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The Development of Calibration Procedures for High Temperature Irradiation Resistant Thermocouples

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Abstract

A detailed description of the optimization of heat treating and calibrating procedure for Idaho National Laboratory's High Temperature Irradiation Resistant thermocouples (HTIR-TC). Also discussed, is the implication of the procedures on the overall performance of the HTIR-TC finished product, in particular, for the case of long lead sections that are typical of sensors deployed in nuclear reactors. The effect on the measurement accuracy of fluctuations of the reference temperature and localized heating along the sensor lead is also investigated. A calibration graphical user interface (GUI) and accompanying script is presented for unifying the calibration process. A 5th order polynomial fit was found to be best for fitting HTIR-TC calibration data. The performance of the HTIR-TCs subject to the optimized heat treatment and calibration procedures is shown to be robust and consistent with other TC industry standards. Electromotive force curves versus length of the TC are presented to clarify positioning of TCs, in general, and how it can affect the calibrated temperatures.

Keywords: irradiation resistant sensors, in-pile instrumentation, high temperature sensors, thermocouple, HTIR TC

I. Introduction

Extensive research studies have been done on the build, performance, and longevity of the Idaho National Laboratory's (INL) patented High Temperature Irradiation Resistant thermocouple (HTIR-TC).[1] However, the heat treatment and calibration processes performed during the sensor fabrication can have important effects on the accuracy of the finished product, and, as of yet, there has been no published study on the development and optimization of such processes.

A brief look at the history on the INL HTIR-TC reveals that it can withstand high temperatures, multiple thermal cycling, and in-pile irradiation for extended periods of time without significant aging (drift) and/or transmutation to the thermoelements and other components of the TC.[2, 3] This has an advantage over other temperature sensors where melting and/or calibration drifting would occur (e.g. fiber-optics, thermochron loggers, etc.). However, inadequate heat treatment processes during fabrication in combination with temperature gradients along the length of the TC during operation have caused significant performance decrease -- sometimes causing nearly 130 °C error in the temperature measurement. The current objective is to demonstrate that performing a significant enough heat treatment leads to temperature readings of the TC that are not overly sensitive to temperature gradients along the TC, and to greatly reduce any drift from long soak times at elevated temperatures (i.e. 1500 °C).[4] Further, by improving the calibration process, measurement errors from minor extrapolation outside the calibrated temperature range are ensured.

II. HTIR-TC Thermocouple Thermometry

In general, a thermocouple is designed to utilize the Seebeck effect of dissimilar metals that create electromotive force (EMF) during temperature differences, that is

$$dV = -S(T)dT, \qquad (1)$$

where dV is the voltage across the two TC thermoelements, S(T) is the temperature-dependent material property known as the Seebeck coefficient, and dT is the overall temperature difference between TC junction (measurement location) and a reference temperature.

It has also been shown that the EMF can be calculated by means of the length of each thermoelement that comprises the circuitry by,

$$EMF = \int_0^L \epsilon_1 \frac{dT}{dx} dx + \int_L^0 \epsilon_2 \frac{dT}{dx} dx \qquad (2)$$

where L is the length of the thermocouple, ε is the total thermoelectric power of the material (equal to the sum of the temperature derivative of the Peltier coefficient and the Thomson coefficient[5]), x is the distance along the wire, and T is the local temperature with respect to x. When the wire is not exactly uniform or multiple wires are integrated into the circuitry (e.g. copper extensions/leads), then each individual wire and/or composition must be accounted for by adding more terms to Equation 2 -- which leads to the following

$$EMF = \int_0^{l_1} \epsilon_1 \frac{dT}{dx} dx + \int_{l_1}^{l_2} \epsilon_2 \frac{dT}{dx} dx + \cdots + \int_{l_n}^0 \epsilon_n \frac{dT}{dx} dx \qquad (3)$$

with n being the n^{th} wire in the series.

There are many TC builds and/or configurations. The most prevalent being the mineral-insulated, metal-sheathed (MIMS) type. Others include the intrinsic junction (i.e. the thermoelements are bare and integral to the substrate being temperature measured), a grounded sheath design, or a double walled TC as seen in Scervini.[6] Each have their own advantages and disadvantages.

Looking more specifically at the HTIR-TC, the simple circuitry can be seen in Figure 1. The sensing temperature, T_{sense}, is where the thermocouple's temperature measurement is located. T_{ref} is the reference temperature in regards to T_{sense} (i.e. in this case the cold junction of the TC), and is ideally held constant. The cold junction can also be necessary when converting from a more expensive and/or exotic thermoelement material to something more common (e.g. copper).[7] T_{meter} is utilized in the recording of the overall difference in temperature in relation to T_{sense} in the absence of a reference temperature. Although some liberties can be taken when connecting a TC to a data acquisition system,[8] it is important to keep T_{ref} on both wires as constant as possible. However, in applications that require the deployment of HTIR-TCs, such as testing of nuclear fuel and materials in Material Test Reactors (MTR), it is often challenging to establish that constant temperature during operation. This study proposes a method to minimize the effect of temperature fluctuations and quantifies the expected error.

III. Heat Treatment Prior to Calibration

Ideally, the entire length of the thermocouple conductors are heat treated to achieve uniform wires from a state A ("as manufactured" condition) to a state B (heat treated condition).[9] Yet, the heat treating of a coil of wire prior to the construction of a TC can lead to fusing the wire to itself if not carefully set up and/or brittle thermoelements that break when handling. Also, after constructing the TC, some thermocouples are 10+ ft [3+ m] long (longer than most commercial furnaces), therefore, in order to achieve a uniform calibration of the thermocouple wires, at least the length of HTIR-TC that undergoes any temperature gradient (>60 °C) should be heat treated above the highest expected temperature to be measured by the TC. In the case of the HTIR-TC, the long history of testing has shown that a period of approximately 4 hours at a temperature typically 50 °C above in-use expected temperatures is sufficient to reach stable heat treated conditions. The limit of 60 °C considered for the temperature gradient is not only a common test reactor coolant temperature, but also the lower limit where the EMF generated in the TC becomes significant. The length of heat treatment being referenced should be from the tip of the TC to the end of the temperature gradient (e.g. outside the furnace, pressure vessel, etc.). If this length is still longer than the heat treating furnace then the process must be done in steps, in which the TC is moved with respect to the furnace isothermal zone and the process is then repeated at each step. The furnace used for this study is a Carbolite TZF 18/600 with a measured isothermal zone of 18 in [46 cm] requiring 3 steps for a typical HTIR-TC used for the Advanced Test Reactor irradiation tests of thermal gradient lengths up to 48 in [122 cm].

IV. Calibration Setup

After finalizing the heat treatment, the TC must be calibrated to relate the measured voltage to temperature. This is done by positioning a calibrated Type B TC into a tube furnace, but opposite of the HTIR-TC being calibrated. The HTIR-TC is in line with an ice-point cell to provide a constant reference temperature needed in the measurement. The furnace is brought up to temperatures between 700 °C and 1400 °C. Each temperature of interest is held for 1 hour with 0.5 Hz sampling rate, guaranteeing a sample size of 1800 samples (at most) at each temperature -- approximately half of the samples in thermal equilibrium based on the inertia of the furnace used in the tests.

These specific temperatures are then repeated on the furnace cool down phase to show the in-process repeatability of the thermocouple. The TC is rejected if any repeat temperature has drifted beyond 1.5% the initial temperature reading.

The HTIR-TC is deployed in high temperature experiments, with a typical range of operation of 700 °C – 1600 °C. The furnace used for calibration at these target temperatures does well in this temperature range, but cannot hold accurately at temperatures below 700 °C. This leads to a significant gap in calibration data below 700 °C that decreases significantly the accuracy of the calibrated response at low temperatures. To improve this, EMF readings of boiling water temperature (~100 °C) and ambient room temperature (~20 °C) are utilized as calibration points.

IV. a. Calibration Setup without Constant T_{ref}

As mentioned earlier, it is important that T_{ref} remains constant, but in the event that an ice-point cell (or similar means) is not readily available in the field, the thermocouple cold junction -- depending on the experimental setup -- could fluctuate significantly enough with ambient room temperature. This, in turn, affects the measurement of the thermocouple by fluctuating both hot and cold ends of the temperature spectrum, ΔT. To accommodate any reference temperature fluctuations, the following procedure can be performed on the HTIR-TC, as seen in Caldwell.[10] A tip of the HTIR-TC is submerged in a boiling water bath (~95 °C, at test location). The reference junction is then consecutively placed in an ice-point cell, ambient room temperature, and finally the palm of a closed fist -- each respectively with a calibrated Type K TC. The HTIR-TC EMF (i.e. voltage) is recorded in each scenario -- as presented here in Figure 2. The data is then fit to a linear trend. This linear fit can now be utilized when recording data that is not utilizing an ice-point cell or similar temperature constant for the reference temperature when the original calibration did use one.

The recorded temperature errors of a HTIR-TC with and without an ice point cell is presented in Table 1. The first column shows the HTIR-TC temperatures as calibrated from an identical run while utilizing an ice point cell in line with the TC circuitry. The second column is a representation of using corrected temperatures from the procedure discussed above and applying the linear curve fit found in Figure 2. The voltage variation of the cold junction temperature is added to the EMF reading of the HTIR-TC -- which allows for a more accurate temperature to be read from the current overall calibration of the HTIR-TC. The improvement is seen most prevalent in the lower temperatures of the reading as the cold junction temperature difference is minimal at best (i.e. <60 °C), which makes up most of the EMF at these lower temperatures.

V. Calibration Software

In order to achieve consistent calibrations, TC EMF data (i.e. voltages) are compared to known Type B TC temperatures through a curve fitting software written as a MATLAB script and a graphical user interface (GUI). This provided the tools necessary to fully import data, calculate curve fits, compare with previous calibrations and/or the EMF of other commercially available thermocouples, and export the data to a report. Also, the report allows a side by side comparison of temperature readings both up and down the calibration run, rejecting any that are greater than the 1.5% relative error criterion.

The calibration GUI, seen in Figure 3, and accompanying script imports the data, followed by plotting the data to a figure that is able to be selected by the user. The user chooses the voltages of interest by clicking first and second locations at each temperature plateau (i.e. 1 hour of data discussed above). The software would then record and average between the two locations. After all the data is selected in this manner, the user moves onto the calibration curve fitting. A drop down menu allows the user to select which polynomial order to use on the calibration between HTIR-TC voltage outputs and Type B TC temperatures. The option is then presented to manually insert any data points of known values into the GUI. For example, the check box to use the origin, (0, 0), as a data point can be selected. This should be typical of all thermocouples using an in-line ice point cell during calibration, i.e., 0 °C should be 0 V -- given that the ΔT at that measured temperature would simply be 0 °C -- and thus would generate no EMF. As well, the option for forcing a minimum extrema at the origin could be selected; where the minimum extrema at the origin may be needed given the specific polynomial order. This gives more control of the slope of the calibration curve at the origin. The final step in the calibration process is to generate a report of all the relevant information into an excel spreadsheet. The option to plot the calibration curve against other common industry standards and to select which values of interest both while heating up the furnace (connoted as Upward) and cooling the furnace (connoted as Downward) are available.

The calibration curve best fit is found using the least squares method by adjusting the polynomial coefficients in order to minimize the residual sum squared of the difference,

$$S^2 = \sum_{i=1}^{n} {r_i}^2 = \sum_{i=1}^{n} (y_i - \hat{y}_i)^2 \qquad (4)$$

with y being the measurement (in this case time-averaged, calibrated temperatures, as described above), ŷ is the modeled temperature output at the point of interest, i is the current index of the data point, n is the total number of data points (i.e. time-averaged measurements), and r is the residual of actual to modeled data.

The goodness-of-fit of the calibration is most usefully seen as the standard error, which is simply the square root of S^2 from Equation 4. This output quantifies to the user how well the polynomial curve is fit to the data by way of temperature variation.

VI. Calibration Results

During the calibration process the voltages are recorded across the wire leads on the HTIR-TC at each temperature. These voltages are then time averaged. Consistently, the voltage readings have a sample size >500 samples, and a relatively small standard deviation about the mean of 6 µV.

The HTIR-TCs, as in times past, have shown robust performance when compared to other thermocouples.[11] The signal is responsive and accurate in the temperature range between 400 °C and 1600 °C. This can been seen in Figure 4 when compared to TC Types B, S, R, and N. The different comparison thermocouple types presented here are common industry standards with the Type B, Type S, and Type R TCs comprising of thermoelements that are Platinum based with varying degrees of a Platinum – Rhodium alloy, and the Type N TC comprising of nickel based thermoelements (i.e. Nicrosil and Nisil).

As mentioned, the calibration process asks for which polynomial order to use. The common school of thought is that the higher the polynomial order the more accurate. This is not always necessarily true for the following reasons: 1) the higher order may not be gaining (much) more information and/or reducing error in the curve fitting, and 2) artifacts of the higher order polynomial will begin to be present which are seen as small wavy lines in between any two data points. Therefore, the order must be high enough to reduce significant standard error, but small enough to avoid the two aforementioned issues. The standard error performance of each curve fit for the HTIR-TC was sequentially computed and recorded starting at 3rd order and increasing to 6th order, with the outcome presented in Figure 5. There is considerable error reduction from 3rd to 4th order (i.e. S = 1528.3 to S = 569.9 or 62.7% decrease), and then again from 4th to 5th order (i.e. S = 569.9 to S = 19.7 or in other words a 98.7% decrease from 3rd order). Finally, from 5th to 6th order there is not significantly enough error reduction to justify a 6th order fit (i.e. S = 19.7 to S = 8.6 or 99.4% decrease from 3rd order). Therefore, this leads to utilizing 5th order polynomial fits for each calibration of a HTIR-TC -- one of which can be seen in Figure 4 to fit the data points very well.

VII. Effect of Temperature Gradients on HTIR-TC Output

Following the graphical analysis of Moffat[8] on the topic of generating EMF curves (i.e. EMF vs. Temperature), the current note provides *spatial* analysis on the EMF generation of HTIR-TCs (i.e. EMF vs. length of TC thermoelements). This is necessary to provide an understanding of proper heat treatment on the thermocouple tip, and, in turn, to emphasize the effect of insufficiently heat treating enough length of the TC wire. Figure 6 shows the individual calibration curves of various TC materials when held against a platinum standard. To generate these various curves, the cold end of an X vs. platinum TC is held at 0 °C while the other end is held at T_{HOT} -- where X is the material of interest. It is interesting to note that both HTIR-TC thermoelements have positive slopes when individually held against platinum. However, when molybdenum and niobium are paired to form the HTIR-TC, molybdenum acts at the positive leg of the TC and niobium acts as the negative leg. To be consistent with others and for the purpose of this section, the following figures use the slopes from Figure 6 -- i.e. the calibrated slopes of molybdenum and niobium when individually held against the industry standard of platinum.

A simple representation of a HTIR-TC circuit is seen in Figure 7 with a corresponding schematic of the EMF being generated along the wires. Position 1 starts the analysis of EMF being generated by the molybdenum thermoelement. Moving along the length of the molybdenum wire to position 2, the wire remains at ambient temperature. As EMF is only generated during a temperature gradient, the EMF curve stays at a horizontal. Going now to position 3 of the TC, the molybdenum wire has gone through a temperature gradient (i.e. cold to hot) from ambient temperature to T_{HOT}. This naturally generates EMF, and the slope provided from Figure 6 is applied to the EMF curve until the temperature gradient has stopped. Also, the TC thermoelements have changed through continuum from the “as manufactured” wire, state A, to the heat treated wire, state B. The state B wire has a slightly smaller slope (i.e. closer to horizontal) than the state A wire. This is shown in Figure 6 as solid or dashed lines near molybdenum and niobium. There may be a length of the TC that is now in the isothermal zone of T_{HOT}, as shown. Following the junction of molybdenum to niobium at position 3, the same procedure is applied along the niobium wire to position 4. This length of wire has the temperature gradient in reverse to the molybdenum wire -- as the length of wire goes out of T_{HOT} (i.e. hot to cold). In this regard, the identical slope from Figure 6 of niobium is now placed here to show the EMF as decreasing. Lastly, the niobium wire is now at ambient temperature until the sensor reads the EMF, at position 5, across the two wire leads.

For an ideal TC the measurement of EMF generated by the thermoelements are between a constant reference temperature (typically 0 °C) and the temperature of interest. This creates the ΔT utilized in Equations 1 and 2 where only one part of ΔT is varying. To analyze the circuitry with a reference temperature Figure 8 is provided. Following the same path as before (e.g. 1 to 2, 2 to 3, etc.) the circuitry starts with copper going from room temperature down to the ice point cell temperature, T_{REF}. Borrowing the slope of copper from Figure 6 going from hot to cold decreases EMF generation. This is immediately followed by a molybdenum junction in the ice point cell as the temperature goes from cold to hot. This leads to applying the slope for molybdenum from Figure 6. From position 3 to position 4 the state B molybdenum wire is now increasing in temperature along this length to the final high temperature, T_{HOT}. The slope is slightly different than the one coming out of the ice point cell as the molybdenum is now state B heat treated wire. Now in reverse direction going down the length of the niobium wire (i.e. going from leg 4 to 5), the niobium is going from hot to cold, hence decreasing the EMF generated. Lastly, the niobium enters the ice point cell and couples with an identical copper wire as before (e.g. position 1 to position 2). The final EMF is measured across the two copper leads; which is larger than the EMF reading found in Figure 7, as the ΔT is now referenced from 0 °C.

In the event that the temperature gradient extends beyond the heat treated wire into the "as manufactured" wire, Figure 9 is provided for analysis. A similar step-by-step process as the two previous figures is used to analyze this circuit. This schematic shows that the slight increase or offset in EMF to the overall EMF generation is from the state A segment of the wire -- between leg 3--4 and leg 6--7. This gives raise to the situation where the TC calibration is being performed within state B wire only, and then EMF is being generated in state A lengths, giving an offset reading to the actual temperature being sensed by the TC. Notice the further increase in EMF being generated from that of Figure 8. This means that care must be taken to position a TC that has only been heat treated partially, or the TC is excessively used then inserted deeper into any temperature gradients. In relation to this, it is of importance to note that if the calibration setup in regards to TC positioning in the furnace is not carefully executed, and temperature gradients extend beyond the state B heat treated wire, then naturally, the TC will only be within calibration at that exact insertion. However, if the TC is calibrated well within the bounds of the state B wire, then the calibration should hold for all positions of insertion -- provided the temperature gradient stays within the state B wire as discussed above.

To give emphasis to the schematic output of Figure 9 an HTIR-TC was inserted into the furnace 6 in [15 cm] from center line, and held at various temperatures: room temperature, 250 °C, and 700 °C. A resistance heater was attached over the transition between state A and state B wire and insulated. The resistance heater was ramped up to 70 °C, and the voltage across the TC leads was recorded. Figure 10 shows that the temperature can be effected by this temperature gradient across the transition from state A wire to state B wire by 6 °C -- 8 °C. The 250 °C case is oscillating due to the furnace having trouble at these lower temperatures, as mentioned above.

VIII. Conclusion

The heat treatment and calibration process for INL's HTIR-TCs was optimized and presented herein. A method for heat treating various lengths of thermocouple wire was shown by segmenting the heat treatment into parts. The calibration process was shown to be reliable.

A fifth order calibration curve fit was applied to the calibration data by reducing the standard error of the curve fit by as much as was reasonable. This showed a curve with fairly good sensitivity when compared to other industry standards. A means for correcting the EMF output from the TC due to a fluctuating reference temperature was discussed. By applying a linear fit to the EMF output of various reference temperatures (while holding the hot end of the TC constant), the TC calibration can be adjusted accordingly by adding the output from the linear fit to the measured TC voltage -- regardless of the temperature being measured by the TC. Finally, schematic drawings of EMF vs. length of the TC were presented. The error due to heat treating an insufficient length of the TC wire was shown to affect a calibration that is only performed within the heat treated portion of the TC.

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Table 1. Comparison between errors associated with and without using the cold junction compensation method described in the present work for High Temperature Irradiation Resistant thermocouples that do not use ice point cells at the cold junction while the calibration procedure did.

	Thermocouple Temperature Error [°C]	
Nominal Temperature [°C]	Without Ice Point Cell	Without Ice Point Cell with Cold Junction Correction
95	-17.72	-0.25
700	-6.17	1.33
800	-5.50	2.29
900	-5.71	2.32
1000	-6.35	2.12
1100	-7.10	2.28
1200	-7.87	3.12
1300	-7.83	5.48

Figure 1. Schematic diagram of High Temperature Irradiation Resistant thermocouple circuitry.

Figure 2. High Temperature Irradiation Resistant thermocouple voltage as a function of cold junction temperature variation.

Figure 3. Example of the Graphical User Interface from the calibration of High Temperature Irradiation Resistant thermocouples.

Figure 4. Typical outcome of High Temperature Irradiation Resistant thermocouples calibration curve compared to industry standards.

Figure 5. Sum squared error performance as polynomial order of curve fit increases for the calibration of High Temperature Irradiation Resistant thermocouples.

Figure 6. Individual calibration curves for the thermocouple materials versus platinum. Solid lines represent, “as manufactured,” wire. Dashed lines represent heat treated wire (specifically for molybdenum and niobium). Individual Type N electromagnetic force readings vs. platinum from Burley et al. [12].

Figure 7. Simple temperature gradient acting entirely within state B wire of the High Temperature Irradiation Resistant thermocouple. (1) Molybdenum wire at the beginning of the circuit at room temperature, (2) molybdenum wire at room temperature just prior to a temperature increase (e.g. furnace), (3) thermocouple junction between molybdenum and niobium at an elevated temperature, (4) niobium wire at room temperature just after temperature decrease, and (5) niobium wire at the completed circuit at room temperature.

Figure 8. Simple temperature gradient acting entirely within state B wire of the High Temperature Irradiation Resistant thermocouple with reference temperature (i.e. ice-point cell). (1) Molybdenum wire at the beginning of the circuit at room temperature, (2)

molybdenum wire at constant reference temperature (e.g. ice point cell) (3) molybdenum wire at room temperature just prior to a temperature increase (e.g. furnace), (4) thermocouple junction between molybdenum and niobium at an elevated temperature, (5) niobium wire at room temperature just after temperature decrease, (6) niobium wire at constant reference temperature, and (7) niobium wire at the completed circuit at room temperature.

Figure 9. Schematic of temperature gradient extending beyond the transition zone between state A and state B wire of the High Temperature Irradiation Resistant thermocouple. The temperature gradient causes the state A wire to increase the overall EMF generation. (1) Molybdenum wire at the beginning of the circuit at room temperature, (2) molybdenum wire at constant reference temperature (e.g. ice point cell) (3) molybdenum wire at room temperature just prior to a temperature increase (e.g. furnace), (4) transition from state A to state B molybdenum wire inside the temperature increase (5) thermocouple junction between molybdenum and niobium at an elevated temperature, (6) transition from state B to state A niobium wire inside the temperature decrease (7) niobium wire at room temperature just after temperature decrease, (8) niobium wire at constant reference temperature, and (9) niobium wire at the completed circuit at room temperature.

Figure 10. Thermocouple temperature reading offset from allowing the temperature gradient to extend into the as-manufactured state A wire, as seen in the schematic of Figure 9. Three (3) temperatures (i.e. 20°C [+], 250°C [x], and 700°C [o]) were recorded by the tip of the thermocouple and the transition between state A wire and state B wire (A|B) was heated to ~65°C.