



# Qualification Test Requirements Report for High-Temperature Irradiation-Resistant Thermocouples

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## **SUMMARY**

The qualification of high-temperature irradiation-resistant thermocouples (HTIR-TCs) is set forth utilizing the Advanced Gas Reactor (AGR) 5/6/7 test in the Advanced Test Reactor (ATR) located at Idaho National Laboratory. The qualification requires the appropriate design, construction, and calibration of the HTIR-TCs, followed by proper installation and performance parameters.

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## ACRONYMS

|         |   |
|---------|---|
| AGR     | Advanced Gas Reactor                                |
| ATR     | Advanced Test Reactor                               |
| HTIR-TC | High-Temperature Irradiation-Resistant Thermocouple |
| M&TE    | Material and Test Equipment                         |
| TC      | Thermocouple  |

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## 1. GENERAL

High-temperature irradiation-resistant thermocouple (HTIR-TC) development has successfully passed through the design and calibration phase and entered the qualification phase. For qualification, the HTIR-TC underwent several months of testing in the Advanced Test Reactor (ATR)'s high-neutron-flux, high-temperature environment as part of the Advanced Gas Reactor (AGR) 5/6/7 test. In this test, several HTIR-TCs were placed throughout a test fixture designed to evaluate the performance of new advanced fuel designs for the AGR program. This report lists the requirements for this HTIR-TC qualification test.

## 2. REQUIREMENTS

The requirements for the HTIR-TC qualification test cover the design, construction, and calibration of the thermocouples (TCs) used in the AGR 5/6/7 test [1], as well as performance requirements based on the function and operational requirement report [2].

### 2.1 Design

The AGR 5/6/7 test included the following requirements for HTIR-TC design:

1. Length (of the mineral-insulated portion): 19–23 ft, in random increments (the unique length of each TC affords a means of distinguishing each TC post-installation)
2. Diameter: 0.062 in.  $\pm$  0.002 in.
3. Insulation: Alumina (Al<sub>2</sub>O<sub>3</sub>), 99.4% minimum purity
4. Junction: Ungrounded
5. Sheath: Nb (Nb 99.9% minimum), chemistry per ASTM B394
6. “+” Thermoelement: La-Mo (La 0.5–1.0%)
7. “-” Thermoelement: Doped Nb ( $P \leq 1000 \mu\text{g/g}$ ,  $Ta \leq 3000 \mu\text{g/g}$ , Nb balance)
8. Transition cup: Epoxy only, 0.19 in. maximum diameter
9. Extension wires: 24 AWG, duplex Teflon insulated, 72 in. long
10. Heat treat after assembly at  $1440 \pm 20^\circ\text{C}$  for 6 hrs  $\pm$  1 hr. At minimum, this heat treatment shall cover the lower 48 in. of the TC assemblies. Heat treat temperatures shall be measured by furnace control TCs.

### 2.2 Construction and Calibration

The AGR-5/6/7 test included the following requirements for HTIR-TC design, construction, and calibration:

1. All TC assemblies shall be fabricated per an approved fabrication plan (step-by-step instructions)
2. Basic assembly instructions will be developed, approved, and serve as a work control document, thus ensuring a reproducible product that will meet the requirements with little or no rework. These instructions will include:
  - a. A logical listing of the work steps needed to complete the TC assembly. The amount of detail on each step should be sufficient to enable a similarly trained individual to complete the TC assembly with little or no assistance from the original assembler.

- b. Non-destructive examination hold points, along with performer signatures to reflect completion of those hold points
  - c. Performer signatures identifying the completion of critical assembly points where measurement and test equipment (M&TE) is used
  - d. M&TE identification numbers will be captured in the work control instructions or inspection forms
  - e. Certifications for materials used in the TC fabrication will be attached to the instructions
  - f. Instructions for TC calibration in the event it is not conducted by the site calibration laboratory
3. These instructions (i.e. 2.a. – 2.f.) will be approved by quality assurance (QA) prior to beginning the TC fabrication/assembly
  4. Material control will be maintained for all TC components identified on the TC traveler. General material control will include the following:
  5. Identification numbers for the materials used in each TC shall be identified on the TC traveler
  6. The TC traveler shall include a verification signature proving that these materials were indeed used in the TC's fabrication
  7. All M&TE used to verify the TC testing shall be calibrated by the Idaho National Laboratory calibration laboratory or another approved laboratory on Idaho National Laboratory's Qualified Supplier List; otherwise, the M&TE will be self-calibrated using traceable standards from the National Institute of Standards and Technology.
  8. Individual TC calibration: Each completed TC assembly shall be individually calibrated in 100°C increments from 700°C to 1400°C. The curve between 0 and 700°C shall be generated by assuming 0 mV for 0°C. Additionally, the mV reading for each TC at the boiling temperature of water shall be provided. Electromotive force values for each TC assembly shall be provided, along with the associated polynomial coefficients.
  9. The TC traveler sheet(s) with the attached material and calibration certifications will be completed and reviewed prior to turning copies over to the AGR program.

## 2.3 Installation

The AGR-5/6/7 test rig, a vertical assembly approximately 48 in. (122 cm) in length, is installed in the ATR core. The test rig assembly holds five capsules, each containing new test fuel that, when irradiated with neutrons, produces the high temperatures measured by the TCs in the capsules. Capsule 3 is the middle capsule located in the core center. Capsule 1 is located toward the bottom of the core, and Capsule 5 toward the top. Several HTIR-TCs are located in Capsule 3 (which sees the highest temperature), along with several HTIR-TCs in Capsule 1 (which sees lower temperatures). Considering the sharp temperature gradient in each capsule, it is important to position the TCs with exact accuracy so as not to incur errors due to misplacement in a temperature gradient. The TCs in Capsule 3 can read temperatures of ~1500°C and neutron fluxes of  $\sim 2.8 \times 10^{14}$  nv thermal and  $\sim 2.2 \times 10^{14}$  nv fast (>1 MeV) (Ref. 3). For comparison purposes, other TC types (i.e., types N and K) are also installed in the test rig, which is designed to flow gas into the capsules to remove the fission gas released from the test fuel. But that gas also flows around the TCs and can affect the TC temperature readings.

## 2.4 Performance

For HTIR-TC qualification testing in the ATR's high-flux, high-temperature environment, the key HTIR performance requirements are as follows:

1. At installation, HTIR-TCs should be able to accurately measure the expected maximum temperature in the AGR-5/6/7 capsules (approximately 1550°C).
2. The accuracy requirement for temperature measurements taken by individually calibrated HTIR-TCs is  $\pm 1\%$ . It is understood that, due to theoretical modeling errors, errors due to potential TC misplacement in high-temperature gradients, and potential changes in the gas flow around the HTIR-TCs, the temperature readings may not agree with the theoretical estimates. The qualification test requires that the HTIR-TC temperature readings agree within  $\pm 5\%$  of the theoretical readings.
3. At constant reactor power, the HTIR-TC temperature readings should constantly fall within the calibrated accuracy of  $\sim 1\%$ . However, variations in HTIR-TC temperature readings could occur due to variations in the ambient temperature caused by variations reactor power and other local ambient temperature fluctuations, detectable by noting whether the variations read by all the capsule TCs are synchronous.
4. When the reactor shuts down from 100% power and then returns to 100% after several hours or days of shutdown, the HTIR-TC readings both before and after the shutdown should be reproducible to within the calibrated accuracy of the TCs in the test rig.
5. For the HTIR-TC qualification test, the maximum sensor drift in the ATR test fixture is -3.5% over a period of approximately 125 equivalent full-power days. This duration is based on the expected thermal, fast flux, and temperature profiles over the length of the HTIR-TC cables in the ATR. The drift is caused by changes to the Seebeck coefficient, primarily due to transmutations caused by thermal neutrons, material damage due to fast neutrons, and lattice changes due to prolonged residence at high temperature. The magnitude of these effects is unknown and would need to be determined from the drift data. Extrapolation of the drift results in the qualification test should show the expected HTIR-TC drift in a commercial boiling-water reactor or pressurized-water reactor to be  $< 1\%$  for 18 months when measuring temperatures of  $< 1000^\circ\text{C}$  and  $< 1\%$  for 24 months using a lower and more realistic neutron absorption cross section.
6. Once the TC reaches -3.5% drift in the ATR test fixture, this is also an appropriate indication of the TC's end-of-life. This is because, for larger exposure, the drift would increase, and the TC reading would become too inaccurate. For fuel test applications in a test reactor, an excessive number of thermal shocks can also limit the life of the HTIR-TC. For the HTIR-TC qualification test, a thermal shock requirement of five rapid startups and five rapid shutdowns (10 shocks in total), each covering a temperature range of room temperature to  $\sim 1500^\circ\text{C}$ , was determined as the thermal shock end-of-life requirement. For the HTIR-TC qualification test, the HTIR-TC life extends until either the 10 thermal shocks or a drift of -3.5%, whichever occurs first.
7. The life requirement described in the item above does not consider the potential effect of insufficient heat treatment for those TCs reading very high temperatures (i.e.,  $> 1450^\circ\text{C}$ ). The TCs were heat treated for 6 hours at  $1450^\circ\text{C}$  and operating at higher temperatures for a prolonged length of time can potentially change the TC Seebeck coefficient, altering the sensitivity and life of the TC. The effect of this heat treatment issue has never been estimated, and evaluation of the reactor qualification test results is expected to provide data for enabling this effect to be quantified.

### **3. REFERENCES**

1. Palmer, A. J., "HTIR Thermocouples for AGR-5/6/7," SPC-1963, Rev. 3, 2017.
2. Skifton, R., "Function & Operational Requirements for High Temperature Irradiation Resistant Thermocouples Developed By The In-Pile Instrumentation Program," INL/EXT-21-63173, 2021.
3. "Advanced Test Reactor National Scientific User Facility Users' Guide," INL/EXT-08-14709, 2009, ATRUsersGuide.pdf.