INL/RPT-22-66266
Revision 0

Thermocouple Testing in Support of the AGR-5/6/7 Experiment

March 2022

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INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance, LLC

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Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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INL ART Program

March 2022

Technical Reviewer: (Confirmation of mathematical accuracy, and correctness of data and appropriateness of assumptions.)

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ACKNOWLEDGEMENTS

This work is supported by the Department of Energy (DOE) Advanced Reactor Technologies Program at Idaho National Laboratory under the U.S. Department of Energy Contract DE-AC07-05ID14517.

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SUMMARY

Temperature measurement is a challenging aspect of very high-temperature irradiation experiments because commonly used high-temperature commercial thermocouples (TCs), such as platinum-rhodium (Types S, R, and B) and tungsten-rhenium (Type C), suffer dramatic drift from neutron-induced transmutation. As a result, these types of TCs, which are used routinely for industrial temperature measurements outside the reactors, are used only in very special circumstances for reactor experiments. Conversely, because of their low-neutron cross sections, Type N TCs are affected to only a limited extent by neutron irradiation. However, the use of these nickel-based thermocouples is limited when the temperature exceeds 1050°C due to drift arising from minor alloying elements migrating from the thermocouple's metal sheath to the thermoelements. This change in the composition of the thermo-elements results in significant decalibration of the signal.

The issues described above were recognized during the early planning stages of the final AGR experiment (designated AGR-5/6/7), and a TC furnace testing program was performed over a 7-year period (2014–2019, 2021) to first select and then characterize the best thermocouple set for the high-temperature regions of the AGR-5/6/7 irradiation experiment. The calculated temperature range of the AGR-5/6/7 experiment was 600–1500°C. For temperatures below 1000°C, standard Type N thermocouples were deemed adequate. The furnace testing campaign identified two thermocouple types suitable for measuring temperatures above 1000°C: a Mo/Nb TC developed at Idaho National Laboratory (INL) called high-temperature irradiation-resistant thermocouple (HTIR-TC) and a Type N TC developed by Cambridge University (called herein Cambridge Type N), which featured a custom high-nickel alloy sheath.

One of the original goals of the furnace testing program was to identify a thermocouple capable of low-drift operation near the peak temperature expected in AGR-5/6/7 (i.e., about 1400°C). The HTIR-TC design appeared promising in this regard; however, a manufacturing difficulty proved to be a barrier, and instead, the furnace testing focused on drift performance at 1250°C. The manufacturing difficulty was that the Nb sheaths of the HTIR-TCs experienced extreme embrittlement when heat treated at 1600°C or greater. Heat treatment is needed to stabilize the electromotive force (EMF) output of this TC type, and the higher the heat treatment temperature, the higher the peak temperature of stable operation. Because of the sheath embrittlement, the heat treatment temperature had to be lowered to 1450°C, resulting in a stable operating temperature of about 1250°C.

One of the successes of the furnace testing program was identification of a shortcoming in the heat treatment procedure traditionally used in the production of HTIR-TCs—the entire length of the HTIR-TC sensor that would be at elevated temperature during operation was not being heat treated during the manufacturing process. Instead, only the part of the sensor expected to experience temperatures above 1000°C was being heat treated. The problem manifested when the thermocouples were removed from the heat treatment furnace and placed in another furnace with a different geometry. At that point, their indicated temperatures would be widely scattered, but mostly in the negative direction. The solution was to heat treat the entire heated length of the sensor. Since the deepest immersion depth in the AGR-5/6/7 experiment was

about 40 in., a heat treatment length of 48 in. was used. After implementing this change, thermocouples that were moved into a new environment with a different temperature profile (i.e., a different furnace) produced more accurate temperature measurements.

Although assembly of the AGR-5/6/7 experiment was completed in September of 2017 (and irradiation was started in 2018), furnace testing of thermocouples continued in 2018 and 2019. The main purpose of this testing was to establish very long-term drift characteristics of the HTIR and Cambridge Type N thermocouples installed in the experiment. Representative thermocouples from the same lots as those installed in the AGR-5/6/7 experiment were used. Additionally, thermocouples of different designs, (particularly variations on the HTIR-TC design) were "piggy-backed" on this testing program to provide insights for instrumenting future very high-temperature irradiation experiments. This 2-year testing program demonstrated that HTIR-TCs and Cambridge Type N TCs could operate at 1250°C for up to 10,000 hours (and in some cases longer) while experiencing negative drifts on the order of 2–4°C/1000 hours. This performance was considered acceptable given the extreme operating environment the sensors faced.

A significant manufacturing improvement was identified in 2019 related to HTIR-TCs. It was discovered that if HTIR-TCs are wrapped in Nb foil prior to heat treatment they maintain full ductility, even when heat treated to 1600°C or 1650°C. This discovery came too late to be of use to the AGR-5/6/7 experiment, but will likely extend the stable temperature range of HTIR-TCs to 1450°C or more for future experiments.

When the maximum stable temperature of the HTIR-TCs installed in AGR-5/6/7 was determined to be about 1250°C rather than 1400°C, the question naturally arose as to whether the experiment would be adequately instrumented. It was decided that this would be acceptable, because only a small fraction of the TCs in AGR-5/6/7 were expected to operate above 1250°C. There was little choice in this regard because assembly of AGR-5/6/7 began the second quarter (2Q) of 2017, and the TC set had previously been established in early 2017 to meet this schedule. Still, even the highest temperature capsules in AGR-5/6/7 were recognized to have zones with temperatures <1100°C, and TCs with proven low-drift rates could be put in these locations. HTIR-TCs would therefore still be placed in locations with temperatures greater than 1250°C, and these TCs were expected to provide reliable temperature information for only a few months (or weeks depending on how much above 1250°C the temperature was). The thermal model was then planned to be "tuned" to these TCs in the first week or two of full temperature operation, and thus these "sacrificial sensors" would still have significant value. The downside of this strategy was a greater reliance on thermal model calculations to project peak fuel temperatures in the latter stages of the experiment.

Given this line of reasoning, the performance of the HTIR-TCs installed in AGR-5/6/7 in temperature zones above 1250°C was somewhat of a mystery during experiment irradiation. With one exception, these TCs did not show obvious signs of negative drift, as would be expected from the furnace testing program, but instead their readings stayed flat or gradually climbed. Furthermore, these HTIR-TCs produced temperature readings well above the thermal model predictions and the deviations increased as irradiation proceeded. The question arose as to whether perhaps these HTIR-TCs might be drifting up. Although several HTIR-TCs had exhibited positive drift in the furnace testing program, none of those using the materials and heat treatment of the lot that went into AGR-5/6/7 showed positive drift. Only three examples of this type were tested in the furnace 2017–2019. It was decided to make an additional three HTIR-TCs with the same materials and manufacturing technique as those which were installed in AGR-5/6/7, and run a furnace test profile between 1250°C and 1450°C. The result of this final furnace test program (conducted in 2021) was that all three HTIR-TC examples drifted down, and their drift profiles were remarkably similar.

By the end of this final furnace test (a total of 4,100 hours), the three TCs exhibited an average negative drift of roughly –85°C, adding weight to the conclusion that the HTIR-TCs in AGR-5/6/7 likely did not experience positive drift.

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CONTENTS

FIGURES

TABLES

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ACRONYMS

AGC	Advanced Graphite Capsule
AGR	Advanced Gas Reactor
ART	Advanced Reactor Technologies
ATR	Advanced Test Reactor
DOE	Department of Energy
ECAR	Engineering Calculation and Analysis Report
EIL	Energy Innovation Laboratory
EMF	electromotive force
HTGR	High-Temperature Gas-Cooled Reactor
HITR-TC	High-temperature irradiation-resistant thermocouple
HTTL	High Temperature Testing Laboratory
ILC	Idaho Laboratories Corporation
INL	Idaho National Laboratory
IRC	Idaho Research Center
MIC	mineral-insulated cable
NIST	National Institute of Standards and Technology
TC	thermocouple
TRISO	tri-structural isotropic

Thermocouple Testing in Support of the AGR-5/6/7 Experiment

1. INTRODUCTION AND BACKGROUND

By far the most common type of thermocouple (TC) used in irradiation experiments is made from mineral-insulated cable (MIC). Figure 1 below shows the major components of this sensor type.

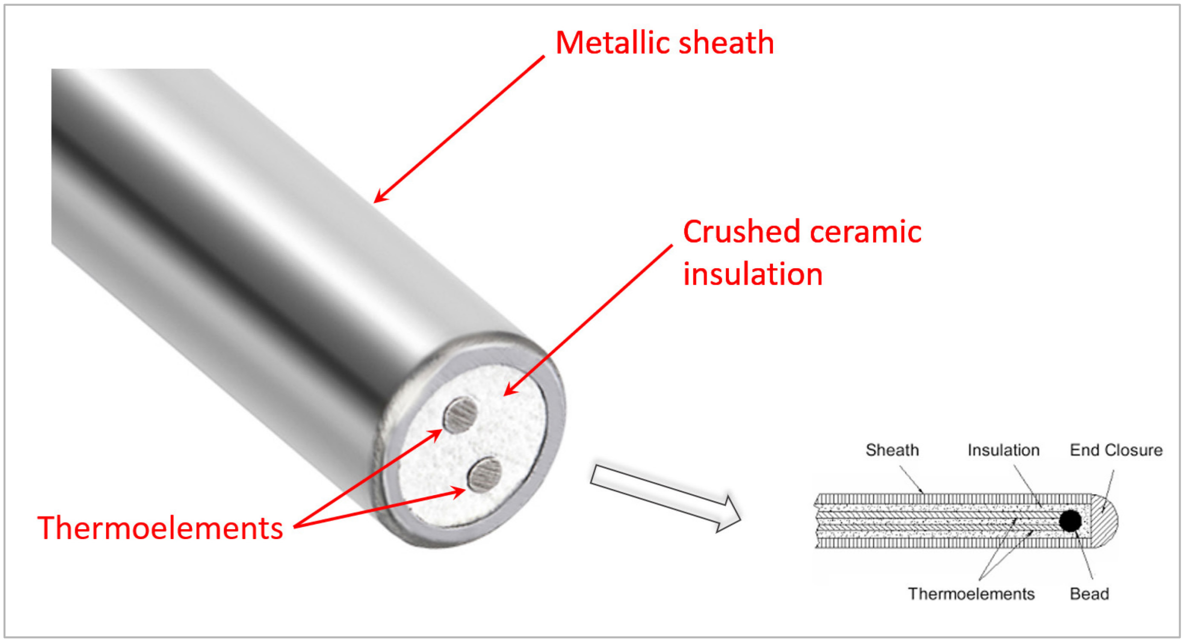

Figure 1. Mineral-insulated cable (MIC) formed into a thermocouple sensor.

The left side of Figure 1 shows the four major components making up MIC: the thermoelement wires (accounting for two components), the crushed ceramic insulating material, and the sheath. As noted in the lower-right corner of the figure, the two thermoelements must be joined together (with a "Bead"), and then an "End Closure" is welded over the end of the metallic sheath to make a hermetically sealed sensor capable of withstanding harsh environments. The four components working together are required to make a functional and accurate temperature-measurement sensor, suitable for use in a reactor experiment.

Very high-temperature in-core irradiation experiments create unique challenges for TC-based temperature measurements. While commercial TCs are available that use platinum-rhodium thermoelements (Types S, R, and B) that can tolerate temperatures up to 1700°C, or tungsten-rhenium TCs (Type C) which are capable of even higher temperatures, these high-temperature industrial TCs suffer rapid decalibration due to transmutation of the thermoelements from neutron absorption. For lower temperature applications, up to roughly 1000°C, Type N TCs (and Type K) are affected by neutron irradiation only to a limited extent (Servini 2013). Outside of special designs (discussed later in this report), the use of these nickel-based TCs is limited when the temperature exceeds 1050°C due to drift related to phenomena other than neutron irradiation.

This situation was recognized at the beginning of the TRISO-fuels program (circa 2005) and indicated that something other than standard commercial TCs would be needed.

1.1 Thermocouple Development and Testing Prior to AGR-1 Experiment

Beginning in 2004, serious efforts were undertaken at INL to pursue a TC design long recognized as promising for high-temperature neutron irradiations (Rempe 2006). This TC design is based on molybdenum and niobium thermoelements. The promise of this element couple for high-temperature reactor experiments is based on the high melting temperatures of Mo and Nb (2617°C and 2468°C, respectively) and the low thermal-neutron absorption cross sections of both elements (2.65 barns and 1.15 barns respectively).

In addition to the Mo/Nb TC design, which was later named high-temperature irradiation-resistant thermocouple (HTIR-TC), several variations on the standard Type N design were deemed worthy of investigation. Type N thermoelements themselves are a variation on the much more common Type K thermoelements. The Type N variation was developed in response to Type K's recognized lack of electromotive force (EMF) stability at high temperature (>900°C).

The "standard" Type N design consists of Inconel 600 sheath, crushed MgO insulation, and the Type N thermoelements. These thermoelements are designated "Nicrosil" (14% Cr, 1.2% Si, balance Ni) and "Nisil" (4.3%Si, 1% Mg, balance Ni).

Of the four components making up a Type N MIC TC, only two are available for design variations—the sheath and the insulation (the two thermoelements cannot be modified or the TC would no longer be Type N). For these special Type N designs, the primary adjustable parameter is the sheath composition. High-temperature Type N (and Type K) most commonly utilize an Inconel 600 sheath. Drift in Type N TCs at temperatures above about 1050°C have been traced to minor elements in the I600 alloy, such as Mn, migrating by solid-state diffusion from the TC's sheath to the thermoelements. This change in the thermolement compositions results in significant decalibration of the signal (sometimes more than 100°C).

A vendor search conducted in 2005 identified several Type N TC manufacturers that claimed to have identified sheath materials with low-drift characteristics at temperatures approaching 1250°C. It was determined that a long-term test should be conducted to evaluate drift in both the HTIR-TC under development, and these Type N designs with claims of superior performance (Rempe 2008). This test was conducted at a furnace temperature of 1200°C over a period of approximately 3600 hours (about 5 months). Figure 2 shows the results of this testing.

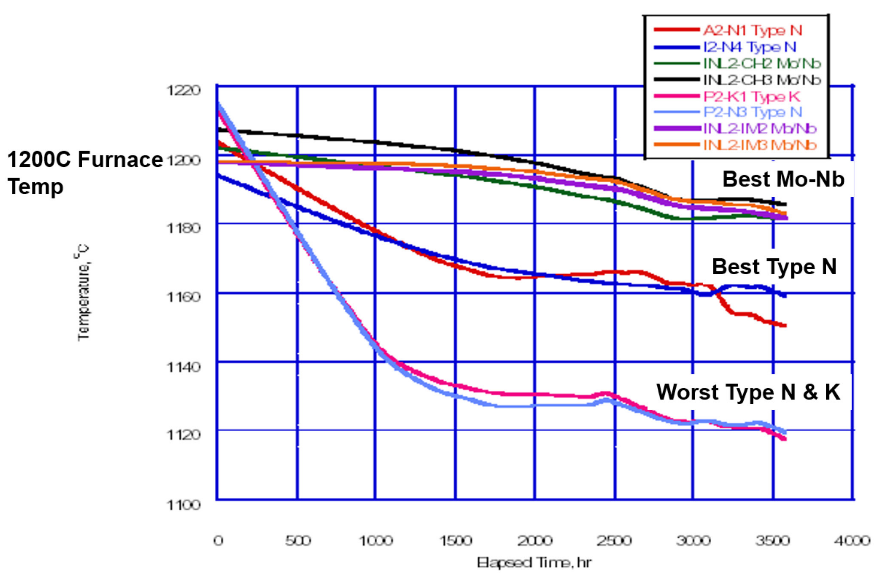

Figure 2. Summary of long-term test conducted at INL in 2005.

As illustrated in the figure, some Type N designs did distinguish themselves from their peers; but none were deemed stable enough to operate at the peak temperatures expected for AGR-1 (about 1200°C). The HTIR-TC design was clearly superior to even the best Type N variations. Standard Type N TCs were therefore placed in the lower temperature regions of AGR-1, and an HTIR-TC was placed in the center of the fuel stacks of some capsules (the highest temperature location in the capsules), as well as in other elevated temperature locations in the capsules, where room was available.

1.2 Thermocouple Performance Summary from the First Three AGR Experiments

The AGR-1 experiment incorporated 19 TCs, three of which failed during assembly of the experiment. Of the 16 that survived assembly, eight were HTIR-TCs and eight were Type N. The HTIR-TCs were placed in the hottest zones of the experiment and the Type N TCs were placed in cooler zones.

Based on observed temperature readings during the irradiation, the performance of the HTIR-TCs in the AGR-1 experiment were generally very good (Collin 2015). The peak temperature recorded by any one HTIR-TC was just under 1200°C, with the majority of indicated temperatures falling in the 1000°C to 1150°C range. There were no obvious signs of drift in the HTIR-TCs used in the AGR-1 experiment. However, it is difficult to accurately detect drift in an irradiation experiment because there is no solid reference against which TCs can be compared. TCs can be compared against each other, but this is not a fool-proof method because TCs of identical construction operating at similar temperatures tend to drift in a similar manner. TCs can also be compared against the thermal model, but the model can gradually experience its own deviation from the actual conditions in the capsules as the experiment progresses and material properties change, and clearances between components change.

As for the Type N TCs used in AGR-1, although they suffered a high open-circuit failure rate, those that survived showed little signs of drift. This can be attributed to their relatively modest operating temperatures, which were in the 700°C to 900°C range (where they would be expected to be drift free).

The second experiment in the AGR series, AGR-2, was configured very similarly to AGR-1, except for the TC complement. All TCs in the AGR-2 experiment were Type N, and 2 mm in diameter (slightly larger than the 1.6 mm TCs used in AGR-1) (Collin 2018). In addition to being larger, the TCs in AGR-2 were sheathed with pure Nb. This was done to protect the fuel compacts from transition elements found in Ni-alloys (such as Ni, Cr, Fe) and to avoid solid-state diffusion of sheath elements to the thermoelements. Given the larger sheath diameter, there was only room for two TCs per capsule except for capsule 6, which was able to accommodate five TCs because of its location at the top of the test train. (The number of TCs is limited by the cross-sectional area of the thru-tubes, which are the conduits that pass through the capsule bodies, enabling instrumentation and gas lines from lower capsules to find their way through the capsules above.)

The Type N TCs in AGR-2 generally measured temperatures in the 800°C to 1000°C range, with one Type N TC indicating temperatures as high as 1100°C for brief time periods. Comparing the TCs against each other and against the model, provides little indication of drift, high rates of open circuit failure occurred, especially in the latter stages of the experiment.

The gas lines in AGR-2 were also niobium. Failures in these gas lines coupled with the eventual failure of all TCs in AGR-2 led to the conclusion that the Nb TC sheaths likely became embrittled due to minute quantities of residual O_2 in the gas streams. As a result, a decision was made not to pursue this sheath type for Type N TCs going forward; however, Nb was retained as the sheath for HTIR-TCs because a ductile refractory metal is needed for temperatures above 1200°C and Nb is the best refractory option.

The AGR-3/4 experiment was in a larger irradiation position than AGR-1 and AGR-2. This allowed for an increase in the number of capsules (12) and TCs (27) (Collin 2016). All TCs in AGR-3/4 were standard design Type N and 1 mm in diameter. All 27 TCs survived assembly and 22 survived the entire irradiation period. The majority of these TCs operated between 600°C and 700°C until the last two cycles, at which point planned temperature increases of 50°C to 100°C were imposed. Two TCs operated at about 1000°C, and one of these showed distinct signs of negative drift (after 240 days of irradiation). The AGR-3/4 test again confirmed that Type N was a good choice for temperature measurements in the 600 to 800°C range, but raised questions about long-term stability for this type running near 1000°C.

1.3 Thermocouple Planning for AGR-5/6/7

The AGR-5/6/7 fuel temperature distribution objective is shown below in Table 1 (Palmer 2016).

Table 1. Planned fuel particle temperature distribution for AGR-5/6/7 experiment.

AGR-5/6	
Desired fraction of particles per temperature range	Number of Particles Based on 500,000 total
30% <900°C	150,000
30% 900°C–1050°C	150,000
30% 1050°C–1250°C	150,000
10% 1250°C–1350°C	50,000
Total	500,000
AGR-7	
Temperature Range	Minimum Number of Particles
1350°C–1500°C	50,000

Although TCs are never directly inserted into TRISO-fuel compacts, the design of the AGR-5/6/7 capsules was such that there were locations in the AGR-5/6/7 capsules, which were suitable for TC insertion, and which would experience temperatures very near the fuel temperatures shown in Table 1. These temperatures would be 200–300°C higher than any measured in the previous AGR experiments. Recognizing both the limitations of existing thermometry to measure such high temperatures and that the previous testing and in-core TC performance were inadequate for temperature ranges this extreme, the sponsor of the AGR-5/6/7 test supported a development and testing program for low-neutron cross section TCs capable of low-drift operation at temperatures above 1100°C. This program included additional development of INL's HTIR-TCs based on molybdenum/niobium thermoelements, as well as testing a new set of Type N TC designs that had been identified in the intervening time between 2005 and 2014.

Initially (2014 campaign), testing focused on new Type N TC designs, rather than HTIR-TC development. However, as testing progressed through 2015 and 2016, it became clear that there was a calibration issue with the established HTIR-TC manufacturing process*,* since the HTIR-TCs would typically produce a lower voltage than they should when placed in the long-term testing furnace. A fix for the calibration issue was identified in 2016 (see Section 3.3) (Palmer 2019).

By the end of 2016 a TC suite for the AGR-5/6/7 experiment had been identified and procurement initiated. This locked in the TC complement for AGR-5/6/7. However, the furnace testing program continued through 2017–2019. The purpose of this additional testing was to characterize the long-term drift characteristics of the TC lots used in the AGR-5/6/7 irradiation experiment as well as to test TCs of different designs (particularly variations on the HTIR-TC design) to provide insights for instrumenting future very high-temperature irradiation experiments.

1.4 Test Objectives

The major test objectives were as follows:

1. Evaluate drift performance of HTIR-TCs in the 1200°C to 1400°C range. As circumstances evolved the peak furnace test temperature was lowered to 1250°C.
2. Evaluate initial calibration of HTIR-TCs.
3. Confirm that changes made to HTIR-TC composition and manufacturing techniques resulted in an accurate, low-drift sensor.
4. Identify Type N TC types capable of low-drift operation in the 1050 to 1250°C temperature range.
5. Characterize the drift performance of the TC set installed in AGR-5/6/7 at temperatures between 1050 and 1250°C.
6. After completion of the AGR-5/6/7 experiment, furnace test a set of HTIR-TCs for drift performance at temperatures in the 1350 to 1450°C range.

Other, secondary, objectives were attached to the testing program such as evaluating the performance of Type K and Type S TCs at similar temperatures.

1.5 Experimental Approach

All tests were conducted in a tube furnace in Idaho Research Center (IRC) Lab C-15. The test setup for the initial (2014) campaign is shown in Figure 3 below. The test was conducted in a tube furnace with the TCs embedded in a block of graphite (see Figure 4). (Note that the Figure 4 photo was taken near the end of the 1150°C phase of the test, after the furnace had been shut down and the internal assembly removed.) The test atmosphere was argon at flow rate of 250 cc/min. The oxygen concentration as measured at the outlet of the furnace was always less than 1 ppb. Some oxygen contamination was noted at the end of the 2014 campaign (and measures were taken in subsequent tests to correct this problem).

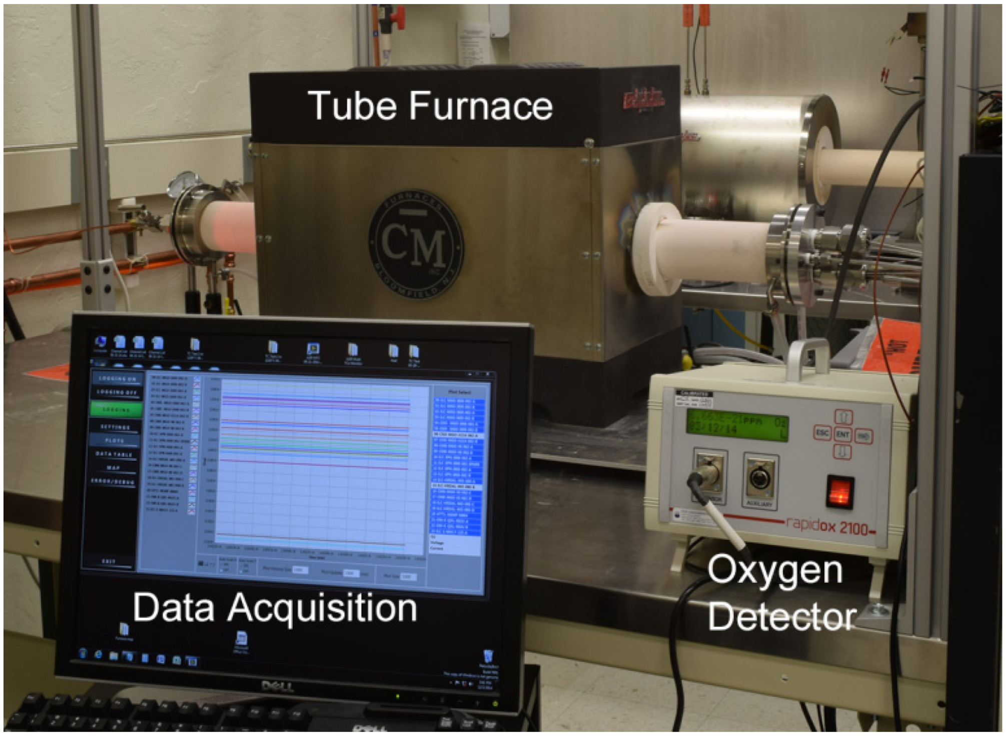

Figure 3. Experiment setup showing a tube furnace in operation, the data acquisition system, and an oxygen detector.

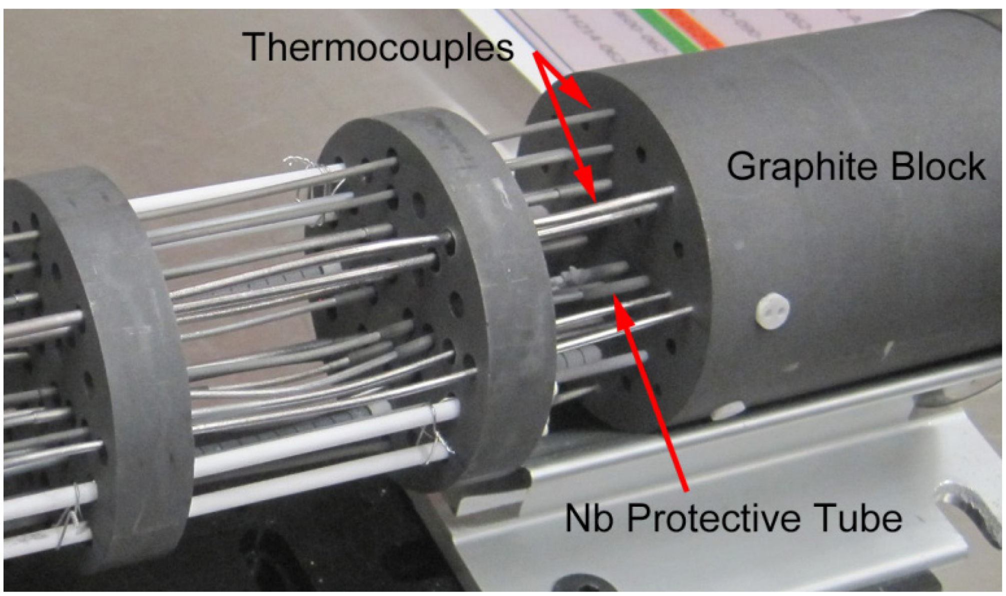

Figure 4. In-furnace assembly after 1,800 hours at 1157°C.

All TC tips were inserted 50 mm into the graphite block and supported periodically by graphite support disks.

In subsequent testing campaigns (i.e., 2015–2019, 2021) the internals consisted of two graphite blocks, a “hot block” and a “cold block” (see Figure 5). The hot block was aligned with the furnace midplane (i.e., by immersion depth) and thus experienced the highest temperature. The cold block was about 7 in. off the furnace midplane and thus experienced a considerably cooler temperature, roughly 200°C cooler. This arrangement allowed TCs to be tested at two temperatures simultaneously.

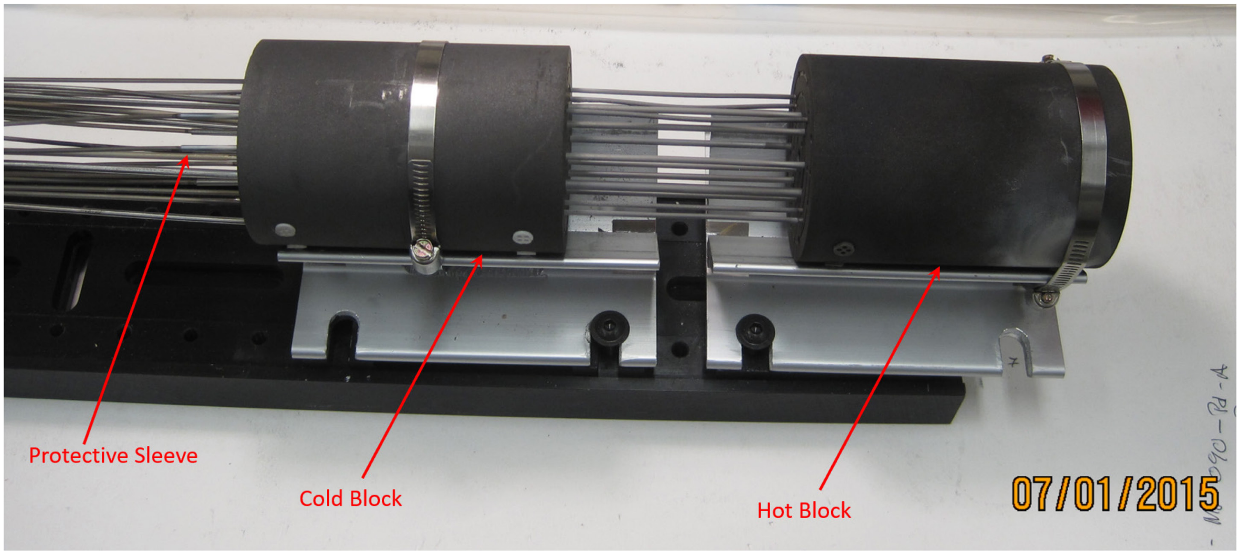

Figure 5. Furnace internals for 2015 testing (internals for subsequent testing were similar).

Figure 6 is a schematic which illustrates how the graphite blocks were arranged in the alumina tube, which passed through the furnace. The furnace was controlled by an exposed junction Type B reference TC placed between the heating elements and the alumina tube. This control TC effectively keeps the furnace temperature stable, but it does not represent the temperature in either of the graphite blocks. An additional Type B reference TC (hermetically sealed loose assembly) was placed in the hot block to measure the actual test temperature. During the 2015 testing campaign the Type B reference TC in the hot block was noted to be experiencing a negative drift.

This behavior was described to Jonathan Pearce of the United Kingdom's National Physical Laboratory (equivalent of the United States National Institute of Standards and Technology) who explained that typically Type B reference TCs are an exposed junction type operated in air (rather than the inert environment which was maintained in the furnace). The exposed junction design maintains a high degree of accuracy because the thermoelements slowly sublimate in the air environment, which maintains their purity.

Because of the many reactive components inside this furnace test, not the least of which are the MIC TCs, it must be a completely inert environment and must be sealed from the outside world. The Type B reference TCs were a sealed loose assembly design. Sublimation of the thermoelements did not occur and the thermoelements were subject to contamination from the sheath and insulator materials, causing the observed decalibration.

The solution to this dilemma was to install a very deep molybdenum thermowell in the furnace that terminated in the hot block. This allowed the reference TC to be retracted into the cooler zone during most of the experiment, except for brief periods (typically 1 hour per week) when temperature checks were taken. This arrangement was implemented in 2016 and remained the same for the balance of the testing.

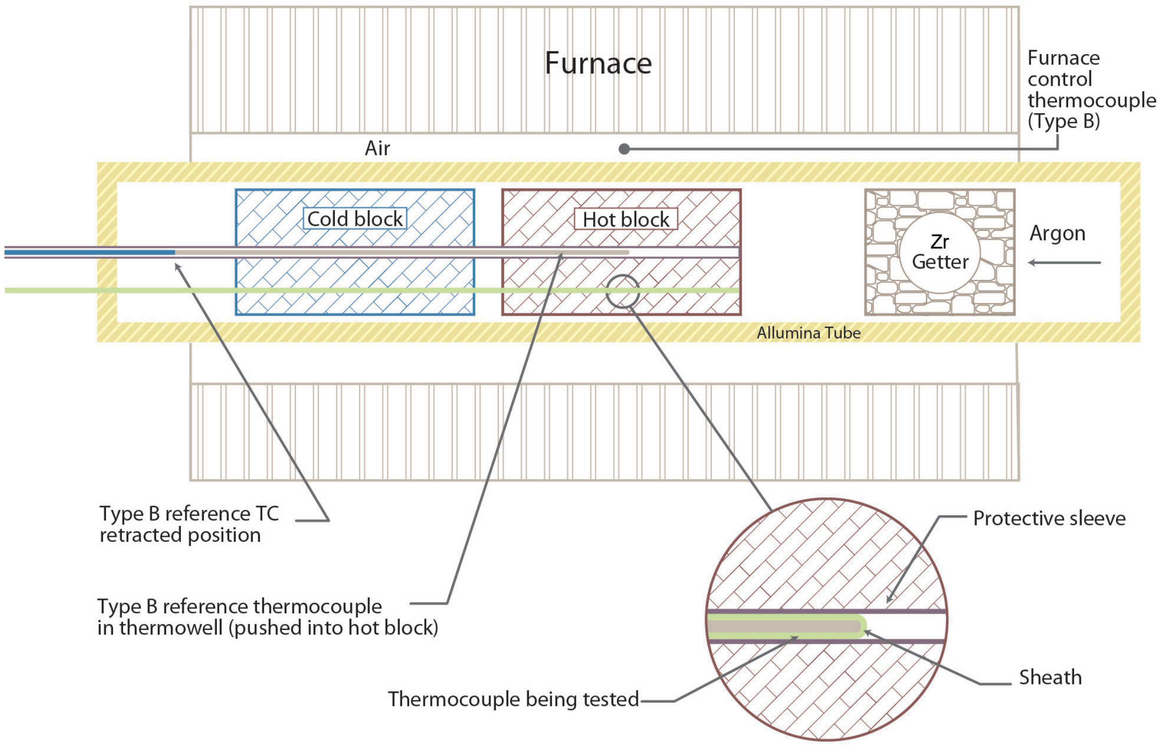

Figure 6. Furnace schematic.

Because oxidation occurred in the furnace in the 2014 campaign, Zr getter material was included in the right side of the alumina tube in subsequent tests (Figure 6). This getter material consisted of a quantity of scrap zirconium metal placed near the inlet of the furnace. By forcing the argon purge gas through this extremely active refractory metal, the gas was purified of all oxygen contamination.

2. SUMMARY OF EACH TEST CAMPAIGN

The test campaigns were conducted on approximately a calendar year basis. This section provides a summary of the year-by-year testing campaigns. These tests were conducted in support of the AGR-5/6/7 experiment design, but on a relatively informal basis. Temperature data were transmitted weekly to the AGR-5/6/7 design team and summary notes were produced. During the early test campaigns these notes were quite brief. In later years these notes evolved into test logs and were more detailed. These year-by-year summary notes and test logs have been reproduced in Appendixes A–G. In some cases, additional narrative and explanatory notes have been added to the test logs.

2.1 2014 Testing

Based on experience from the first three AGR experiments and the objectives of the AGR-5/6/7 experiment, which projected very high irradiation temperatures, the project decided to conduct a follow-on "long-term" test similar to that conducted in 2005 in support of AGR-1. This test included a variety of novel Type N designs, and a very small set of HTIR-TCs (see Appendix A and Palmer 2015 for additional detail).

Previous work and published literature demonstrated that among standard high-temperature, low-cross section TCs suitable for in-pile use, Type N TCs are more stable at high temperature than are Type K TCs. Type N TCs were used in all of the previous AGR tests. Therefore, the TCs being tested were compared against a "standard" Type N TC design with the following attributes:

- Size: 1.57 mm
- Sheath: Inconel 600
- Insulation: High purity MgO

The following variations on the standard Type N design were tested:

1. Larger size – 2.34 mm
2. Haynes 214 alloy sheath rather than Inconel 600
3. Spinel insulation ($MgAl_2O_4$) rather than MgO insulation
4. Special high-nickel sheath material developed at Cambridge University rather than Inconel 600
5. Loose assembly (hard-fired, uncrushed alumina insulation) with molybdenum sheath, similar to the design used in the AGR-1 top capsule.

Also, two examples of the then-current version of the HTIR-TC design were included in the test.

The temperature was ramped in a stepwise fashion as follows (listed temperatures are nominal):

- 2,060 hours at 1150°C
- 2,006 hours at 1200°C
- 201 hours at 1250°C
- 266 hours at 1300°C.

Near the end of the 1150°C test period, the furnace was shut down and its internals were removed (see Figure 4). At this juncture, two Type S with crushed insulation (typical MIC design), and two Omega Type K standard MIC TCs were installed. Both designs were 0.062-in. diameter.

The results of the 2014 testing campaign were as follows:

The first three variations on the Type N TC design listed above provided only a limited improvement in drift rate versus standard 1.57-mm Type N TCs. The fourth variation, with a special sheath material developed at Cambridge University, reduced the drift rate markedly; and this TC design offers the advantage of being just as flexible and physically robust as a standard Type N TC. The Cambridge design appears well suited for operating in the 1000–1200°C range. The fifth variation, with a loose assembly design, showed promise for being drift resistant, but needs further development to prevent open circuit failure when cycled repeatedly. The HTIR-TC design again demonstrated low-drift characteristics; however, the two examples supplied for this test suffered from calibration inaccuracy, and an early open circuit failure.

The two Type S TCs with crushed insulation, and two Omega Type K standard MIC TCs installed mid-way through the testing performed poorly (see Appendix A for further discussion).

2.2 2015 Testing

As was previously stated, the 2014 furnace testing was focused on Type N TCs and narrowed the candidates for incorporation into the AGR-5/6/7 test to Cambridge Type N, and Type N loose assembly. However, the 2015 campaign was more focused on HTIR-TCs. Specifically, 2015 was taken as an opportunity to test variations on the standard HTIR-TC design, although several other temperature sensors were again incorporated. See Appendix B for summary notes and test log.

The major results from the 2015 test program were as follows:

- There was huge scatter in the HTIR-TC readings in the hot block (almost 200°C), upon initial startup.
- None of the variations on the standard HTIR design appeared promising.
- Heat treatment of an additional 8 inches of two standard HTIR-TCs improved their calibration and drift performance markedly. (This presaged the 2016 discovery that the entire heated length of HTIR-TCs must be fully heat treated to achieve accurate calibration and low-drift performance.)
- A Pt/Pd loose assembly design was tested at the recommendation of Jonathan Pearce of the UK's National Physical Laboratory. This TC type appeared to have low-drift characteristics; however, our indicated temperatures were about 40°C off. Research on this TC type was discontinued because, although Pt and Pd have thermal-neutron cross sections lower than in Type C and Type B/S, they are not as low as cross sections of Mo/Nb HTIR-TCs, or the cross sections of Type N TCs.
- Cambridge Type N TCs were tested in a "cold block" running at temperatures between 1110°C and 1210°C at various times during the 2015 testing. The temperature of the cold block was not controlled directly by the furnace TC and so it was not possible to precisely determine drift. No significant drift was observed. Precise determination of drift of Cambridge TCs would have to wait until the test campaign of 2017.
- Four Type N loose assembly TCs were also tested in the cold block. Two of these had a Mo sheath and two had a Ni sheath. The Mo sheathed versions showed no propensity to drift, but they failed much earlier than the Cambridge Type N TCs. The Ni sheathed versions drifted downward more than 60°C and then failed.
- Because of the substantial scatter in the HTIR-TC readings upon initial startup, a test was run to verify the graphite blocks were producing isothermal conditions. Four standard Type N TCs were embedded in various locations in the hot block and run up to a temperature of 1185°C. All four TCs indicated within 1.5°C of each other, which provided assurance that the hot block was running isothermal enough for our testing purposes. These results can be assumed to apply to the testing performed using similar setups in subsequent years.

2.3 2016 Testing

The 2015 testing campaign showed there was something wrong with the HTIR-TC production process. As soon as the TCs were removed from the heat treat furnace and placed in another furnace with a different geometry, their indicated temperatures were widely scattered. The focus of 2016 furnace testing was on getting the materials and heat treat recipe for HTIR-TCs right. No Cambridge Type N TCs were tested in 2016, and the testing done on standard Type Ns was not very useful because the temperatures were too high, and they drifted dramatically.

Part way through 2016, the problem with heat treating only part of the HTIR-TCs' heated length was identified. However, this was not corrected until the last run of the year. As a consequence, the calibrations of all HTIR-TCs were –60 to –160°C off upon initial startup in the IRC furnace until the final furnace run of the year. See (Palmer 2019) for further discussion as to why HTIR-TCs heat treated in this manner tended to indicate low.

The testing was done primarily at 1385°C because this was near the high end of the expected temperatures in the AGR-5/6/7 test. This required a heat treatment of 1600°C or 1650°C. During the year, sheath material of choice for HTIR-TCs transitioned from Nb/1% Zr to pure Nb because it was thought that alloying with 1% Zr may have been contributing to calibration errors or drift. Heat treating pure Nb at 1600°C or 1650°C was later determined to have resulted in very brittle TCs. (This problem was solved in 2019 by encasing the TC with Nb foil during heat treatment.) See Appendix C for summary notes and test log.

2.4 2017 Testing

The AGR-5/6/7 test train was slated to begin assembly during Q2 of Calendar Year (CY) 2017. This made it necessary to pick a TC set at the end of CY 2016 because of the significant lead time in procuring (Type N) and producing (Type HTIR-TC) TCs. There was still a degree of uncertainty in the correct HTIR-TC recipe at this juncture, but based on the information available, the following design elements were selected: Pure Nb sheath, Al_2O_3 insulation, and 1450°C heat treatment formula (see Palmer 2019 for additional details). A production run of approximately 22 HTIR-TCs was made using this recipe.

Preproduction and production versions of HTIR-TCs were tested in 2017. The first test was conducted at 1315°C with preproduction versions of HTIR-TCs heat treated at 1650°C and 1450°C. The calibration of these two TCs was very good (within 1.5% of the furnace setpoint) upon startup, and this was the first set that exhibited good initial calibration performance in the IRC furnace. As expected, the unit that had been heat treated at 1650°C exhibited superior drift characteristics compared to the unit heat treated at 1450°C.

Thermal modeling of the AGR-5/6/7 experiment indicated that only a handful of TCs would experience a temperature greater than 1250°C, and so a decision was made to conduct the balance of the furnace testing at 1250°C (including 2018 and 2019 testing campaigns). This was a temperature at which both Cambridge Type N TCs, and HTIR-TCs heat treated at 1450°C were expected to perform reasonably well. This proved to be the case as both TC types drifted on the order of –3 to –4°C per 1000 hours of furnace operation. The basic problems with HTIR-TC construction and heat treatment were solved early in 2017. Further details of the 2017 furnace testing campaign may be found in (Palmer 2019) and Appendix D.

2.5 2018 Testing

Assembly of the AGR-5/6/7 test was completed September 2017; however, the furnace testing supporting this experiment continued for several reasons:

1. Just before assembly of AGR-5/6/7 began, the program decided that the Cambridge Type N TCs in the hottest part of the experiment should be protected by ZrO2 sleeves rather than Mo sleeves, because Mo sleeves had shown a propensity to crack. We had no furnace test data with ZrO2 sleeves and so this configuration was installed in the furnace.
2. Again, because of molybdenum's propensity to crack, Nb sleeves were selected to replace Mo in the cooler areas of the test for both the Cambridge Type N and standard Type N. Prior testing demonstrated that Nb sleeves resulted in higher drift rates versus Mo when tested at >1150°C, it was hoped that at a cooler temperature (~1080°C), Nb would not be deleterious.
3. To determine whether Nb/1% Zr would be a viable sheath material when combined with Al2O3 insulation. A TC of this design was tested in 2017, HTIR-TC-ZR1, and it drifted upward rapidly. To answer the question of whether this was an anomaly, or whether there was something inherently flawed with this combination; three more examples were tested in 2018.
4. To get more time on TC22 (HTIR-TC from the AGR-5/6/7 production lot), and more time on two Cambridge Type N TCs from the AGR-5/6/7 production lot.

The results from this testing were as follows:

1. ZrO2 sleeves had no deleterious effects. Drift performance of Cambridge Type N TCs with ZrO2 was just as good or better than those protected with Mo.
2. At the lower temperature of 1080°C, Cambridge Type N TCs exhibited no discernable drift when protected with Nb sleeves.
3. The HTIR-TCs with Nb/1% Zr sheaths showed a propensity to drift upward, but not nearly as much as was seen in the 2017 testing.

4. The HTIR-TCs and Cambridge Type N TCs selected from the production lots continued to perform very well with drift performance on the order of –4°C/1000 hours of operation at 1250°C.

Additional details and test logs for the 2018 test campaign may be found in Appendix E.

2.6 2019 Testing

The primary purpose of the 2019 furnace testing campaign was to get additional time on the TCs installed in 2018, with a goal to approach a time at temperature on the order of 450 days, which was the original target for the AGR-5/6/7 experiment. Ultimately, some TCs were held at temperature (1250°C) for more than 570 days, and so the original goal was considerably exceeded.

Additionally, for the 2019 campaign, a few new TCs were installed. Two additional Cambridge Type N TCs were inserted into the hot block, primarily to obtain better testing statistics, and two new HTIR-TCs, both heat treated at 1600°C, one with MgO insulation and the other with the now-typical Al_2O_3 insulation (designated HTIR-TC65 and TC70, respectively). Crushed MgO insulation is softer than crushed Al_2O_3, thus it is easier on the thermoelements during the swaging/drawing process. Therefore, if MgO insulation was found to provide equivalent drift performance, it may indicate that it would be a superior material for future experiments.

The results from the 2019 campaign were:

The surviving Cambridge Type N TC from the 2018 campaign operating at 1250°C (CAMB-TC-8) continued to exhibit low-drift characteristics and lasted through the entire testing period. The two new Cambridge Type N TCs operating at 1250°C drifted about –4°C/1000 hours.

The two new HTIR-TCs installed, HTIR-TC65 and HTIR-TC70, did not exhibit good calibration on startup (–20°C and +35°C respectively). HTIR-TC65 insulated with MgO suffered –15°C/1000 hours drift, while HTIR-TC70 drifted on the order of –2°C/1000 hours. This result coupled with the poor drift performance of HTIR-TC64 (also insulated with MgO), seemed to confirm that Al_2O_3 is a much better insulator for HTIR-TCs.

Additional details and test logs for the 2019 test campaign may be found in Appendix F.

2.7 2021 Testing

In 2021[a] the final furnace campaign supporting the AGR-5/6/7 experiment was conducted. The purpose of this campaign was to provide additional insight into the performance of HTIR-TCs operated at temperatures well above the 1250°C used in the 2017–2019 furnace testing campaigns. Specifically, several HTIR-TCs in the hottest zones of AGR-5/6/7 Capsules 1 and 3 provided indications in the 1300 to 1500°C range, and these indications were well above model predictions. One possible reason for this was that the TCs drifted up. If this was not the case, the conclusion is that some fuel in Capsules 1 and 3 ran 100 to 200°C above model predictions. During the 2015–2019 testing, there were a few examples of HTIR-TCs drifting up, but these were typically versions sheathed with Nb/1% Zr. No examples of HTIR-TCs from the lot used in AGR-5/6/7 were available (all had either been used in the irradiation experiment or for testing in 2017). In lieu of actual lot representatives, several HTIR-TCs were fabricated using the same materials, assembly technique, and heat treatment as was used for the lot that went into AGR-5/6/7.

Five TCs were produced, but only three functioned in the furnace. These were two examples with foil wrap to minimize brittleness and one without the foil wrap. All were pure Nb sheath, Al_2O_3 insulation, and 1450°C heat treatment.

[a] No furnace testing was conducted in the IRC furnace during CY 2020.

Upon initial startup of the IRC furnace, all three TCs read low (two significantly), based on their specific calibration curves (which were developed in the EIL furnace). The main purpose of the furnace test was to evaluate drift and so the readouts of all three TCs were converted to a standard calibration curve, and this brought them up into rough agreement with the furnace temperature (see Figure 7).

The testing plan was to approximate the temperature profile of TC-3-12 and TC-3-14 in Capsule 3 of the AGR-5/6/7 experiment. This would also roughly match the temperature profiles of TC-1-12, TC-1-13, and TC-1-14 in Capsule 1.

The heating profile was:

- 1,700 hours at 1250°C
- 1,730 hours at 1350°C
- 670 hours at 1450°C.

During the 1250°C heating phase, the three TCs drifted a minimal amount, about –5°C total, or –3°C/1000 hours (very comparable to the drift rate seen during the testing conducted 2017–2019 [see Sections 3.4 and 3.5]). During the 1350°C heating phase the drift was much more pronounced, about –22°C total, or –13°C/1000 hours. At 1450°C the drift rate was steeper yet, approximately –30°C total and –45°C/1000 hours.

One of the reasons for conducting this test was to see if there was much scatter in the drift performance among the tested thermocouples. As can be seen in Figure 7 below, the three TCs exhibited very similar drift behavior.

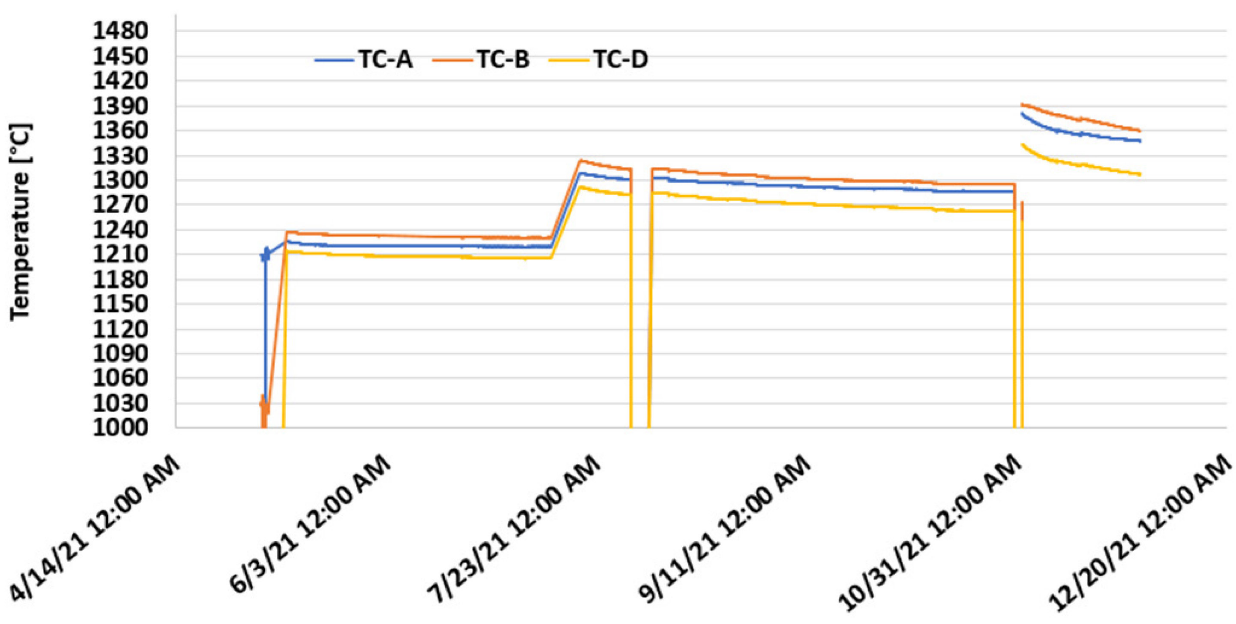

Figure 7. Temperature profile of three prototypical HTIR-TCs run at 1250°C, 1350°C, and 1450°C.

All three TCs drifted down and by about the same amount. Although they each had slightly different initial calibration on startup, the three curves track each other remarkably closely. This behavior implies that the difference between the AGR-5/6/7 TC measurements and the thermal model are likely not due to a spurious positive drift in the TCs.

Another way to look at this is that the initial startup calibration error was about –25°C (i.e., the average of the three TCs was 1225°C at 1250°C furnace temperature). At the end of the test the average reading of the three TCs was 1340°C at a furnace temperature of 1450°C, for a difference of 110°C. If we subtract the initial calibration error, the average negative drift was 85°C.

For additional discussion of the performance of these TCs as related to observations from Capsules 1 and 3, see Appendix G.

3. SUMMARY OF RESULTS

This section summarizes significant results from the entire test program described in the previous section. Some results were specifically related to the AGR-5/6/7 experiment, while others would be useful to users of high-temperature TCs in general. These are summarized in the following two sections.

3.1 Directly Related to AGR-5/6/7 Experiment

1. As configured for the AGR-5/6/7 test, both HTIR-TCs and Cambridge Type N TCs experience drift rates on the order of –2 to –4°C per 1000 hours at 1250°C.
2. Within the uncertainty limits of the test apparatus, Cambridge Type N TCs exhibited no drift over >10,000 hours at 1080–1090°C.
3. Standard Type N TCs exhibited a drift rate of –3 to –4°C per 1000 hours at 1080–1090°C (only two examples were tested, so this was a very small sample size).
4. With the exception of Type N loose assembly TCs, Cambridge Type N TCs are much superior in terms of drift at high temperature than any other Type N TC ever tested, including the testing conducted in 2005.
5. Some of the Type N loose assembly TCs were remarkably drift free, but this design suffered open circuit failures at a significantly higher rate than the crushed insulation versions. (See Appendix I for a description of loose assembly TCs.)
6. Heat treatment of HTIR-TCs must be over the entire “heated length” they will see in service (Palmer 2019).
7. A HTIR-TC heat treat temperature of 1600°C, or greater, reduces the drift rate compared to 1450°C, however 1450°C heat treat versions were used in the AGR-5/6/7 test because of embrittlement of sheath material at the higher heat treat temperature (see following entry).
8. If Nb foil is wrapped around HTIR-TCs prior to heat treatment, they retain full ductility even after being heat treated at 1600–1650°C (this discovery came after the AGR-5/6/7 test began irradiation).
9. HTIR-TCs with Al_2O_3 or HfO insulations demonstrated good performance when combined with pure Nb sheath material.
10. HTIR-TCs with MgO insulation consistently performed poorly by exhibiting high rates of negative drift.
11. Nb/1% Zr sheath material combined with HfO insulation performed well, but the same sheath combined with Al_2O_3 correlated with substantial positive drift.
12. To provide more insight into the behavior of the HTIR-TCs installed in AGR-5/6/7, a set of similarly constructed TCs were tested for 4,100 hours at temperatures ranging from 1250 to 1450°C. The average drift of this set was –85°C.
13. “Fat End” TCs and TCs insulated with $MgAl_2O_4$ did not demonstrate any improvement over the standard Type N design in terms of longevity during irradiation (see Appendix I).

3.2 Other Points of Note

1. The Type B loose assembly reference TCs used to monitor temperature in the test zone experienced significant negative drift when held at temperatures >1250°C. The problem was solved by installing a deep thermowell in the furnace which allowed the reference TC to be pulled out of the hot zone and inserted only briefly once per week.
2. The first year of testing (2014) showed evidence of oxidation in the test zone. The was prevented in subsequent testing campaigns by forcing the furnace purge gas to flow through a section with hot Zr metal, which served as an oxygen getter.
3. At temperatures ≥1150°C, Type N TCs drift less than Type K, which was an expected result and was confirmed.
4. Type S TCs with crushed insulation experience rapid negative drift at temperatures ≥1150°C. Because of their similar composition, it is likely that a Type B TC of similar construction would behave roughly the same.
5. A Type S loose assembly TC performed very well at 1200°C (no measurable drift over 2000 hours). This seems to be in conflict with Observation 1. However, most likely there is a breakpoint at around 1200 to 1250°C where the solid-state diffusion effects subside, and both the Type S and Type B loose assembly designs would be virtually drift free.

4. CONCLUSIONS

An extensive series of furnace tests were conducted to develop a set of TCs capable of measuring the extreme temperatures projected for the AGR-5/6/7 experiment, and later to characterize the performance of that set over long-term operation.

The early years of testing confirmed that a unique Type N TC developed by Cambridge University specifically for use in high-temperature neutron irradiation experiments would perform well at temperatures up to 1250°C. This early testing also involved another TC for high-temperature neutron irradiation experiments, the HTIR-TC, which had been developed in-house at INL. A significant issue was identified in the HTIR-TC manufacturing process. This TC type requires heat treatment to stabilize the EMF, and the traditional way of doing this was to heat treat only the length expected to operate above 1000°C. It was determined that the entire heated length must be fully heat treated, otherwise the Seebeck coefficient will not be uniform along the length of the cable, and the TC will produce inconsistent results.

The heat treatment issue was solved by the end of 2016, but a problem with HTIR-TC sheath embrittlement persisted. This required a lowering of the HTIR-TC heat treatment temperature from 1650°C to 1450°C resulting in a limit on the stable operating temperature of about 1250°C. The HTIR-TC sheath embrittlement problem was solved in 2019 by encasing the sheath in Nb foil prior to heat treatment. However, this discovery was too late for incorporation in the AGR-5/6/7 experiment.

After selecting a TC set for AGR-5/6/7, testing continued to characterize the performance of these TCs over very long time periods. The result of this testing was that both Cambridge Type N TCs and HTIR-TCs are expected to drift –2 to –4°C per 1000 hours at 1250°C. At lower temperatures these TCs would be expected to drift less and more at higher temperatures.

A final set of HTIR-TCs, configured the same as those that were installed in the AGR-5/6/7 experiment, were furnace tested at temperatures well above the previous furnace tests. This was done to better understand the performance of HTIR-TCs in AGR-5/6/7 that experienced temperatures in the 1300 to 1500°C range. The temperature profile for this test was stepwise starting at 1250°C and ending at 1450°C. The result of this final furnace test program was that all three HTIR-TC examples drifted down, and their drift profiles were remarkably similar. By the end of this final furnace test (covering a total of 4,100 hours), the three TCs exhibited an average negative drift of roughly –85°C, adding weight to the conclusion that the HTIR-TCs in AGR-5/6/7 likely did not experience positive drift.

5. REFERENCES

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APPENDIX A

2014 Testing Summary

Detailed test logs were not kept in 2014, but a summary is provided below. Additional details of this testing may be found in (Palmer 2015).

- A large fraction of the TCs tested failed open circuit before the end, or at the end of the 1250°C heating phase. This may have been partially due to a tiny oxygen leak at the inlet of the furnace. As a minimum, more oxygen was getting to the sensors compared to later testing.
- With the exception of the loose assembly design, all temperature drifts in Type N thermocouples (TCs) were ultimately negative (i.e., eventually mineral-insulated cable (MIC) Type N TCs put out less EMF for a given temperature than they did at startup). A few TCs might have experienced a few degrees of positive drift initially (over the first 2 or 3 days), but eventually these slight positive drifts were overwhelmed by much larger negative drift.
- The comparison or "control" TCs (i.e., standard) Type N TCs experienced a drift of –48°C by the end of the 1200°C heating phase.
- Size increase provided no drift reduction through the 1150°C heating phase, but about a 35% improvement (over the control Type N TCs) at the end of the 1200°C heating phase.
- Haynes 214 alloy sheath provided a 30% reduction in drift at the end of the 1200°C heating phase.
- Spinel insulation provided minimal (15%) reduction in drift at the end of the 1200°C heating phase.
- Spinel insulation combined with size increase (2.34 mm) substantially reduced drift (38%) at the end of the 1200°C heating phase.
- Cambridge high-nickel alloy sheath provided markedly reduced drift (68%) at the end of the 1200°C heating phase. In other words, this design produced approximately one third the drift of the standard design.
- The Type N loose assemblies initially read about 10°C low and then drifted up about 15°C over the first 600 hours of testing. After that they remained essentially flat (no downward drift). After the initial temperature rise, they tracked the Type S loose assembly reference TC nearly exactly. Ultimately, four examples of this design were incorporated into the 2014 furnace testing. They all failed before the end of the 2014 testing campaign.
- Results from the two HTIR-TCs were mixed. One failed open after only 200 hours. The surviving HTIR drifted up about 15°C, followed by a 25°C downward drift at the end of the 1250°C heating phase, for a very respectable –10°C net drift over 4000 hours. However, both HTIR-TCs read about 40°C low upon startup (the source of this calibration error was discovered in 2016).

Additional information gained from the 2014 testing campaign, that was not specifically relevant to the AGR-5/6/7 TC suite, and not included in (Palmer 2015) were the results from Type S and Type K TCs. These results are illustrated in Figure A-1 and A-2 below.

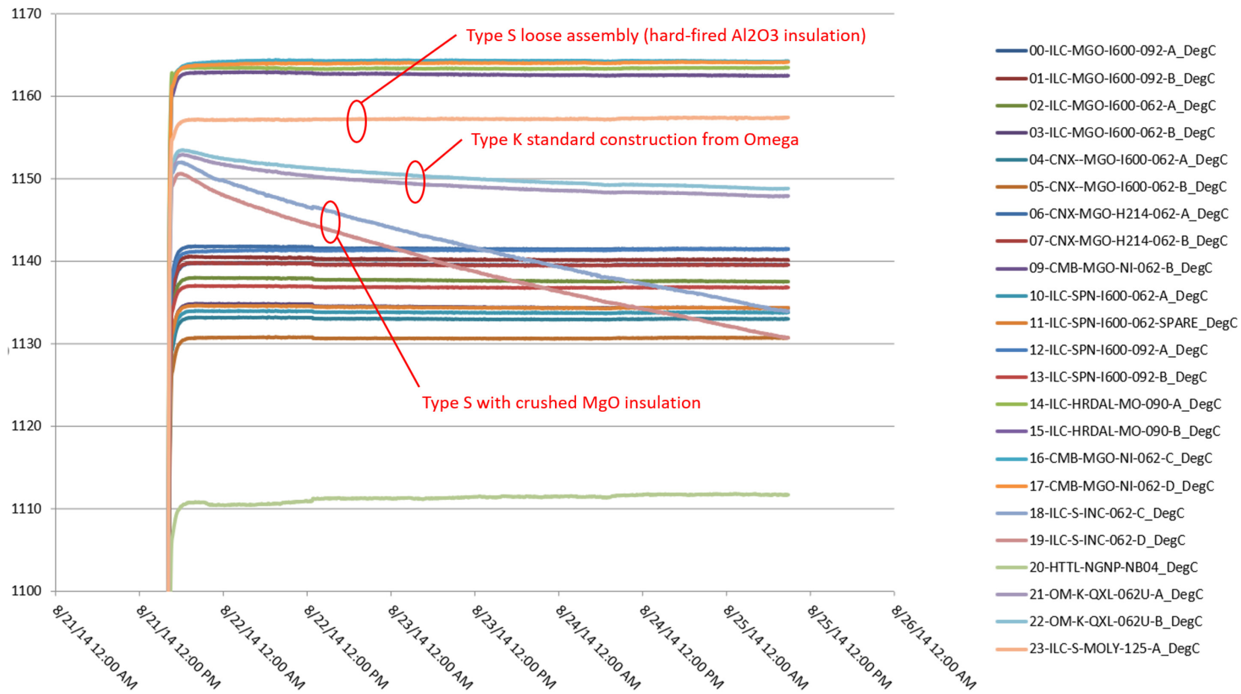

Figure A-1. Initial startup of the furnace at 1150°C. Drift rate of Type S and Type K identified.

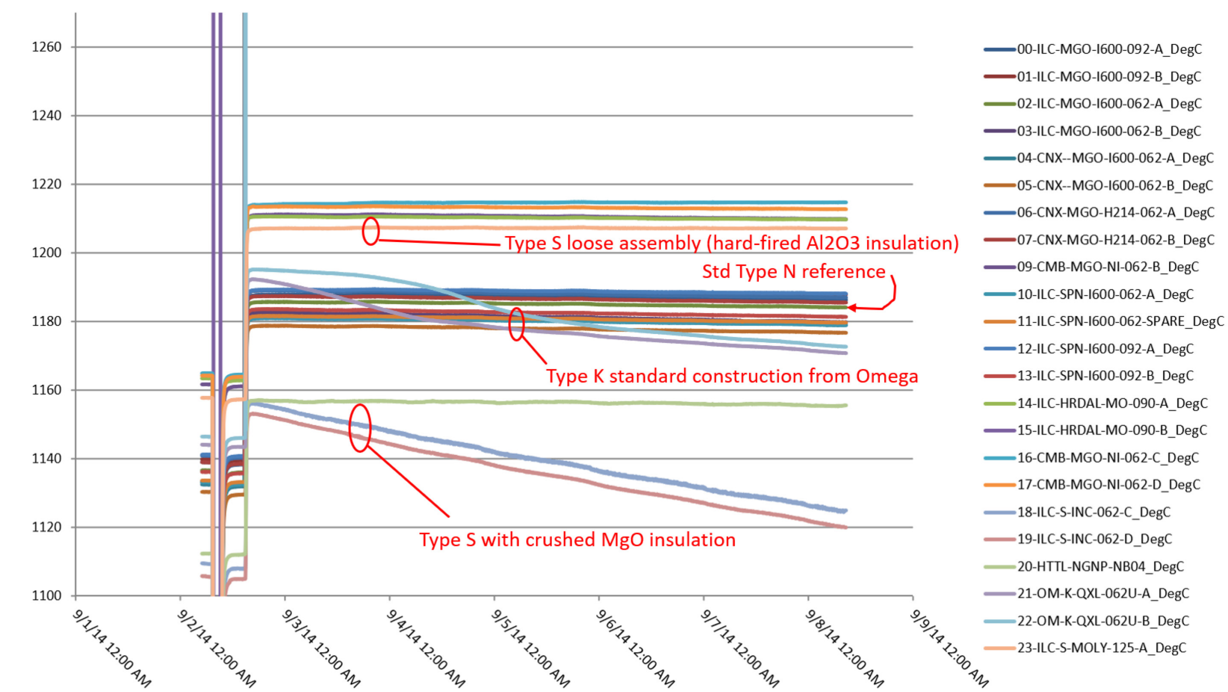

Figure A-2. Initial startup of the furnace at 1200°C. Drift rate of Type S and Type K thermocouples identified.

It was surprising to see the poor performance of crushed insulation Type S platinum/rhodium TCs. Type S TCs are used across industry because of high accuracy and stability. Apparently, this reputation is based primarily on the exposed junction design, or perhaps the loose assembly design (discussed in Appendix I). In the exposed junction configuration, the thermoelements slowly sublimate thus maintaining their purity. The design shown in Figure A-1 and A-2 featured Type S thermoelements, crushed MgO insulation, and I600 sheaths (.062 OD), a typical MIC construction. The drift rate of this design is clearly dramatically greater than any of the other TC systems tested in Phase 2 of 2014. By then end of the 1200°C test period (2000 hours), this MIC Type S design had drifted down approximately 190ºC[b]. (These were removed 1 month prior to the end of the 1200°C phase because they showed signs of shorting to sheath)

At the end of the 1200°C test period the two Omega Type K TCs had drifted down 125°C. Comparatively, the similar construction Type N drifted down less than half this amount (48°C) over the same test period.

Conversely, the performance of the Type S loose assembly TC was excellent. It read 1207°C for a nominal 1200°C furnace temperature at the beginning of the 1200°C test period, and 1206°C at the end of the 1200°C test period (about 2000 hours).

[b] Idaho Laboratories Corp confirmed that they had heard reports of dramatic drift in Type S crushed insulation design.

APPENDIX B

2015 Test Logs and Summaries

There were three separate furnace runs with some sensor exchanges between each run. Table B-1 shows the initial complement of HTIR-TC variations.

Table B-1. TC designations for the 2015 (second) AGR-5/6/7 furnace test

Designation	Reduction (to 0.062" OD)	Comments
HTTL-NGNP-TC06 HTTL-NGNP-TC07	drawn	Conventional HTIR-TC (Nb1Zr/KW-Mo thermoelements, HfO_2 insulation, Nb1Zr sheath) as a reference for other TCs.
HTTL-NGNP-TC08 HTTL-NGNP-TC09	swaged	Single departure from reference where reduction is by swaging instead of drawing.
HTTL-NGNP-TC10 HTTL-NGNP-TC11	drawn	Single departure from reference where insulation is Y_2O_3 instead of HfO_2.
HTTL-NGNP-TC12 HTTL-NGNP-TC13	swaged	Multiple departure from reference where reduction is by swaging instead of drawing and insulation is Y_2O_3 instead of HfO_2.
HTTL-NGNP-TC14 HTTL-NGNP-TC15	swaged	Multiple departure from reference where reduction is by swaging instead of drawing and La-Mo is used in place of KW-Mo.
HTTL-NGNP-TC16 HTTL-NGNP-TC17	swaged	Multiple departure from reference where reduction is by swaging instead of drawing and Ti48Nb is used in place of Nb1Zr.
HTTL-NGNP-TC18 HTTL-NGNP-TC19	swaged	Multiple departure from reference where reduction is by swaging instead of drawing, insulation is Y_2O_3 instead of HfO_2, La-Mo is used in place of KW-Mo, and Ti48Nb is used in place of Nb1Zr.
HTTL-NGNP-TC20 HTTL-NGNP-TC21	drawn[a]	Multiple departure from reference where drawing reduction ends at 0.067" OD, insulation is Y_2O_3 instead of HfO_2, La-Mo is used in place of KW-Mo, and Ti48Nb is used in place of Nb1Zr.
HTTL-NGNP-TC22 HTTL-NGNP-TC23	swaged	Multiple departure from reference where reduction is by swaging instead of drawing, insulation is Y_2O_3 instead of HfO_2, La-Mo is used in place of KW-Mo, and P-Nb is used in place of Nb1Zr.

a. To 0.067" OD.

A short initial furnace run was conducted for 6 days (April 28, 2015, to May 4, 2015) at a hot block temperature of about 1275°C. This test included 12 HTIR-TCs, two Cambridge Type N, and four standard Type N loose assembly. There was more scatter in the Type N readings in cold block than was expected. A Type S and two standard Type Ns read 1100°C, so that is probably the most likely cold block temperature. The two Cambridge Type N TCs read significantly higher at 1115°C and 1130°C. All the Type N readings (except one loose assembly, which was drifting up) were nearly flat—no drift. There was huge scatter in the HTIR-TC readings in the hot block (almost 200°C). The furnace was shut down after 6 days because of all the scatter in the HTIR-TC readings.

The scatter in the HTIR-TCs was not random. The Yttria insulated TCs read high and the HfO insulated TCs read low. This could have been because the Yttria TCs had very low insulation resistance (5 to 20 ohms), which would have been even lower in the original heat treatment and calibration furnace located at the High Temperature Test Laboratory (HTTL), which was longer than the Idaho Research Center (IRC) furnace. That is, the longer furnace at HTTL (where the HTIR-TCs were calibrated) meant that more voltage was being lost through stray currents to ground, which resulted in higher readings in the shorter IRC furnace. The HTIR-TCs insulated with HfO were reading below the furnace temperature because the gradient in IRC furnace was closer to the junction with most wire fully heat treated while the gradient at HTTL during calibration included some wire that was only partially heat treated.

The scatter in the Yttria insulated HITR-TCs may have been due to low insulation resistance, however the fundamental issue with poor calibration performance in the HTIR-TCs was eventually traced to the heat treatment process. In order to produce a uniform Seebeck coefficient throughout the heated length of the TC, it is necessary to heat treat this entire length to the full heat treat temperature.

The furnace was restarted on June 8, 2015 and was set at 1275°C. Heat treated and calibrated TC06 and TC09 again to try to achieve more accurate temperatures. The heat treatment included a deeper insertion of TC06 and TC09 into the furnace, approximately 8 inches deeper. Upon restart at 1275°C in the hot block, TC06 and TC09 read 1257°C and 1267°C, respectively, which was a great improvement. Insulation resistance of TC06 and TC09 was 349 ohms and 377, respectively. Type Ns in the cold block read about the same as before except the two Cambridge TCs were now reading about 1108°C, closer in temperature to each other and to the other Type Ns in the cold block.

Increased temperature of hot block to 1380°C–1400°C on June 16, 2015, (not sure of exact temperature because all reference TCs in the hot block had drifted badly). TC06 = 1337°C, TC09 = 1382°C. Both TC06 and TC09 drifted up roughly 10°C over the 14 day period until shutdown. Insulation resistances of TC06 and TC09 during this 2-week period were 120 ohms and 180 ohms, respectively.

During the 2-week higher temperature run (with the hot block at 1380–1400°C) the cold block was running at roughly 1210°C and the Cambridge TCs were indicating about 1218°C with no discernable drift. Interestingly, the insulation resistances for the Cambridge TCs were very good: 35,000 ohm and 200,000 ohm. The loose assembly Type N TCs also showed very little drift and high insulation resistance.

Shut down furnace on June 29, 2015

Restarted with two Pt/Pd loose assembly TCs on July 1, 2015, and with a hot block goal setpoint of about 1390°C. Ran until October 6, 2015, and this ended this phase of the testing. During this time, it appeared that the furnace temperature was drifting up (more particularly in the cold block) and a 10°C decrease of the setpoint was executed on August 6, 2015. The period encompassed about 95 days of nearly continuous operation. The Pt/Pd TC in the hot block failed almost immediately, but the one in the cold block operated through the end of the year.

Drift on TC06 and TC09 was low—about 10–15°C. It was difficult to tell because the furnace was shut down and restarted at least once, and it appeared the labels for TC06 and TC09 were perhaps swapped at a restart around September 22, 2015. Also, there was the 10°C setpoint decrease discussed above. Insulation resistance on TC06 degraded to about 75 ohm, but TC09 stayed around 200 ohm.

In the cold block during this period, the Cambridge TCs suffered very little drift, if any. It is difficult to be precise because of the unplanned shutdowns, set point change, and possible drift in temperature of the furnace. The Pt/Pd TC tracked the Cambridge TCs very closely, but it read about 40°C low. This temperature difference was partly due to no icepoint for the Pt/Pd and partly because the Cambridge TCs seemed to be running 10–15°C high (calibration data from Idaho Laboratories Corporation (ILC) (the manufacturer) indicated this batch of Type N wire was running about 10–15°C high). The Cambridge TC insulation resistance varied during this period but was always above 10,000 ohms on both TCs.

A check was performed on October 7, 2015, to verify graphite block runs iso-thermal and the agreement between four Type N TCs, which were spread throughout the hot block, indicated that it did. The hot block temperature was 1185°C. The four standard Type N TCs agreed within about 1.5°C of each other (although they began drifting down immediately as seen in Figure B-1).

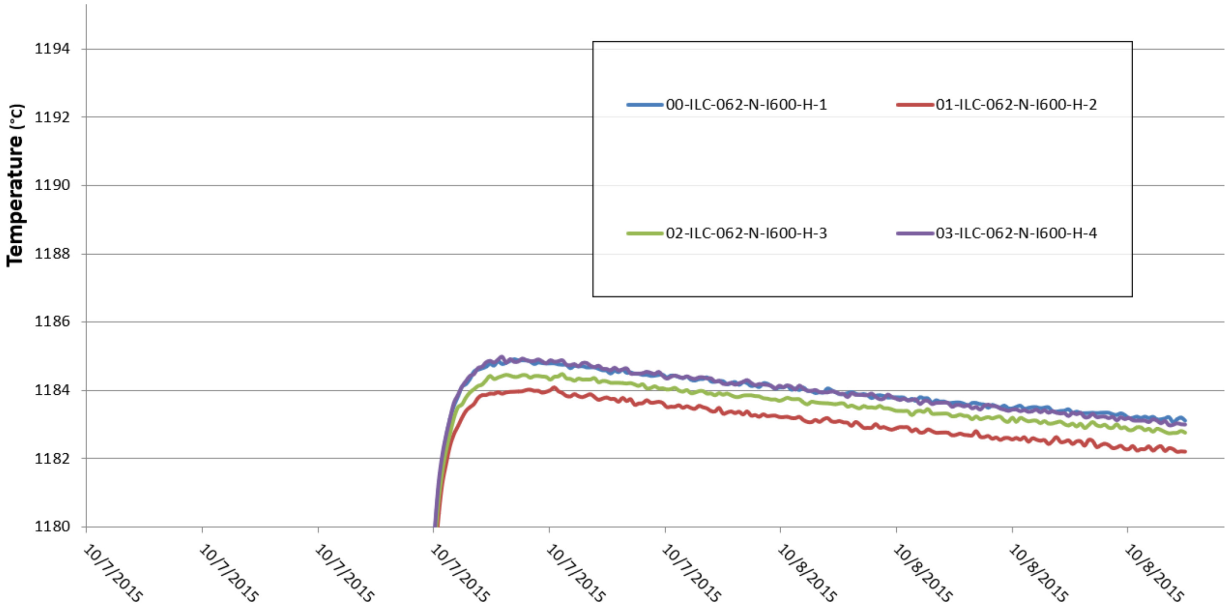

Figure B-1. Four Type N thermocouples spread throughout hot block

Between October 7, 2015, and December 7, 2015, the furnace was kept running. TC06 and TC09 failed at the beginning of this period, so the only real useful information was from the Cambridge Type N in the cold block and the Pt/Pd in the cold block. At the beginning of this period the cold block temperature was about 1040°C, as read on Cambridge TC "E." During this time the furnace set point was increased twice in 40°C increments. During this 2-month period the Cambridge TCs and the Pt/Pd TC drifted upward a few degrees (which could have been a result of the furnace control TC drifting down), and by almost exactly the same amount, indicating little if any drift in the Cambridge TCs.

APPENDIX C

2016 Test Log and Summary

5/3/2016 – 6/30/2016.

Started furnace on May 3, 2016, at 1275°C with a completely new batch of thermocouples (TCs) (listed below). This run continued until June 30, 2016, (with a couple outages and installation of TC42 on May 26, 2016). Temperature was increased on May 9, 2016, to 1385°C in hot block. Although the problem with heat treating only part of the HTIR-TC length had been identified on March 30, 2016, this batch of high-temperature irradiation-resistant thermocouples HITR-TCs still had the basic no-move heat treatment. This resulted in all HTIR-TCs having a substantial negative calibration bias.

Table C-2. Summary log of first furnace run in 2016

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC024 Operated for 5 days, failed	Old HTIR-TC design. HfO insulation Nb/1% sheath	Read 160°C low at 1275. Failed (shorted to sheath) when temp was increased to 1385°C on May 9, 2016	Removed after the furnace was shut down on 6/30/2016
HTIR-TC025 Operated for 119 days w/o failure	Same as TC024	Read 110°C low at 1275°C. Read 145°C low when temp was increased to 1385°C. Drifted down about 30°C throughout the balance of 2016. Insulation – 1000 ohms @1385°C setpoint	Removed before the furnace was restarted in 2017
HTIR-TC029 Operated for 119 days w/o failure	HfO insulation and Nb/1% sheath, but different thermoelements versus traditional design	Read 140°C low at 1275°C and 185°C low at 1385°C. Drifted very little (10°C) throughout the balance of 2016. Insulation – 1000 ohms	Removed before the furnace was restarted in 2017
HTIR-TC40 Operated for 240 days w/o failure	Al_2O_3 insulation pure Nb sheath. Also, different thermoelements from original HTIR	Read 60°C low at 1275°C and 90°C low at 1385°C. Basically no drift throughout 2016. Insulation – 50,000 ohms. Showing perhaps the superiority of Al_2O_3 insulation	Showed some upward drift during 2017 (up 50°C and down 30°C). Removed before the 5/23/2017 restart
HTIR-TC41 Operated for 540 days, failed	Same as TC40	Read essentially identical to TC40 throughout 2016, both temperature and insulation resistance	Read extremely stable from 1/17/2017 through 4/24/2017 about 95°C below the 1315 setpoint. As of 11/27/2017 still operating and reading 47°C below 1250°C setpoint. It had an initial calibration problem, but this has been a very stable TC. As of 8/27/2018 – now this TC is reading nearly dead-on at 1247°C. Perhaps just happenstance. 9/24/18 = 1254°C. Failed during cooldown 1/30/19.

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC42 Failed after 375 days.	Old HTIR design (HfO insulation)	Installed 5/26/2016 and began a gradual upward climb of about 100°C. The insulation resistance was only 200 ohms	Eventually, after many months of operation, this TC read reasonably close to the set point during 2017. Read 1338°C in Feb 2017 with setpoint at 1315°C. Insulation – 575 ohms. As of 9/18/2017 reading 1360°C versus 1250°C setpoint, failed during 10/9/17 power outage. Removed after Nov 27 shutdown. Given the large swings in reading, this has not been a successful TC.
090-B	.090 moly sheathed loose assembly. Hard-fired alumina insulation. Reference TC.	Embedded in hot block centerline. Started off dead-on at 1275°C. But started drifting immediately and the drift accelerated when the temperature was increased to 1385°C. Total drift by 6/30/2016 was 35°C	Has continued to drift down through 2016 and 2017, although not as rapidly with furnace at lower temperatures. As of 9/18/2017 reading 1188°C versus 1250°C set point. As of 11/27/2017 reading 1193°C so has not drifted last 2 months.
125 B –Twell	.125 moly sheathed loose assembly. Hard-fired alumina insulation. Reference TC.	Installed in a thermowell which terminates at the centerline of the hot block. Dead-on at 1275°C startup and no discernable drift through 6/30/2016.	As of 9/18/2017 reading 1247°C versus 1250°C setpoint. Ordered a new TC of this type, and installed it 10/30/2017. It seemed to agree with the old one within about 1°C. Read 1147.5°C upon shutdown on 11/27/2017. Swapped TCs Aug 2018. Replacement TC read about 1243°C could be because the old TC's tip was still in the thermowell.
N-I600-H6	Std Type N (.062) in hot block	Appeared to be pretty accurate for first few readings at 1275°C, but rapidly drifted down (55°C) in six days	When temperature was increased to 1385°C on 5/9/2016 this TC melted and failed.
N-I600-H7	Same as H6	Same as H6	Same as H6
N-I600-H8	Std Type N (.062) in cold block	Initial reading was 1100°C with hot block at 1275°C. Drifted down 1.5°C in six days	After temperature increase on 5/9 read 1190°C and drifted down about 50°C over the next 51 days (6/30/2016). These two TCs (H9 also) are still in the cold block as of 9/18/2017, but they are not providing any useful information. Removed Dec 2017.
N-I600-H8	Same as H8	Same as H8	Same as H8

Summary of this test period is that the HTIR-TCs were way off in calibration upon startup. The alumina insulated worked better than Hafnia insulated (better drift performance). Two alumina insulated TCs showed very stable operation. It was noted that Type B loose assembly TCs do drift particularly at temperatures above 1300°C. Our scheme of inserting a Type B into a molybdenum thermowell and only periodically fully inserting it into the hot block (once per week for 1 hour) seemed to prevent drift. However, subsequent experience confirmed that for the most accurate measurements, the Twell TC needs to be replaced every six months or so.

8/17/2016 to 9/6/2016

Table C-3. Summary log of second furnace run in 2016. Restarted 8/17/2016 @ 1385°C. HTIR-TC51 was installed in place of HTIR-TC024.

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC51 Failed after 20 days.	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements. Not sure about heat treatment.	Read about 110°C low upon initial startup with hot block at 1385°C. Drifted down another 10°C over the test period. Insulation was about 25,00 ohms, so that wasn't the problem.	Failed open during cool down on 9/6/2016. We didn't get much out of this TC.

Summary of this test period. The one new HTIR-TC we installed read more than 100°C low just like the others. Nothing substantial was learned from this test period.

11/21/2016 to 1/3/2017

Table C-4. Summary log of third furnace run in 2016. Restarted 11/21/2016 @1385°C. Installed three new HTIR-TCs. TC53, TC54, TC55. These had all been heat treated at 1600°C or 1650°C.

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC53 Failed after 286 days	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements.	Read about 15°C high upon initial startup with hot block at 1385°C. Insulation was about 5,000 ohms, and increased to 20,000 ohms by the end of the year.	Drifted up just slightly to 1403°C at the end of 2016. So very stable operation for about six weeks and reading about 15°C high. When temp was lowered to 1315°C in 2017 read 1327°C, with very slight downward drift to 1325°C May 2017. Last half of 2017 setpoint has been 1250°C and this TC varied from 1255°C to 1262°C (9/18/2017). So, this is more than 6000 hrs with less than 10°C total drift. Failed during 10/9/2017 power outage.
HTIR-TC54 Failed after 20 days	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements.	Read about 60°C high upon initial startup with hot block at 1385°C. Insulation was about 2,000 ohms, and increased to 6,000 ohms by the end of the year.	Drifted up 15°C so that it read 75°C high by the end of 2016. Failed open at end of 2016.
HTIR-TC55 Operated w/o failure for 141 days	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements.	Read about 85°C high upon initial startup with hot block at 1385°C. Insulation was about 5,000 ohms, and increased to 12,000 ohms by the end of the year.	Drifted up to 5°C so that it read 90°C high by the end of 2016. So pretty stable. During the 1315°C part of 2017 drifted up 30°C. Removed May 2017.

Summary of this test period. HTIR-TC53 has performed very well since installation. TC54, and TC55 were thought to be the same construction, heat treatment, and calibration. But they had poor calibration performance, and middling drift performance. The performance of this set of three TCs added confusion to the overall picture, calibration was off, slight upward drift, but not enough to be of major concern, which was different from other HTIR-TCs with pure Nb sheaths. All the others drifted down.

APPENDIX D

2017 Test Log and Summary

1/17/2017 – 5/16/2017.

Started January 17, 2017, at 1315°C. Installed two new high-temperature irradiation-resistant thermocouples (HTIR-TCs): TC2 and TCSAMPLE1. These were essentially the same construction, but TC2 was heat treated at 1650°C and TCSAMPLE1 was heat treated at 1450°C. This was done because pure Nb TCs heat treated at 1650°C were very brittle, while those heat treated at 1450°C retained reasonable ductility. We wanted to compare the performance of these two at a somewhat lower temperature than the 1385°C temperature we had been running. Only one TC in AGR-5/6/7 will run above 1350°C. It seemed likely 1385°C would cause significant drift in the TC heat treated at 1450°C (TCSAMPLE1); so 1315°C was picked as a compromise.

Table D-1. Summary log of first furnace run in 2017

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC2 308 days of operation w/o failure	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements. 1650°C heat treat.	Read dead-on (1316°C) upon initial startup with hot block at 1315°C. Drifted gradually up about 5°C and then back down to 1315°C over the test period. So outstanding performance. Insulation resistance was about 1500 ohms, which is kind of low for 1315°C.	In May 2017 when the set point was lowered to 1250°C this TC read 1247°C. As of (11/27/2017) read 1241°C so it has drifted down about 6°C over four months. Overall, this TC has performed very well. We can't realistically expect better performance than this considering there is at least 5°C uncertainty in the furnace temperature and stability. Removed after 11/27 shutdown for examination.
HTIR-TCSAMPL1 308 days of operation, failed	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements. 1450°C heat treat.	Read about 15°C high upon initial startup with hot block at 1315°C. Drifted down 30°C over the test period. Insulation resistance was about 7000 ohms.	In May 2017 when the set point was lowered to 1250°C this TC read 1231°C. Just before shutdown on 11/27/2017 it read 1237°C so it has drifted slightly up over the last 6 months at the lower setpoint temperature. Failed (probably from handling) prior to 1/11/2018 startup)

Summary of this test period. As feared, the TC with the lower heat treat temperature drifted significantly at 1315°C. On the bright side this TC (TCSAMPLE1) was stable once the temperature was lowered to 1250°C. TC2 performed great at 1315°C (and 1250°C); calibration was dead-on and the drift was very low.

5/23/2017 – 11/27/2017.

Started 5/23/2017 @1250°C. Installed two new HTIR-TCs. TC22 and TC1ZR. TC22 was a spare TC left over from the same lot of HTIR-TCs that went into the AGR-5/6/7 test. TC1ZR was a design suggested by Nathan Jerred, which incorporated Al_2O_3 insulation and Nb/1% Zr sheath. Previously, it was thought that these insulation and sheath materials were incompatible with each other. However, Nathan pointed out that Al_2O_3 may be detrimental to Nb/1% Zr thermoelements, but probably would not significantly attack a Nb/1% sheath because of the much greater volume of material in a sheath versus thermoelement. TC1ZR was heat treated at 1650°C and still retained at least a little ductility (a small bend was tested on this TC before it was installed in the furnace). The test temperature was lowered from 1315°C to 1250°C. This was done in the hopes that the HTIR-TCs used in the test, which were heat treated at 1450°C, would remain stable when operated at 1250°C or less. (Note that the 1250°C temperature discussed here is the hot block temperature. During this phase of testing nothing of real interest occurred in the cold block and there was no undrifted TC in the cold block that could be used as a reference temperature.)

Two new Cambridge TCs from the same lots as were used in AGR-5/6/7 were also installed in this test. CAMB-TC11 was .093 in. diameter (last 8 in. - the rest of the TC was .062 in.) and CAMB-TC13 was .062 in. diameter. Two standard Type N (Inconel 600 sheathed) from the same lots as were used in AGR-5/6/7 were also installed. I600-TC4 used Spinel insulation and was .093 in. diameter (last 8 in. - the rest of the TC was .062 in.) and QA25562-2 was .062 in. diameter (standard MgO insulation).

Table D-2. Summary log of second furnace run in 2017

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC22 340 days of operation, failed	Al_2O_3 insulation, pure Nb sheath. La-Mo/P-Nb thermoelements. 1450°C heat treat.	Read about 20°C high upon initial startup with hot block at 1250°C. Drifted down about 5°C over next four months. Insulation resistance was about 2000 ohms, which is quite low for 1250°C (TC53 had 100,000 ohms at 1250°C).	Just before shutdown on 11/27/2017 it read 1262°C so it has drifted down about 8°C over 6 months (getting closer to the setpoint in the process). This provides reasonable assurance that the HTIR-TCs in AGR-5/6/7 will provide meaningful temperature data at temperatures of 1250°C and below for long periods of time. Operated until 5/22/2018 at which point it failed open when the furnace was shut down . Was reading 1257°C prior to shut down . So, this HTIR-TC performed excellent throughout its life and the only disappointment was its failure.
HTIR-TCZR1 187 days of operation w/o failure	Al_2O_3 insulation, Nb/1% Zr sheath. La-Mo/P-Nb thermoelements. 1450°C heat treat.	Read nearly dead-on upon initial startup (1248°C) with hot block at 1250°C. Drifted up about 20°C to 1269°C over the test period ending 9/18/2017. Insulation resistance was about 60,000 ohms, which is good.	Unfortunately, between 9/18/2017 and 11/27/2017 the upward drift accelerated, and it read 1297°C at the end of the period (50°C upward drift over six months). Further investigation (Jan 2018) revealed that the calibration curve for this TC was unusually low (i.e., not much EMF for a given temperature). Was removed Dec 2017 for analysis.

TC ID	Construction Details	Performance Summary	Ultimate Fate
CAMB-TC11 760 days of operation w/o failure	Special high purity Ni sheath, MgO insulation. The 8 in. of the sheath nearest the junction is .093 in. diameter. The rest of the cable is drawn down to .062 in. Mo protective sleeve.	Read just 4°C high upon initial startup (1254°C). Drifted down 6°C over the 4-month test period (to 9/18/2017). Insulation resistance 2000 ohms which gradually decreased to 500 ohms. This is much lower than the preproduction sample Cambridge TCs provided in 2014.	Just before shutdown on 11/27/2017 it read 1248°C So basically no additional drift over last two months. As of 8/27/2018 still working and reading 1250°C. So this is outstanding performance. Developed a grounded junction ~4/23/18, but still seems to provide valid readings. 9/24/2018=1247.5°C 1/30/2019=1237°C 12/2/2019=1215°C 18,200 hrs with –39°C drift for –2°C/1000 hrs drift.
CAMB-TC13 340 days of operation, failed	Special high purity Ni sheath, MgO insulation. .062 in. constant diameter. Mo protective sleeve.	Read 10°C high upon initial startup (1260°C). Drifted down 7°C over the 4-month test period. Insulation resistance 1400 ohms which gradually decreased to 800 ohms. This is much lower than the preproduction sample Cambridge TCs provided in 2014.	Just before shutdown on 11/27/2017 it read 1252°C So basically no additional drift over last two months. As of 8/27/2018 still working and reading 1249°C. So this is outstanding performance. Insulation resistance still high >1500 ohms. 9/24/2018=1250°C. Failed 1/30/2019 during cooldown. Read 1243°C. –17°C drift over 340 days. 8,160 hrs. So –2°C/1000 hrs drift.
I600-TC4 187 days of operation w/o failure	I600 sheath, Spinel insulation. The 8 in. of the sheath nearest the junction is .093 in. diameter. The rest of the cable is drawn down to .062 in.	Read 3°C low upon initial startup (1247°C). Drifted down 90°C over the 4-month test period. (This was expected.) Insulation resistance 100 ohms. This is a very low insulation resistance, but these TCs will operate at much lower temperatures in the AGR-5/6/7 test (<1000°C).	Still functioning as of 11/27/2017. Total drift now –105°C. Removed after furnace shutdown on 11/27/2017.
QA255562-2 277 days of operation, failed	I600 sheath, MgO insulation, .062 in. diameter.	Read 7°C high upon initial startup (1257°C). Drifted down 120°C over the 4-month test period. (This was expected.) Insulation resistance 300 ohms.	Failed 4/10/2018. This TC and I600-TC4 both read with 0.6% of setpoint at startup. This is within standard limits for Type N, and was a good cal check on these lots of TCs, which were used in AGR-5/6/7.

The furnace was shut down 11/27/2017 and TCs 1% Zr and TC2 were removed from the assembly for examination. The furnace was not restarted until after the holiday break.

Summary of this test period. The performance of the two new HTIR-TCs was encouraging. They both read within 1.5% of the setpoint for the entire four months. However, the increasing upward slope of the drift of TC1ZR is concerning. Perhaps there is some interaction between the Nb/1% Zr sheath and the thermoelements of this TC. The performance of the two HTIR-TCs installed at the beginning of 2017 was very stable during this 4-month time frame at 1250°C. Overall, we can say that a lot of progress was made in the design, heat treatment, and calibration of the HTIR-TCs during 2017, especially heat treatment and calibration. During the testing performed in 2016 only one of the HTIR-TCs was within 60°C of the setpoint upon initial startup, while in 2017 none of them read more than 20°C off at startup and half of them were with 5°C of the setpoint at startup.

The Cambridge TCs performed very well during this six-month time frame.

The excellent agreement of the two standard Type N with the hot block set point at startup provided a nice calibration check on the lots of standard Type N TCs used in AGR-5/6/7. This TCs drifted rapidly, which was expected. Standard Type N TCs such as these were only installed in AGR-5/6/7 locations with temperatures below 1000°C.

APPENDIX E

2018 Test Log and Summary

1/11/2018–5/21/2018.

Table E-1. New thermocouples installed for first furnace run in 2018.

TC ID	Type	Hot Block or Cold Block
N-CAMB-TC6	Camb N	hot
N-CAMB-TC8	Camb N	hot
N-CAMB-TC9	Camb N	cold
N-CAMB-TC10	Camb N	cold
HTIR-TC-ZR2	HTIR	hot
HTIR-TC-ZR3	HTIR	hot
HTIR-TC-ZR4	HTIR	hot
N-I600-TC6	Std N	cold

CAMB-TC6, TC8 are from the same lot as those installed in AGR-5/6/7. These TCs were placed in the hot block in zirconia sleeves the same as the hottest Type N TCs in the AGR-5/6/7 test, which are in Capsule 3 in zirconia sleeves. The previous batch of Cambridge TCs were put in the furnace in Mo sleeves. CAMB-TC9, TC10 were placed in the cold block in Nb sleeves the same as Camb TCs in Capsule 1 of AGR-5/6/7. The first testing of Cambridge thermocouples (TCs) (in 2014) was done in Nb sleeves but at higher temperatures (1150, 1200, 1250°C) and they experienced drift even at 1150°C. Because the Cambridge TCs in Capsule 1 are expected to run <1050°C, there is a reasonable chance that they will not drift at all at these temperatures, even though they are in Nb sleeves. With the hot block at 1250°C the cold block runs at about 1080°C. If the drift is low at this temperature, we may assume that the Cambridge TCs in Capsule 1 will perform just as well. N-I600-TC6, a standard Type N TC from the same lot as those put in AGR-5/6/7, was also placed in the cold block to compare its performance against the Cambridge TCs at this lower temperature (note that all std Type Ns in AGR-5/6/7 run at temperatures well below 1000°C).

HTIR-TC-ZR2, ZR3, and ZR4. These HTIR-TCs were constructed after the AGR-5/6/7 test was built using Nb/1% Zr sheath material and alumina insulation (for ZR2 and ZR3) and HfO insulation for (ZR4). We wanted to see if the rapid upward drift of HTIR-TC-ZR1 was due to the 1% Zr, or just an anomaly with that particular unit.

Table E-2. Thermocouples retained from the previous run.

TC ID	Type	Hot Block or Cold Block
ILC-062-N-CAMB-TC11	N	Hot
ILC-062-N-CAMB-TC13	N	Hot
NGNP-HTIR-TC41	HTIR	Hot
HTIR-TC22	HTIR	Hot

Table E-3. Summary log for first furnace run of 2018

TC ID	Construction Details	Performance Summary	Ultimate Fate
N-CAMB-TC-6 298 days of operation, failed	Special high purity Ni sheath, MgO insulation. .062 in. constant diameter.	Read about 3°C high upon initial startup with hot block at 1250°C. Drifted up about 6°C over next four months. Insulation resistance was about 1500 ohms and gradually decreased to about 500 ohms.	As of 8/27/2018 still reading a little high (about 12°C high) compared to the nominal furnace temperature of 1250°C, but showing almost no drift, indicating that the ZrO_2 sleeves are not affecting the thermoelements through solid-state diffusion. 9/24/2018=1260°C 1/29/2019=1256°C Failed during cooldown on 1-30-19 +3°C drift over 7,150 hrs. ~+0.5°C/1000 hrs.
N-CAMB-TC-8 573 days of operation, no failure	Replicate of CAMB-TC-6.	Read about 5°C high upon initial startup with hot block at 1250°C. Drifted up about 5°C over next week and then drifted down about 12°C over the next four months. Insulation resistance was initially about 1000 ohms and gradually decreased to about 500 ohms.	As of 8/27/2018 reading only 3°C low (1247°C) compared to a nominal furnace temperature of 1250°C. Again, a good demonstration that ZrO_2 is a good sleeving material. 9/24/2018=1245°C 12/2/2019=1213°C (–42°C drift in 13,800 hrs, which is –3°C/1000 hrs) Removed from furnace 12/5/2019 w/o failure.
N-CAMB-TC-9 573 days of operation, no failure	Replicate of CAMB-TC-6.	Read 1076°C upon initial startup. Because there is no reference junction in the cold block, this temperature is taken as the starting temperature. After four months showed a 5°C temperature rise, but this appears to be due to an actual change in the cold block temperature when the control furnace controller and TC were swapped. Insulation resistance 50,000 ohms which gradually increased to 150000 ohms.	As of 8/27/2018 reading 1084°C, which is 8°C higher than the start temperature but this seems to be due to actual change in the cold block temperature. Essentially no drift has occurred in this TC. 9/24/2018=1083°C 5/9/2019=1073°C 7/20/2019=1074°C 12/2/2019=1073°C 573 days total (13,750 hrs). These two Cambridge TCs (9 & 10) operating at 1075–1080°C, appear to have not drifted at all.

TC ID	Construction Details	Performance Summary	Ultimate Fate
N-CAMB-TC-10 573 days of operation, no failure	Replicate of CAMB-TC-6.	Read 1081°C upon initial startup, which agreed well with CAMB-TC9 initial temperature. After four months showed a 5°C temperature rise, but this appears to be due to an actual change in the cold block temperature when the control furnace controller and TC were swapped. Insulation resistance 150000 ohms which gradually decreased to 130000 ohms.	As of 8/27/2018 reading 1091°C, which is 10°C higher than the start temperature but most of this difference seems to be due to actual temperature change in the cold block temperature. Essentially no drift has occurred in this TC. 9/24/2018=1089°C 5/9/2019=1084°C 7/20/2019=1075°C 12/2/2019=1085°C 573 days total (13,750 hrs). 1084°C to 1075°C back to 1085°C bobble is probably a result of changes in-furnace temperature and not instability in the TC reading.
N-I600-TC6 0 days of successful operation	I600 sheath, MgO insulation. Cable diameter of .062 in.	From Day 1 read 160°C low compared to the two Cambridge Type N in the cold block. Something is wrong with this TC.	Still read 160°C low when furnace was shut down 5/22/18 so it was removed from the test.
HTIR-TC-ZR2 275 days of operation, failed	Al_2O_3 insulation, Nb/1% Zr sheath. La-Mo/P-Nb thermoelements. 1600°C heat treat for 6 hrs. Mo protective sleeve.	Read 9°C high upon initial startup (1259°C). Drifted up 30°C over four months to 1289°C. Insulation resistance 1500 ohms to start, increased to 3000 ohm over time.	Seems to have stopped drifting up and has read flat since furnace restart on 7/19/2018 (reading 1288°C as of 8/27/2018). Failed on heatup 1/7/2019.
HTIR-TC-ZR3 573 days of operation w/o failure	Same as ZR2.	Read 34°C high upon initial startup (1284°C). Drifted up another 24°C over four months to 1308°C. Insulation resistance 2000 ohms to start, increased to >30000 ohm over time.	Continued to drift up and is reading 1324°C as of 8/27/2018. 5/12/2019 = 1324°C 7/22/2019 = 1337°C Drifting up. This is similar to other Nb/1% Zr HTIR-TCs.
HTIR-TC-ZR4 153 days of operation w/o failure	HfO insulation, Nb/1% Zr sheath. La-Mo/P-Nb thermoelements. 1600°C heat treat for 6 hrs.	Read 21°C high upon initial startup (1271°C). Drifted up to 1287°C and back down to 1273°C at the end of the 4-month period. Insulation resistance 350 ohms to start, increased to 480 ohm over time.	Removed for examination after 5/22/2018 shutdown. Was reading 1273°C at that time.

The furnace was shut down 5/22/2018 and TCs HTIR-TC-ZR4 and HTIR-TC22 were removed from the assembly for examination (TC22 had failed).

Summary of this test period. The performance of the three new HTIR-TCs was not as good as TC22 from the AGR-5/6/7 lot. The two alumina insulated TCs (HTIR-TC-ZR2, and HTIR-TC-ZR3) read 30–50°C high at the end of the period. The slope of the upward drift for these two was not as high as TCZR1, which was tested in 2017. The HfO insulated HTIR-TC-ZR4 performed somewhat better and only read 21°C high at the end of the period (after having read up to 37°C high half-way through the testing period).

All the Cambridge TCs in the hot block, both old and new, continued to perform well with the greatest error being 12°C high, and most of them reading within 5°C of the nominal furnace temperature. The most important conclusion here is that the ZrO protective sleeves do not affect the thermoelement output (similar to Mo protective sleeves).

The Cambridge TCs in the cold block appeared to drift not at all (certainly not downward as is typical for a Type N TC at high temperature). There was about an 8°C temperature rise on both TCs but this appeared to be due to an actual temperature rise in the cold block when the furnace controller was changed.

The one standard Type N TC in the cold block was flawed from the beginning. One or more will be installed the next time the furnace is disassembled.

7/19/2018–1/30/2019.

Table E-4. New thermocouples installed for second furnace run in 2018. Started 7/19/2018 @1250°C hot block temperature

TC ID	Type	Hot Block or Cold Block
N-TD-TC-66	Type N	hot
HTIR-TC-62	HTIR	hot
HTIR-TC-64	HTIR	hot
HTIR-TC-66	HTIR	hot
N-I600-TC-Al_2O_3	Type N	Cold
N-I600-TC-10	Type N	Cold

N-TD-TC-66 is a Type N TC manufactured by Idaho Labs, Inc utilizing a “TD Alloy” sheath produced by Special Metals Corp. This sheath material was formulated to eliminate Mn and other elements known to contaminate Type N thermoelements and produce drift. This alloy still incorporates Cr which gives it good corrosion resistance in PWR water environment, and so it was hoped that this alloy could have all the advantages of the Cambridge Type N and still be able to be used in, for example, ATR Loop 2A experiments.

HTIR-TCs 62, 64, and 66 all used Nb/1% Zr sheath material, but each has a different insulation Al_2O_3, MgO, and HfO_2. The thermoelements and heat treatments were also the same. However, the calibration of these three TCs did not include the boiling water and room temperature points.

N-I600-TC-Al_2O_3 and N-I600-TC-10 are standard Type N TCs manufactured by Idaho Labs. Al_2O_3 has alumina insulation and –10 is from the same lot as the AGR-5/6/7 Type N TCs and has MgO insulation. These TCs were installed to take the place of the std Type N from the previous run, N-I600-TC6, which operated poorly from day 1.

Table E-5. Thermocouples retained from the previous run.

TC ID	Type	Hot Block or Cold Block
N-CAMB-TC6	N	Hot
N-CAMB-TC8	N	Hot
N-CAMB-TC9	N	Cold
N-CAMB-TC10	N	Cold
ILC-062-N-CAMB-TC11	N	Hot

TC ID	Type	Hot Block or Cold Block
ILC-062-N-CAMB-TC13	N	Hot
NGNP-HTIR-TC41	HTIR	Hot
HTIR-TC-ZR2	HTIR	Hot
HTIR-TC-ZR3	HTIR	Hot

Table E-6. Summary log for second furnace run of 2018

TC ID	Construction Details	Performance Summary	Ultimate Fate
N-TD-TC-66 420 days of operation w/o failure	TD alloy sheath, MgO insulation, .062-in. diameter.	Read about 12°C high upon initial startup with hot block at 1250°C. Drifted down about 70°C over next 6 weeks with no end in sight. Insulation resistance was initially about 1300 ohms and gradually increased to about 2500 ohms.	As of 8/27/2018 reading 1190°C. This sheath alloy is not a substantial improvement over standard I600 as far as high-temperature drift is concerned. Read 1082°C at shutdown on 12/2/19.
HTIR-TC-62 420 days of operation w/o failure	Al_2O_3 insulation, Nb/1% Zr sheath. La-Mo/P-Nb thermoelements. 1600°C heat treat for 6 hrs. Mo protective sleeve.	Read about 44°C high (1294°C) upon initial startup with hot block at 1250°C. Drifted up about 4°C over next 6 weeks. Insulation resistance was initially about 7000 ohms and increased to about 25000 ohms.	As of 8/27/2018 reading 1298°C. So the drift has been minimal since startup. 9/17/18=1300°C. 9/24/2018=1301.5°C 11/20/18=1317°C 12/19/18=1321.7°C 1/30/19=1326.3°C 7/22/19=1284°C. It's back near where it started. Read 1281°C at shutdown on 12/2/19. 420 days of operation, 10,800 hrs. 13°C drift or 1.2°C/1000 hrs, which is very good. But this included a substantial "hump" in its reading (i.e., positive drift), which then turned negative.
HTIR-TC-64 145 days of operation, failed	Same as TC-62 except MgO insulation	Read about 38°C low (1212°C) upon initial startup with hot block at 1250°C. Drifted up about 2°C over the next 6 weeks. Insulation resistance was initially about 40 ohms and gradually increased to about 140 ohms. 40 ohms is sure low, but it doesn't seem to be affecting the reading much and it did increase as the test progressed.	As of 8/27/2018 reading 1214°C. So the drift has been virtually nil since startup. 9/17/18= 1210.5°C. 9/24/2018=1211.5°C 11/20/18=1196°C 12/19/18=1182°C 1/30/19=1173°C No chart readings after 1/30/2019 (failed) although resistances still look good. It had drifted a lot by the time of the last reading. MgO does not appear to be a good insulation matl (see HTIR-TC-65).

TC ID	Construction Details	Performance Summary	Ultimate Fate
HTIR-TC-66 190 days of operation, failed	Same as TC-62 except HfO_2 insulation	Read about 54°C low (1196°C) upon initial startup with hot block at 1250°C. Stayed essentially dead flat over the next 6 weeks. Insulation resistance was initially about 230 ohms and gradually increased to about 270 ohms.	As of 8/27/2018 reading 1197°C. No drift since startup. It is easier to solve calibration errors than drift problems so this is promising. 9/17/18=1197.5°C. 9/24/2018=1195.5°C 11/20/18=1201°C 12/19/18=1207°C 1/30/19=1208°C 3/26/19=1198°C 5/7/19=1184°C. Failed 5/9/2019. It did very well up through Mar 2019.
N-I600-TC-Al_2O_3 420 days of operation w/o failure	I600 sheath, Al_2O_3 insulation. Cable diameter of .062 in. Nb protective sleeve	Read 1093°C upon initial startup. This temperature generally agrees with the Cambridge Type Ns in the cold block (within 8°C). After six weeks showed a 2°C temperature decrease. Insulation resistance initially 7,000 ohms and stayed steady.	As of 8/27/2018 reading 1091°C. 11/20/18=1089°C 12/19/18=1088°C 1/30/19=1086°C 5/9/19=1065°C 7/20/19=1065°C 12/2/19=1062°C. 420 days of operation. 10,080 hrs. 31 degrees drift or -3°C/1000 hrs.
N-I600-TC-10 After 145 days of operation failed via virtual junction	I600 sheath, MgO insulation. Cable diameter of .062 in. Nb protective sleeve	Read 1085°C upon initial startup. After six weeks showed a 4°C temperature decrease. Insulation resistance initially 14,000 ohms which gradually increased to 24,000 ohms.	As of 8/27/2018 reading 1081°C. 11/20/18=1076°C 12/19/18=1075°C 1/30/19=1072°C This TC apparently developed a virtual junction when the furnace was restarted 3/26/2019. –13°C over 145 days (3500 hrs). about –4°C/1000 hrs drift. It started reading 200°C lower than before and continued the behavior through 12/2/2019 These two std Type N TCs (especially the MgO insulated version) are drifting a little more than the Cambridge TCs, and we would expect drift when operated above 1050°C. The dramatic superiority of the Cambridge Type N is not apparent until at least a temperature of 1100°C or 1150°C is reached.

The furnace was shut down 12/19/2018, restarted after the holiday break on 1/8/2019 and shutdown for good (for this phase) on 1/30/2019.

Summary of this test period. Of the three new HTIR-TCs the HfO insulated version performed the best and the MgO version performed the worst. We thought perhaps that the 1% Zr in the Nb sheaths would always result in upward drift, but that doesn't seem to be the case with this batch. The performance of the TD alloy sheathed Type N TC was disappointing. It drifted about as much as a standard Type N TC at 1250°C. The standard Type N TCs at 1085°C are drifting, but not severely at this temperature. In hindsight it would have been nice to have some data on standard Type N TCs in the 950 to 1000°C range. Likely their drift would be minimal in this range and this represents the top of the range for standard Type N TCs in the AGR-5/6/7 test. And would in general better define the "no drift" threshold temperature of standard Type N.

CAMB-TC13 failed at the end of this phase (1/30/19) upon furnace cooldown. Total operating time was approx. 10,400 hours and it had drifted downward approx. 25°C.

CAMB-TC6 and HTIR-TC41 also failed during cooldown 1/30/19.

HTIR-TC-ZR2 failed shortly after startup on 1/7/2019

APPENDIX F

2019 Test Log and Summary

3/25/2019–12/2/2019.

Started 3/25/2019 @1250°C hot block temperature.

Table F-1. New thermocouples installed for furnace run in 2019

TC ID	Type	Hot Block or Cold Block
N-CAMB-TC-1	Cambridge Type N	hot
N-CAMB-TC-5	Cambridge Type N	hot
HTIR-TC65	HTIR (MgO)	hot
HTIR-TC70	HTIR (Al_2O_3)	hot

CAMB-TC-1 and 5 are both .062 in. diameter Cambridge Type N TCs from the same lots as were used in the AGR-5/6/7 experiment. They were installed with zirconia protective sleeves in the high-temperature gradient area, (we did not have enough ZrO_2 sleeves to line the passageways in both the hot and cold block and so ZrO_2 sleeves were placed in the hot block only, which was thought to be adequate because most of the deleterious effects appear only at temperatures >1100°C). HTIR-TC65 and 70 are HTIR thermocouples (TCs) heat treated at 1600°C and insulated with either MgO (TC65) or alumina (TC70).

Table F-2. Thermocouples retained from the previous run

TC ID	Type	Hot Block or Cold Block
01-N-CAMB-TC8	HTIR	Hot
02-N-CAMB-TC9	HTIR	Cold
03-N-CAMB-TC10	HTIR	Cold
ILC-062-N-CAMB-TC11	N	Hot
HTIR-TC-ZR3	HTIR	Hot
N-I600-TC-Al_2O_3	N	Cold
N-I600-TC-10	N	Cold
HTIR-TC-62	HTIR	Hot
HTIR-TC-66	HTIR	Hot

Table F-3. Summary log of furnace run in 2019

TC ID	Construction Details	Performance Summary	Ultimate Fate
N-CAMB-TC-1 275 days of operation w/o failure	Special high purity Ni sheath, MgO insulation. .062-in. constant diameter.	Read about 7°C high upon initial startup with hot block at 1255°C. Drifted down about 7°C over the next 10 wks. Insulation resistance was initially about 800 ohms and gradually decreased to about 450 ohms.	As of 7/22/2019 reading 1246. Furnace is now at 1251°C. It has drifted about 12°C since March installation. Drifting about –4°C per 1000 hrs. 12/2/21 reading 1237°C. Nearly 6000 hrs. 22°C in 6000 hrs or –4°C per 1000 hrs. ZrO_2 protective sleeve.
N-CAMB-TC-5 275 days of operation w/o failure	Replicate of CAMB-TC-1	Read about 9°C high (1264°C) upon initial startup with hot block at 1255°C. Drifted down about 5°C over next 10 weeks. Insulation resistance was initially about 850 ohms and decreased to about 450 ohms.	As of 7/22/2019 reading 1251°C. Furnace is now at 1251°C. About a –9°C drift since March installation. Drifting about 3°C per 1000 hrs. 12/2/21 reading 1243°C. Drift of –21°C in 6000 hrs is –3.5°C per 1000 hrs.
HTIR-TC65 155 days of operation, failure	MgO insulation, pure Nb sheath. La-Mo/P-Nb thermoelements. 1600°C heat treat for 6 hrs. Mo protective sleeve.	Read about 20°C low (1235°C) upon initial startup with hot block at 1255°C. Drifted down about 30°C over the next 10 weeks. Insulation resistance was initially about 100 ohms and gradually increased to about 800 ohms.	As of 7/22/2019 reading 1179°C. So the drift is much worse than the Al_2O_3 insulated version. Failed 8/26/19. Temperature was 1170°C at failure.
HTIR-TC70 275 days of operation w/o failure	Same as TC-65 except Al_2O_3 insulation	Read about 35°C high (1290°C) upon initial startup with hot block at 1255°C. Drifted up about 3°C over the next 10 weeks (essentially flat). Insulation resistance was initially about 10,000 ohms and gradually increased to about 35,000 ohms.	As of 7/22/2018 reading 1282°C. So almost no drift since installation (the furnace temperature has been lowered about 5°C). It's easier to solve calibration errors than drift problems so this is promising. As of 12/2/201 read 1278°C. 6000 hrs testing. For an overall drift of roughly –2°C/1000 hrs.

The furnace was shut down 12/5/19, but the last data report was 12/2/19.

N-CAMB-TC-8 @1250°C continued to drift down but never failed

N-CAMB-TC-9 & 10 @1080°C didn't drift, didn't fail, and lasted 573 days without open circuit failure; i.e., totally successful performance in every way. We've never seen an in-core TC operate this long at a temperature this high.

HTIR-TC-ZR3 never failed and lasted 573 days but continued to drift up until it was 180°C above the furnace temperature (1250°C).

HTIR-TC 65 failed on 7/22/2019. Prior to this it exhibited a –65°C drift. It should be compared with HTIR-TC-64 which also suffered dramatic negative drift prior to its failure on 1/30/2019. Both had MgO insulation, and 1600°C heat treatment, but different sheath compositions.

See Appendix H for a summary of all the HTIR-TCs tested 2016-2019.

APPENDIX G

2021 Furnace Testing – Implications for AGR-5/6/7 Temperature Measurements

Figure G-1 shows a cross section of Capsule 3 in AGR-5/6/7 with calculated temperature contours about two weeks into the first irradiation cycle.

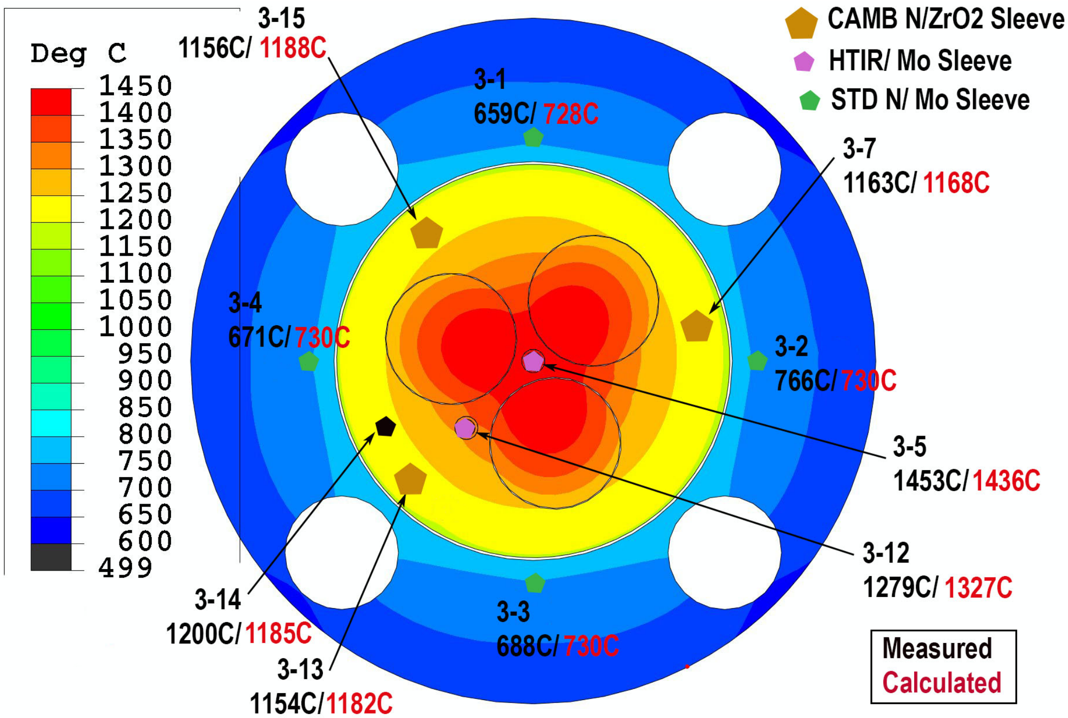

Figure G-1. Measured versus calculated temperatures for Capsule 3 early in first irradiation cycle.

When the AGR-5/6/7 experiment was started the expectation was that TC-3-5, which was placed in the middle of the fuel stacks in Capsule 3 (see Figure G-1), would immediately begin drifting down. This was because all the HTIR-TCs had been heat treated at 1450°C, and this was roughly the expected measurement temperature for TC-3-5. Once this capsule was brought to temperature (using the control TC, TC-3-13); TC-3-5 read about 1475°C. It was expected that this indicated temperature would immediately begin to fall (not because of changing temperatures but because the EMF was expected to drop because the TC had not been fully stabilized via a 1650°C, or higher, heat treatment). Instead, TC-3-5 climbed to 1500°C and held there steady for about 50 days, as shown in Figure G-2.

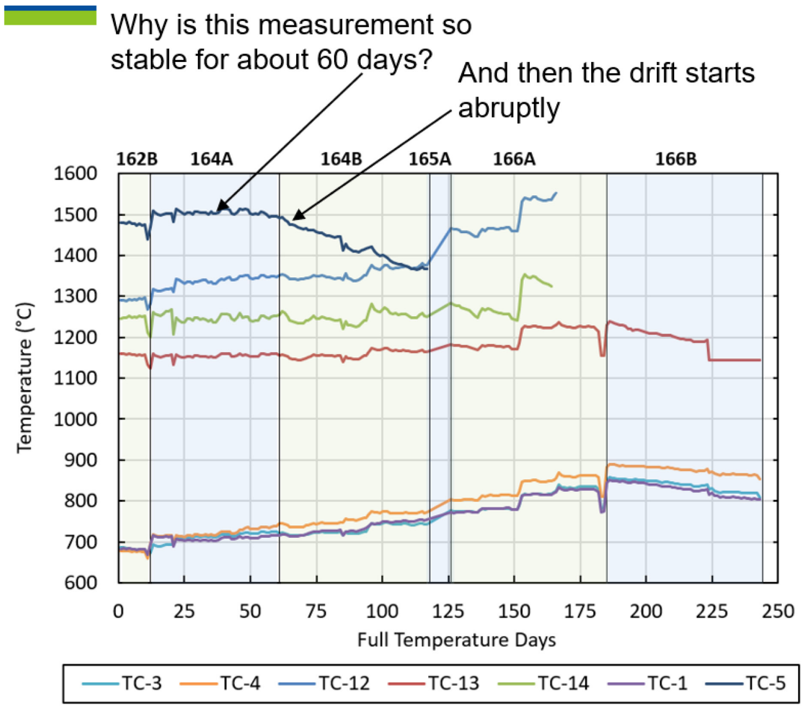

Figure G-2. AGR-5/6/7 thermocouple data, TC-3-5.

Based on the furnace testing performed in support of the AGR-5/6/7 experiment (Section 3.7), TC-3-5 should not have held steady at 1500°C, if the actual temperature being measured was roughly 1500°C. Could the actual temperature at this location have been ramping up and essentially canceling the downward drift? A nearby TC in Capsule 3, TC-3-12, was operating above 1300°C and gradually rose during the first 60 days or so rather than drifting down as would be expected from furnace testing (see Figure G-6). (However, TC-3-14, the next cooler TC, remained relatively flat.)

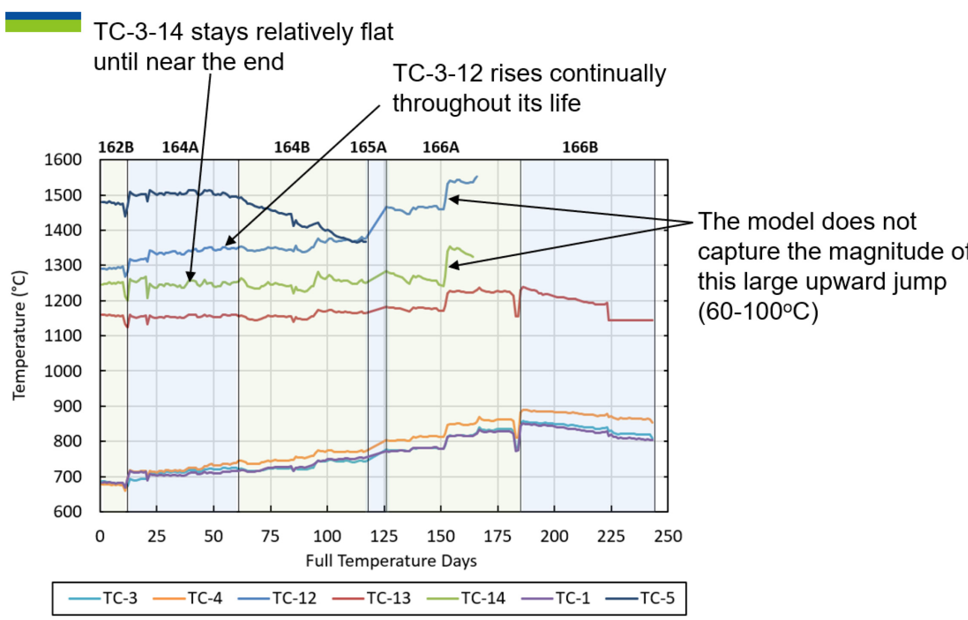

Figure G-3. AGR-5/6/7 thermocouple data, TCs-3-12, 13 and 14.

Figure G-4 shows a cross section of Capsule 1 in AGR-5/6/7 with calculated temperature contours about two weeks into the first irradiation cycle.

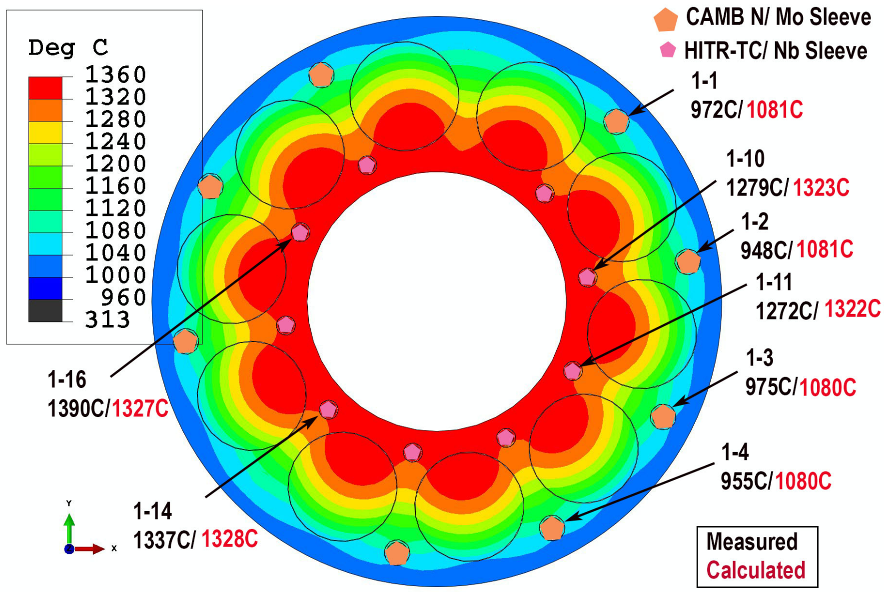

Figure G-4. Measured versus calculated temperatures for Capsule 1 early in first irradiation cycle.

The peak measured temperatures in Capsule 1 were about 150°C lower than in Capsule 3, or roughly 1400°C as shown in Figure G-5.

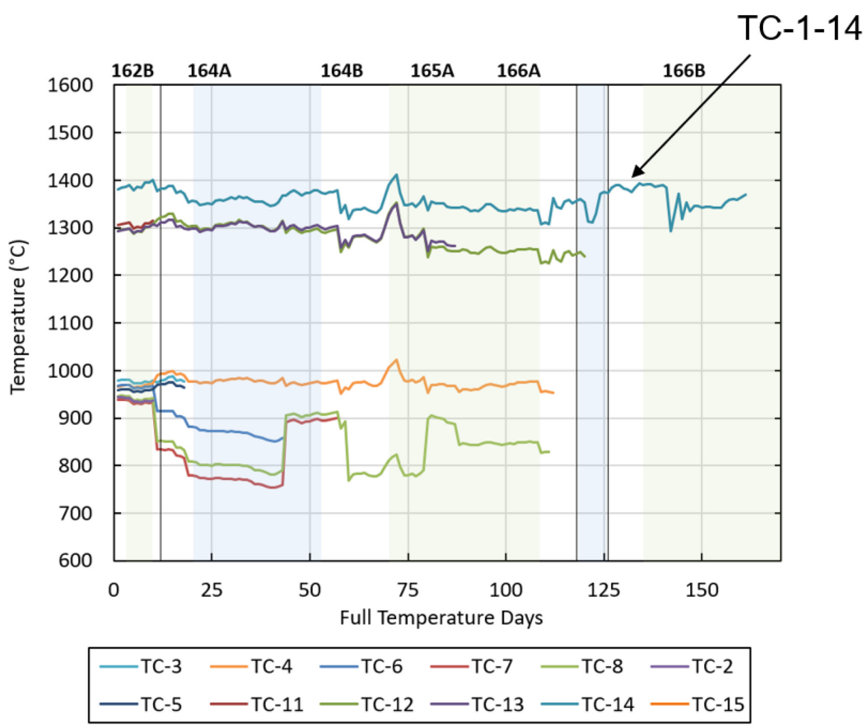

Figure G-5. AGR-5/6/7 Capsule 1 thermocouple data with TC-1-14 trend identified.

Based on testing described in Section 3.7, and Figure 7, and reproduced below as Figure G-6 below, any TC operating at 1350°C or above should have experienced substantial negative drift. With a temperature on TC-3-12 of 1550°C just before failure, we would assume a negative drift of more than 100°C (based on the trends shown in Figure G-6), and the difference between peak fuel and the temperature of TC-3-12, we would add another 100°C (based on Figure G-1). Based on this logic, a peak fuel temperature of 1750°C in Capsule 3 is not out of the question.

For Capsule 1, TC-1-14 had a maximum indicated reading of 1400°C, and assuming a TC drift of 50°C plus the difference in peak fuel temperature versus TC temperature of 30°C, results in a peak fuel temperature of perhaps 1480°C.

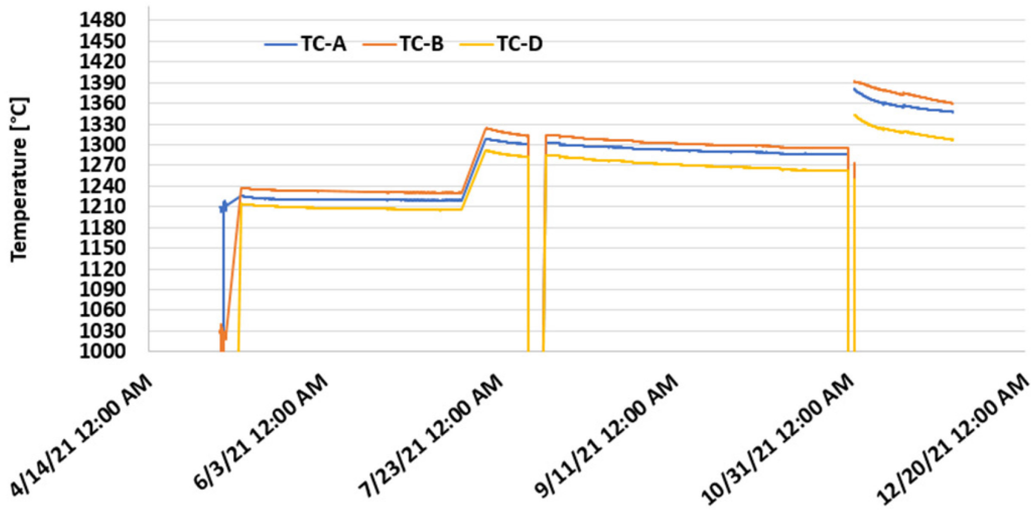

Figure G-6. Furnace test drift summary.

APPENDIX H
HTIR-TC Test Matrix/Summary

There are 12 permutations on the three HTIR-C variables: sheath composition (2), insulation (3), and heat treat temperature (2). With few exceptions, this summary covers 2016-2019 testing. Most of the TC variations tested in 2015 were considered non-prototypical and were discarded from further consideration.

Table H- 1. HTIR-TC testing summary

Permutation Code	TC ID	Initial Cal Error (°C)	Drift (°C/1000 hrs)	Time to Fail (days)$^{\pi}$	Comments
Nb/HfO/1600	HTIR-TC025	–110	–21	119	Improper heat treatment (2016). Information only.
Nb/HfO/1600*	TC06	–50	+12	10/7/2015	These two TCs were close to the original HTIR design (different sheath). They were made in 2015 and so had improper heat treatment. For info only.
Nb/HfO/1600*	TC09	–10	–25	10/7/2015	
Nb/HfO/1450	None				
Nb/Al_2O_3/1600	HTIR-TC53	+15	–1	286	All three of these TCs were tested at end of 2016, when calibration details were still being worked out.
Nb/Al_2O_3/1600*	HTIR-TC54	+60	+31	20	
Nb/Al_2O_3/1600*	HTIR-TC55	+85	+10	141NF	
Nb/Al_2O_3/1600*	HTIR-TC2	+1	–1.5	308NF	Excellent all-around performance.
Nb/Al_2O_3/1600*	HTIR-TC70	+35	–2	270NF	Minimal drift although calibration was about 3% off.
Nb/Al_2O_3/1450	HTIR-TCSAMPL1	+15	–4	308	These two TCs were the recipe used in AGR-5/6/7. Higher heat treat temperature provides even better performance
Nb/Al_2O_3/1450*	HTIR-TC22	+20	–1.7	340	
Nb/MgO/1600	HTIR-TC65	–20	–15	155	Dramatic negative drift. Compare with HTIR-TC-64
Nb/MgO/1450	None				
1%Zr/HfO/1600	HTIR-TC-66	–54	–2.5	190	This is the original HTIR recipe. Up until last 1000 hrs showed no net drift at all.
1%Zr/HfO/1450	None				
1%Zr/Al_2O_3/1600	HTIR-TCZR2	+9	+5.5	275	Drifted up like ZR1 but not as fast
1%Zr/Al_2O_3/1600*	HTIR-TCZR3	+34	+13.5	573NF	Rapid positive drift
1%Zr/Al_2O_3/1600*	HTIR-TCZR4	+21	+0	153NF	This unit did not drift like the other ZR units

Permutation Code	TC ID	Initial Cal Error (°C)	Drift (°C/1000 hrs)	Time to Fail (days)[π]	Comments
1%Zr/Al_2O_3/1600*	HTIR-TC-62	+54	–1.5	420NF	This TC broke the pattern compared to the others of similar construction (drifted down slightly)
1%Zr/Al_2O_3/1450	HTIR-TCZR1	–2	+11	187NF	Rapid positive drift made us concerned about this design
1%Zr/MgO/1600	HTIR-TC-64	–38	–11	145	MgO insulation in other designs caused rapid negative drift. Maybe this overwhelmed the tendency of 1%Zr to drift up.
1%Zr/MgO/1450	None				

*Replicate of the TC listed immediately above it

[π]NF = No Fail. Did not fail prior to removal from furnace

[a]Rows with gray shading designate sensors produced prior to correct heat treatment procedure being developed and are for information only (i.e., they do not represent the true performance of the materials combination).

[b]HTIR-TCs from 2021 testing are not included in this table because they were tested at higher temperatures.

What can be concluded from this table?

1. MgO insulation seemed to correlate with rapid negative drift.
2. 1% Zr sheath material combined with Al_2O_3 insulation correlated partially with positive drift. However, the one example of the original HTIR-TC design consisting of 1% Zr sheath material combined with HfO insulation exhibited very low drift.
3. Accurate initial calibration of HTIR-TCs was fairly rare.

APPENDIX I

"Fat End" TCs, Spinel Insulated TCs, Loose Assembly TCs

This appendix describes three Type N thermocouple (TC) variants that were tested in the IRC furnace. As well of the performance of these variants in AGR experiments.

FAT END THERMOCOUPLES

Larger thermocouples are known to have longer life while operated at high temperature. Figure I-1 reproduced below from Omega Corp shows their maximum recommended temperature versus TC size (for standard Mineral-Insulated (MI) cable).

Upper Temperature Limit in °C (°F) of OMEGACLAD®
Vs. Sheath Diameter

T/C \ Sheath Dia.	0.032" 0.8 mm	0.040" 1.0 mm	0.062" 1.6 mm	0.093" 2.4 mm	0.125" 3.2 mm
J	260 (500)	260 (500)	440 (825)	480 (900)	520 (970)
K & N	700 (1290)	700 (1290)	920 (1690)	1000 (1830)	1070 (1960)

Figure I-1. Omega Corp maximum recommended temperature versus thermocouple size (for standard MI cable).

We have also seen good performance in the Advanced Graphite Capsule (AGC) test series with 0.125-in. TCs. With this in mind, there was a decision to make when the TCs were selected for AGR-5/6/7, "would it be good to stick with the typical 0.062-in. design or go up a size to 0.093-in"? The number and size of TCs is limited by the cross-sectional area of the thru-tubes, which are the conduits through which TCs from lower capsules pass through upper capsules. An important point to note is that the highest temperature the MI cable experiences is typically at the junction or a region very close to the junction. With these constraints it seemed that perhaps a superior configuration would be to have the end of the MI cable (near the junction) larger than the rest of the cable (the part that passes through the thru-tubes). This design is illustrated in Figure I-2 below. Note that temperatures shown are just for illustrative purposes.

Two examples of TCs with this geometry were furnace tested in 2017 (see Appendix D), one was a Cambridge Type N design, the other utilized a standard I600 sheath but had Spinel insulation. The Cambridge design was tested in the furnace the longest of any TC, 18,200 hours, had low drift, and did not fail. The other, with I600 sheath, drifted rapidly (as expected), but also did not suffer open circuit failure.

Under the assumption that TCs of this geometry would likely survive better than standard 0.062-in.-diameter TCs, eight Fat End TCs were incorporated into Capsule 1 of AGR-5/6/7, seven were the Cambridge design, and one was I600 sheath with Spinel insulation. Also, four Fat End TCs were installed in Capsule 3. All four were I600 sheath and Spinel insulation.

These TCs did not perform particularly well during irradiation. All but one of the Capsule 1 Fat End TCs failed by the end of the third cycle and the last one failed in the middle of the next cycle. They were operating at temperatures in the 800 to 1000°C range. Standard design 0.062 diameter Type N TCs would be expected to perform approximately the same in similar conditions based on previous AGR experiments.

The four Fat End TCs in Capsule 3 lasted much longer than those in Capsule 1; they failed during the seventh of nine cycles. However, they would be expected to last longer than those in Capsule 1, since Capsule 3 is higher in the test train and the temperatures measured were lower (700–900°C). In comparison, only one of the standard 0.063-in. Type N TCs in Capsule 4 failed prior to the end of irradiation, and these TCs were operated at about the same temperature as the Fat End TCs in Capsule 3. Here again, this was not particularly good performance.

In summary, although there were good theoretical reasons for supposing the Fat End design would show superior performance to standard 0.062 inch TCs in this application, the in-service record did not bear it out.

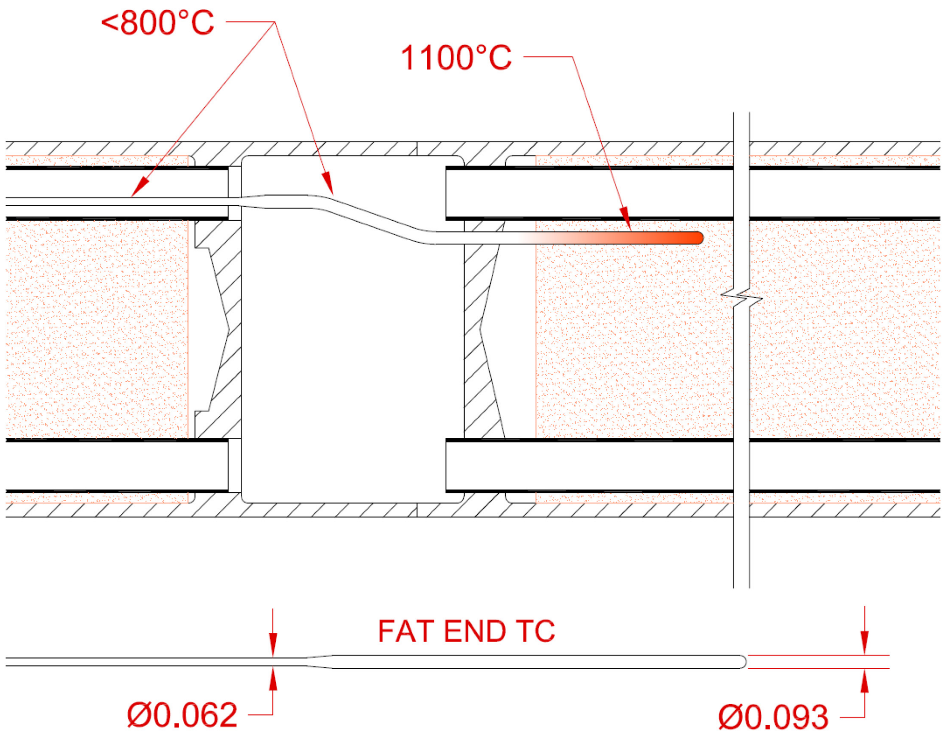

Figure I-2. Fat end TC illustration.

Spinel Insulated Thermocouples

The two most common insulators for MI cable are magnesia (MgO) and alumina (Al_2O_3). Another relatively common insulator is Spinel ($MgAl_2O_4$), which is essentially a combination of MgO and Al_2O_3. A patent (U.S. Patent WO1993005520A1) for $MgAl_2O_4$ insulated MI cable claims that $MgAl_2O_4$ is more stable under fast neutron irradiation than either MgO or Al_2O_3. Additionally, $MgAl_2O_4$ is relatively soft, like MgO, and thus is less likely to damage the thermoelements in a drawing or swaging process. Furnace testing of $MgAl_2O_4$ insulated cables indicated that they are marginally more resistant to drift than MgO insulated cables (see Section 3.1).

With these positive features in mind, $MgAl_2O_4$ was specified as the insulation material for some of the Fat End TCs incorporated into AGR-5/6/7, but as described in the previous section, this combination provided no observable benefit during service in the AGR-5/6/7 experiment.

Loose Assembly Thermocouples

Standard MI cable is made by threading “crushable” ceramic preforms over thermoelement wires, then sliding the sheath tubing over the strung together preforms. Finally, this assembly is pulled through a drawing die or through a swaging machine. In the drawing or swaging process, the insulators are crushed and tightly compacted around the wires.

In contrast, a loose assembly TC is made by threading “hard-fired” ceramic forms over thermoelement wires. A metal sheath is then placed over the ceramic pieces, but the insulation is never crushed tightly around the wires. The compaction process is what enables MI cable to be formed into relatively tight radii, while still keeping the thermoelement wires separated from each other and from the sheath. Also, the tightly compacted insulation protects the wires from uneven strain when the cable is bent.

What then is the advantage of loose assembly TCs? Because the ceramic is typically hard-fired and because it is not in tight contact with the sheath and thermoelement wires; the wire to wire, and wire to sheath electrical resistances are much greater at high temperature. Furthermore, as demonstrated in the testing performed in 2014 and 2015, some loose assembly TCs suffer much less drift than the standard Type N design with compacted insulation. Since the drift is due to solid-state diffusion of atoms from the sheath to the thermoelements, it makes sense that the loose assembly design would exhibit less drift. The atoms must jump the gap between the sheath and ceramics, and a second gap between ceramics and wires; additionally, the hard-fired ceramics themselves are potentially less permeable to solid-state diffusion.

Irradiation Performance of Loose Assembly Thermocouples

Three Type N, Mo sheathed, loose assembly TCs were incorporated into the top capsule (Capsule 6) of the AGR-1 experiment, and all three survived the entire irradiation while producing consistent readings. However, this was not a severe service environment. The TCs were only inserted 0.75 inch into the graphite fuel holder, giving them a very short heated-length, and the temperatures measured were relatively modest (700–800°C). Standard Type N TCs of the same size would likely have performed similarly. So this installation provided very little insight as to the usefulness of loose assembly TCs in very high-temperature reactor experiments.

2014 Furnace Testing

A total of six loose assembly Type N TCs were furnace tested in 2014. All were 0.090 inch diameter and sheathed with Mo. Four of these were incorporated into the initial complement of TCs in the first phase of 2014 testing (see first four entries in Table I-1 below). Two of these used crushable ceramic preforms rather than hard-fired insulation. This was done in the hopes that a TC of this type might survive the bending required to install in an AGR type test better than a hard-fired version. For unknown reasons, both units from this pair suffered premature failure (i.e., it is not clear if the insulation type had anything to do with the premature failure).

A final pair (with hard-fired insulation) was inserted into the furnace several months later and they performed very well until the furnace temperature took a short excursion above 1300°C. No base metal TC would be expected to operate long at 1300°C, and so the potential operating life of this pair at more reasonable temperatures is unknown.

Table I-1. Loose assembly TCs furnace tested in 2014.

ID	Insulation	Hours to Failure	Op. Temps	Comments
HRDAL-MO-090-A	Hard-fired alumina	4600	1150°C 1200°C 1250°C 1300°C	20°C positive drift over first 500 hrs. Extremely stable thereafter (see Figure I-3). Failed because 1300°C is too hot for base metal TCs.
HRDAL-MO-090-B	Hard-fired alumina	1900	1150°C	20°C positive drift over first 500 hrs. Stable thereafter (see Figure I-3).
CRSHAL-MO-090-A	Crushable alumina	250	1150°C	Very short life
CRSHAL-MO-090-B	Crushable alumina	1500	1150°C	Premature failure but no detectable drift prior to failure
HRDAL-MO-090-C	Hard-fired alumina	930	1200°C 1250°C 1300°C	No detectable drift prior to failure. Failed because 1300°C is too hot for base metal TCs.
HRDAL-MO-090-D	Hard-fired alumina	930	1200°C 1250°C 1300°C	No detectable drift prior to failure. Failed because 1300°C is too hot for base metal TCs.

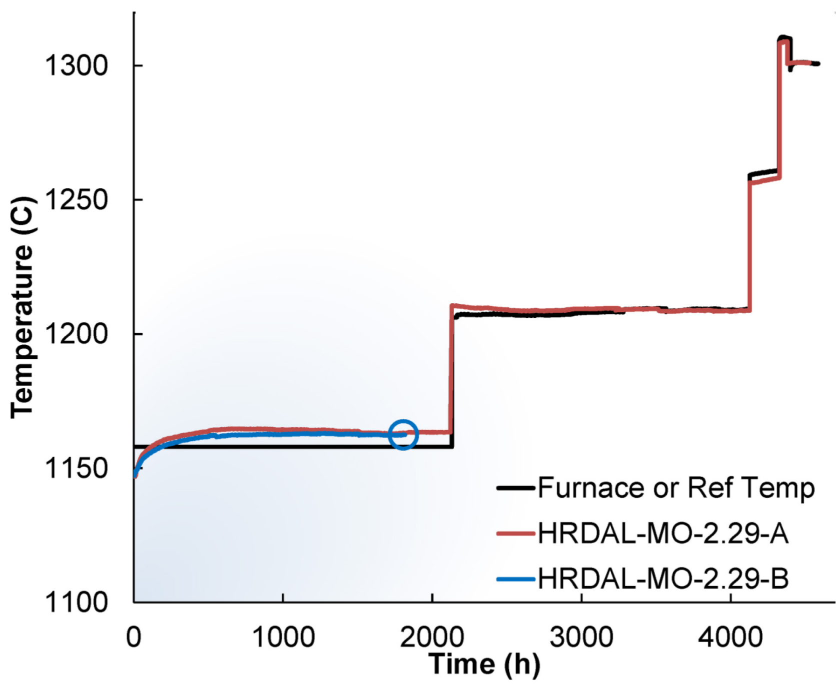

Figure I-3. First two hard-fired loose assembly thermocouples versus furnace temperature (2014 furnace run).

2015 Furnace Testing

A total of five loose assembly Type N TCs were furnace tested in 2015. All were 0.090 inch diameter and had hard-fired Al_2O_3 insulation. Two of the five had a pure Ni sheath, while the other three had standard Mo sheaths. Four of them were placed in the cold block, which operated at about 1100°C for a short time before the hot block's temperature was increased and the cold block's temperature followed to about 1200°C. The fifth TC was placed in the hot block, which ran at roughly 1300°C for 300 hours before the hot block temperature was increased to 1400°C, at which point this TC failed from thermoelement melting.

Table I-2. Loose assembly TCs furnace tested in 2015.

ID	Sheath	Hours to Failure	Op. Temps	Comments
HRDAL-MO-090-E	Mo	1200	Mostly 1200°C	No measurable drift prior to failure.
HRDAL-MO-090-F	Mo	1900	Mostly 1200°C	Showed some intermittency after 150 hrs (when furnace was shut down). Started drifting rapidly down after 700 hrs then failed open circuit about 500 hrs later.
HRDAL-NI-090-A	Ni	~1000	Mostly 1200°C	800 hrs with minimal drift followed by steady downward drift.
HRDAL-NI-090-B	Ni	~600	Mostly 1200°C	400 hrs with minimal drift followed by steady downward drift.
HRDAL-MO-090-SPARE	Mo	300	1300–1350°C	This TC showed some intermittency before the thermoelements failed by melting when the hot block temperature was raised.

Bend Test

Molybdenum-sheathed loose assembly TCs are typically not meant to be bent. However, because incorporation into an experiment such as AGR-5/6/7 requires a bit of offset (bending) to allow the cabling to pass through the thru-tubes (see Figure I-2), some practice bends were made in a TC of this type (see Figure I-4 below), showing that it is possible.

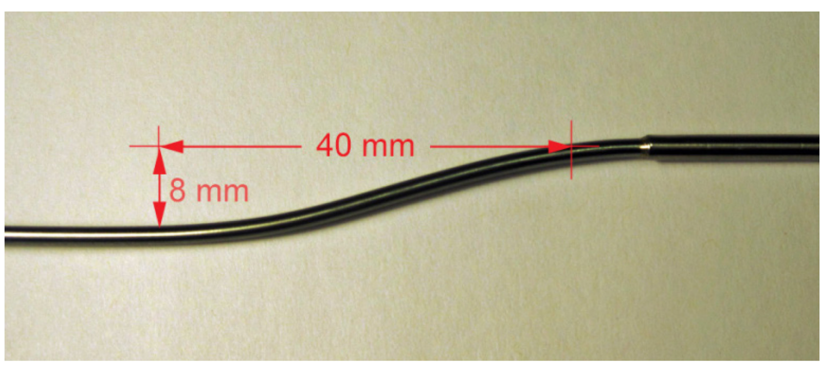

Figure I-4. Offset bend in a loose assembly, molybdenum-sheathed thermocouple.

Loose Assembly TC Summary

As seen from Tables I-1 and I-2, and Figure I-3, a few of the loose assembly Type N TCs were remarkably drift free. However, because of the higher than normal failure rate, no examples of this type were incorporated into the AGR-5/6/7 experiment. The potential of this TC design is intriguing, and they are worth further investigation. Perhaps with some modest design improvements this could be a very useful option for future experiments.