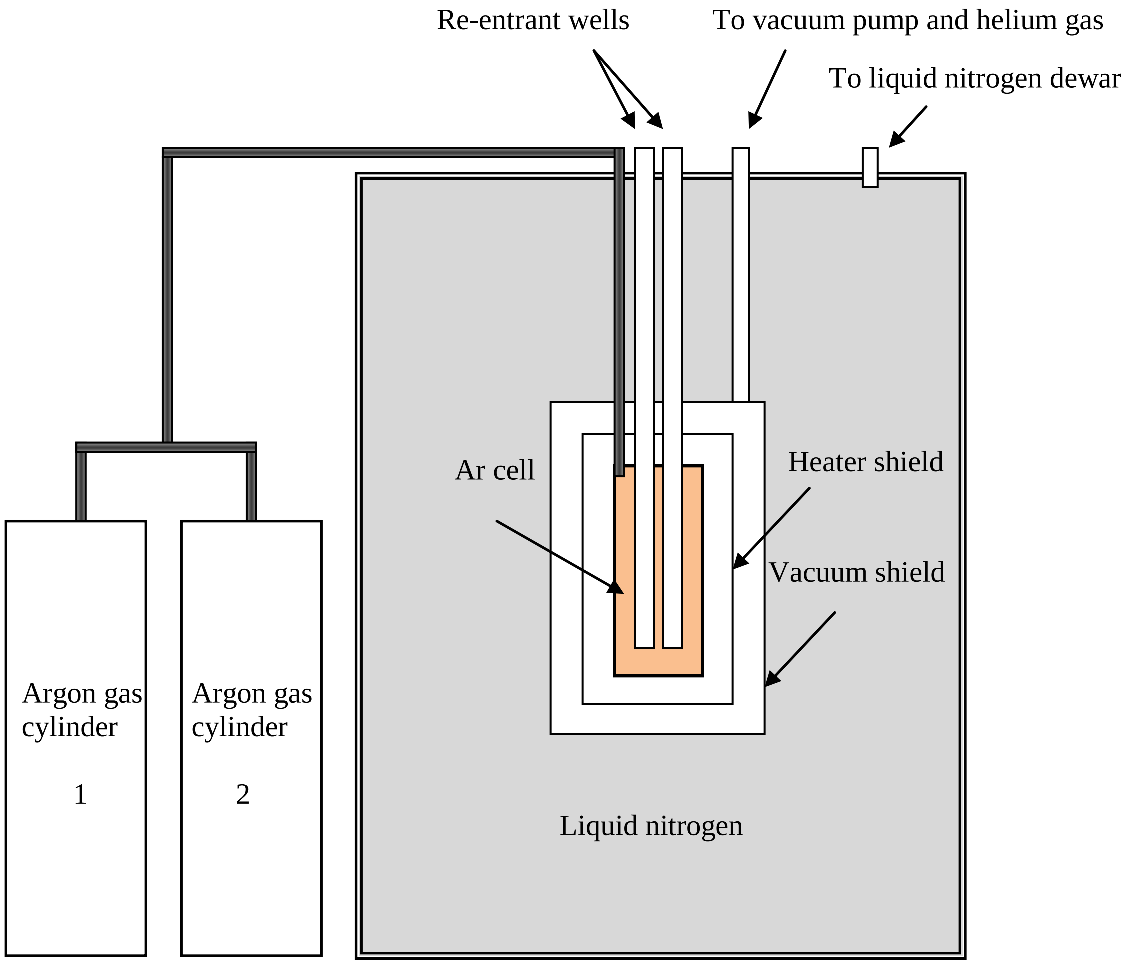

Figure 1. Schematic drawing of the triple point of argon system.

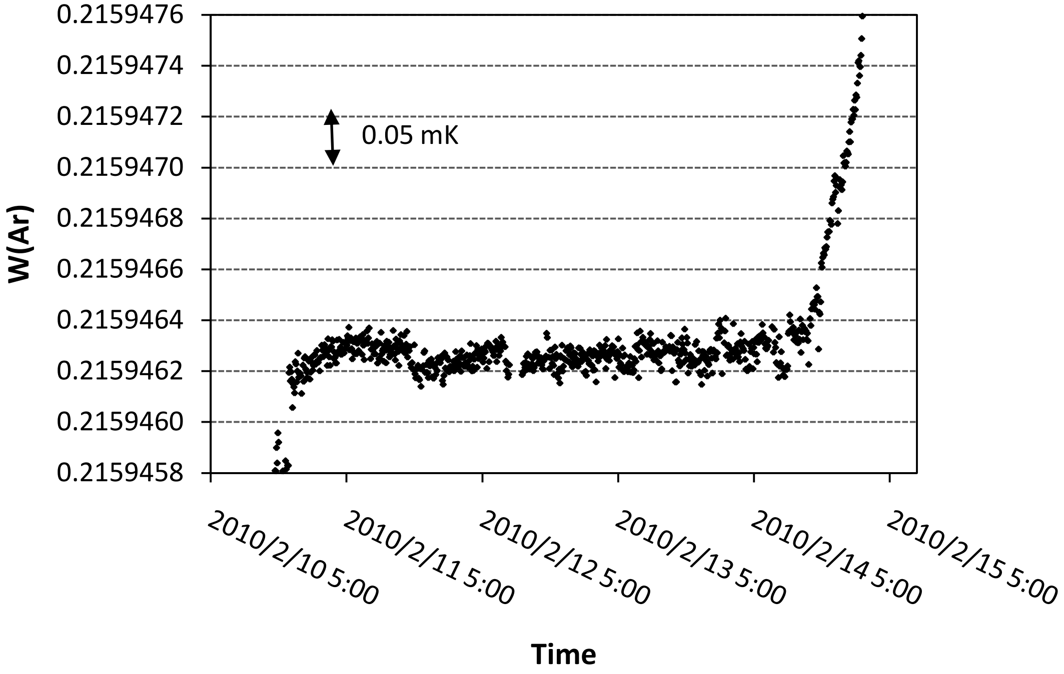

Figure 2. Plateau with the maintenance temperature 20 mK above the triple point of argon.

The immersion profile of the argon system re-entrant wells was tested in this study. One 25 Ω SPRT (Fluke model 5681) was placed at the bottom of a re-entrant well. It was measured with currents of 1.0 mA, 1.414 mA, and 1.0 mA again to correct for self-heating. After testing at the bottom of the re-entrant wells, the SPRT was raised 10.0 mm above the bottom and measured again. This testing process was repeated at heights of 20, 30, 40, 50, and 60 mm above the bottom. After testing at the height of 60 mm, the SPRT was lowered back down to the heights of 50, 40, 30, 20, 10, and the bottom. After testing, the mean value of the two measurements at each height was calculated to obtain the immersion profile of the re-entrant wells.

Figure 3 shows the tested immersion profile of one of the four re-entrant wells compared with the ITS-90 hydrostatic values. It can be seen that the tested results agree with the ITS-90 hydrostatic values in the range from the bottom of the re-entrant well to a height of 30 mm. The discrepancy increases with height after 30 mm. The closer the SPRT is to the top of the central argon cell, the lower the measured temperature is. The reason is that the heat transfer of the liquid nitrogen through the stem of the SPRT and the wall of the re-entrant wells cools down the upper area of the re-entrant wells.

The four re-entrant wells in the triple point of argon system allow calibration of up to four SPRTs at the same time. Temperature homogeneity of the re-entrant wells is very important for calibration accuracy and consistency. In order to test the temperature homogeneity of the four re-entrant wells of this argon system, the testing was carried out using three SPRTs (serial numbers 1614, 4223, 4227). The fourth re-entrant well was used to monitor the triple point plateau of the argon system during the overall testing process. Each of the three re-entrant wells was tested three times using three SPRTs by moving the three SPRTs among the three re-entrant wells. The testing results are presented in Table 1. It can be seen that the temperature difference among the three re-entrant wells is very small. The largest difference is 0.12 mK between re-entrant well #1 and #2. This means that the temperature is consistent among the re-entrant wells.

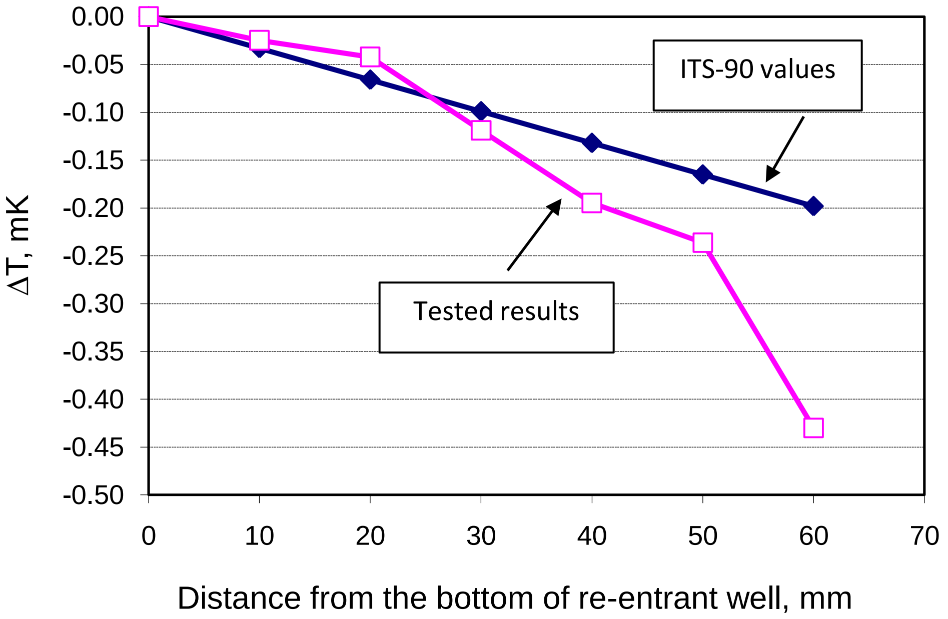

Figure 3. Comparison of the tested immersion profile of the re-entrant wells and the ITS-90 hydrostatic values.

Table 1. Temperature consistency testing of the re-entrant wells.

	Re-entrant well #1	Re-entrant well #2	Re-entrant well #3
SPRT 1614 (W(Ar))	0.215946497	0.215946683	0.215946612
SPRT 4223 (W(Ar))	0.216576694	0.216576163	0.216576577
SPRT 4227 (W(Ar))	0.216581826	0.216581929	0.216581662
Average (W(Ar))	0.216368339	0.216368258	0.216368283
Difference (W(Ar))	0	-8.06275E-08	-5.54852E-08
Difference (mK)	**0**	**-0.018**	**-0.013**

The estimated uncertainties of the triple point of argon system are listed in Table 2. The purity of the argon used in the argon system is 99.9999%. The estimated uncertainties (k=2) are 0.25 mK for the triple point of argon plateau.

Table 2. Estimated uncertainties of the triple point of argon system.

Source of uncertainty	Value (mK)
Resistance reading (A)	0.020
Reproducibility (A)	0.100
Total A	**0.102**
Impurities (B)	0.015
Hydrostatic correction (B)	0.030
Pressure correction (B)	0.050
Immersion (B)	0.010
SPRT self heating (B)	0.020
Propagated from TPW (B)	0.020
Bridge non-linearity (B)	0.010
Total B	**0.068**
Total standard uncertainty	0.123
Expanded uncertainty (k=2)	**0.246**

4. Modeling of heat transfer of the argon system parts

In order to study the heat transfer process during realization of the triple point of argon and to investigate the influence of the experimental conditions on the thermal equilibrium of the argon system, a finite element analysis (FEA) modeling of the heat transfer of the argon system was carried out by using the FEA program ABAQUS. Figure 4 shows the mesh of the simulated central assembly of the argon system. The simulated central assembly includes the four re-entrant wells, the vacuum can, the heater shield, and the central argon cell (also see Figure 1). The central assembly are immersed in liquid nitrogen.

The heat transfer of the argon system is a quite complex process. It includes heat power provided from the heater shield, heat conductivity of all of the shields and the four re-entrant wells, convection between the shields and liquid nitrogen outside of the simulated central parts and the condensed argon inside the central argon cell, and radiation of the shields' surfaces. All of these sources of heat transfer will influence the thermal equilibrium and the temperature distribution of the central parts. In the simulation, the liquid nitrogen temperature is assumed to be -196.5°C. The initial temperature of the whole central assembly is assigned to be -196.5°C. It is also assumed that in the beginning of the simulation, the central argon cell is full of solid argon with the temperature of -196.5 °C, which is the same as that of the liquid nitrogen outside after overnight freezing.

Two analysis steps are used in the simulation. The first step is the heating process which is used to quickly raise the temperature of the whole central assembly to the temperature of the triple point of argon by using a heating power of 12 watts at the outside surface of the heater shield. The second step is to maintain the temperature of the heater shield at -189.324 °C (20 mK above the triple point of argon) during the plateau of the triple point of argon to keep the thermal equilibrium in the three processes: cooling from the outside liquid nitrogen, heating from the heater shield, and the phase transition from solid and liquid argon to argon gas occurring inside the central argon cell.

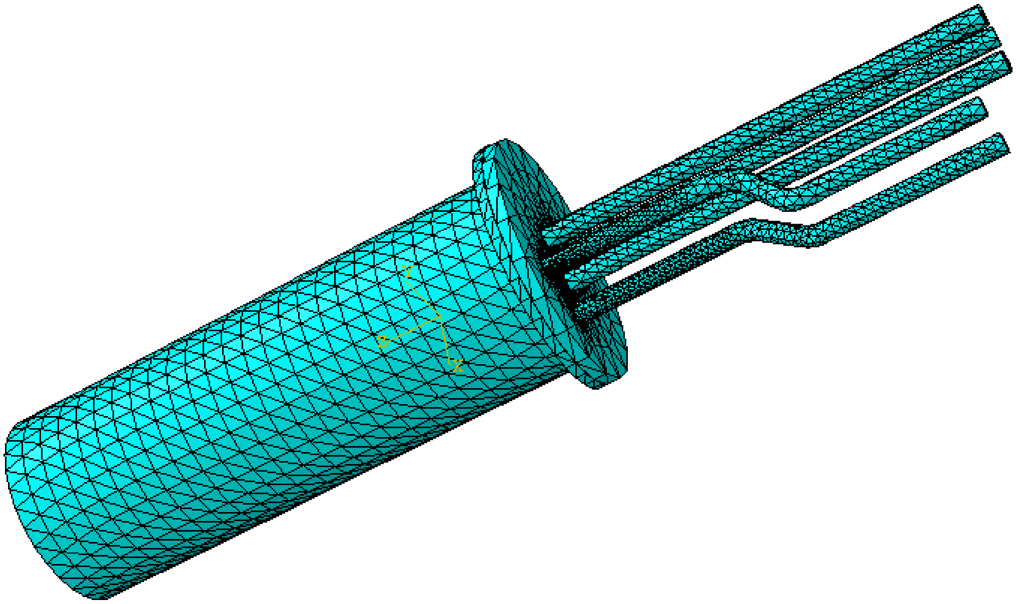

Figure 4. FEA mesh of the simulated central assembly of the argon system.

Figure 5 shows the simulated temperature distribution of the heater shield and the central argon cell when the argon system reaches thermal equilibrium at the triple point of argon. Figure 6 is the simulated temperature of the four re-entrant wells. It can be seen from Figure 5 that the temperature of the four re-entrant wells is uniform except for the top area close to the top lid. Figure 6 shows that the temperature of four re-entrant wells is consistent because of the symmetric structure of the four re-entrant wells. The simulation results agree well with the experimental results. If the heater shield's temperature is controlled at a higher value (e.g. 100 mK above the triple point of argon), the simulated temperature of the four re-entrant wells is the same. The only difference is that the plateau duration becomes shorter because more heat is transferred to the central argon cell, inducing a faster phase transition of argon.

According to the experimental and simulation results, the maintenance temperature has a great influence on the duration of the argon triple point plateau. The closer the maintenance temperature is to the triple point of argon, the longer the plateau lasts. The recommended maintenance temperature is 20 to 60 mK above the triple point of argon in order to obtain a stable and flat plateau that can last a reasonably long duration.

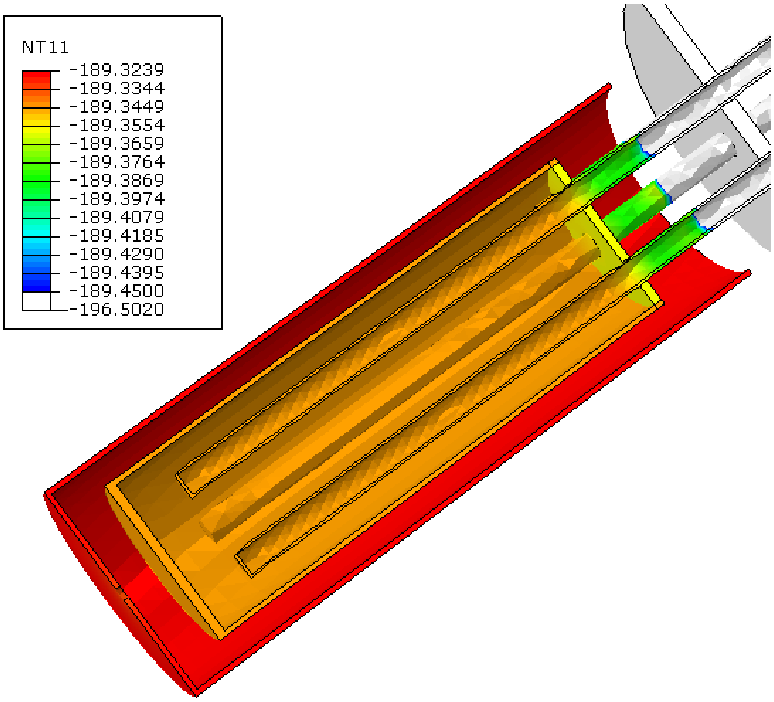

Figure 5. Simulated temperature of the heater shield and the central argon cell.

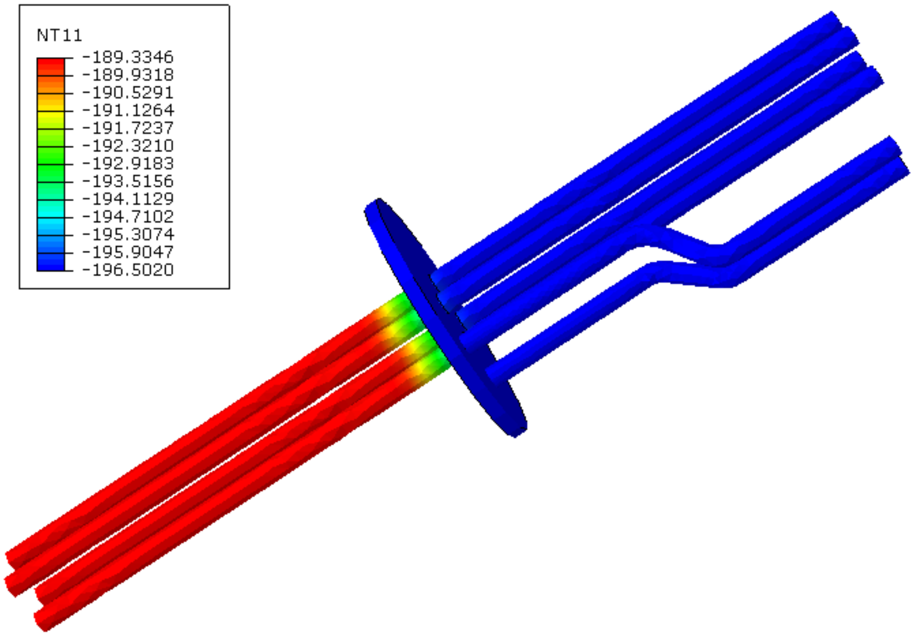

Figure 6. Simulated temperature of the four re-entrant wells.

5. Conclusions

A new triple-point of argon system was developed to realize the triple point of argon for the calibration of long-stem SPRTs. The triple-point of argon plateau can be easily realized with the high-accuracy temperature controller. This system was tested at different experimental conditions. The testing results show that the triple point of argon plateau can be as long as 100 hours with the temperature change less than 0.05 mK when the maintenance temperature is 20 mK above the triple point of argon. The temperature is consistent among the multiple re-entrant wells. The estimated uncertainty of the argon system is 0.25 mK (k=2). The FEA simulation of the heat transfer of the argon system shows that the temperature is consistent in the four re-entrant wells. The temperature of the re-entrant wells is the same with different maintenance temperatures, but the duration of the triple point of argon plateau becomes shorter at a higher maintenance temperature. In order to obtain a stable and reasonably long plateau, the optimal maintenance temperature is 20 to 60 mK above the triple point of argon.

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