INL/CON-06-11875
PREPRINT

Initial Results from Investigations to Enhance the Performance of High Temperature Irradiation-Resistant Thermocouples

15th International Conference on Nuclear Engineering (ICONE 15)

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April 2007

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

Idaho National Laboratory

15th International Conference on Nuclear Engineering
Nagoya, Japan, April 22-26, 2007
ICONE15-10842

INITIAL RESULTS FROM INVESTIGATIONS TO ENHANCE THE PERFORMANCE OF HIGH TEMPERATURE IRRADIATION-RESISTANT THERMOCOUPLES

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1. INTRODUCTION

Several options have been identified that could further enhance the lifetime and reliability of INL-developed thermocouples for in-pile testing, allowing their use in higher temperature applications (up to at least 1700°C (3092°F)). A joint University of Idaho (UI) and INL University Nuclear Energy Research Initiative (UNERI) is underway to investigate these options and, ultimately, provide recommendations for an enhanced thermocouple design. This paper presents preliminary results from this UI/INL effort. Results are reported from tests completed to evaluate the ductility, temperature resolution, transient response, and stability of thermocouples made from non-commercially available alloys of molybdenum and niobium. In addition, this paper reports preliminary insights gained by comparing the performance of thermocouples fabricated with various diameters.

2. Experimental Methods

Ductility tests were performed on candidate materials, listed in Table I.

Table I: Molybdenum and Niobium Alloys Evaluated

Designator	Description
+ wire	
KW-Mo	Molybdenum doped with W, K, and Si
Doped Mo	Molybdenum doped with LaO
Mo-1.6% Nb	Molybdenum-1.6% Niobium alloy
Mo-3% Nb	Molybdenum-3% Niobium alloy
- wire	
Nb-1Zr	Niobium-1% Zirconium alloy
Nb-4Mo	Niobium-4% Molybdenum alloy
Nb-6Mo	Niobium-6% Molybdenum alloy
Nb-8Mo	Niobium-8% Molybdenum alloy

Samples of 0.254 mm (0.010") diameter wire were tested for ductility after being exposed to high temperatures (1400, 1600, and 1800°C (2552, 2912, 3272°F)) for various durations (2, 5, and 12 hours). Wire ductility was then tested by wrapping the samples tightly around mandrels of 2, 5, 10, and 20 times the wire diameter.

Calibration tests were performed on prototype thermocouples fabricated from the candidate wires in order to compare their temperature resolution. Three types of thermocouples were fabricated for testing: as received bare wire (ARBW), as received swaged (ARS), and swaged then heat treated (SHT).

Prototype thermocouples were also fabricated from KW-Mo and Nb-1Zr thermo-element wires with diameters of 0.127 mm (0.005"), 0.254 mm (0.010"), and 0.508 mm (0.020") to test the effect of wire diameter on thermoelectric response.

3. Results

Ductility testing seems to indicate that the ODS-Mo and KW-Mo samples retain suitable ductility after all tested temperatures and heat times. However, the Mo-1.6%Nb samples became brittle after heating for 5 and 12 hours at 1800°C; and the Mo-3%Nb samples became brittle after 12 hours at 1600°C (2912°F) and for heating durations at 1800°C (3272°F).

Evaluations indicate that doped molybdenum alloys, either ODS molybdenum or KW-Mo, retain ductility better than the two non-commercial, molybdenum niobium alloys (Mo1.6% Nb and Mo3%Nb) evaluated. Thermocouples containing doped molybdenum were also observed to have better high temperature resolution.

Initial evaluations indicate that molybdenum-niobium alloy thermocouples containing larger diameter thermoelement wires are even more stable than thermocouples containing 0.254 mm (0.010") diameter thermoelement wires. Several difficulties were experienced in initial efforts to develop thermocouples containing smaller diameter thermoelements (0.127 mm (0.005")). Efforts are underway to improve the fabrication process for these miniature thermocouples so that they will be more robust.

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Keywords: Thermocouples, Radiation, High Temperature, In Pile Measurements

ABSTRACT

Several options have been identified that could further enhance the lifetime and reliability of INL-developed thermocouples for in-pile testing, allowing their use in higher temperature applications (up to at least 1700°C (3092°F)). A joint University of Idaho (UI) and INL University Nuclear Energy Research Initiative (UNERI) is underway to investigate these options and, ultimately, provide recommendations for an enhanced thermocouple design. This paper presents preliminary results from this UI/INL effort. Results are reported from tests completed to evaluate the ductility, temperature resolution, transient response, and stability of thermocouples made from non-commercially available alloys of molybdenum and niobium. In addition, this paper reports preliminary insights gained by comparing the performance of thermocouples fabricated with various diameters.

1. INTRODUCTION

New fuel, cladding, and structural materials offer the potential for safer and more economic energy from existing reactor and advanced nuclear reactor designs. However, insufficient data are available to characterize these materials in high temperature, radiation conditions. To evaluate candidate material performance, robust instrumentation is needed that can survive these conditions. However, standard thermocouples either drift due to degradation at high temperatures [above 1100°C (2012°F)] or due to transmutation of thermocouple components. Thermocouples are needed which can withstand both high temperature and high radiation environments.

To address this need, the Idaho National Laboratory (INL) recently developed a design and evaluated the performance of a high temperature radiation-resistant thermocouple that contains commercially-available alloys of molybdenum and niobium (Rempe, *et al.*, 2006). Candidate thermocouple component materials were first identified based on their ability to withstand high temperature and radiation. Then, components were selected based on data obtained from materials interaction tests, ductility investigations, and resolution evaluations. Results from long duration (over 4000 hours) tests at high temperatures (up to 1400°C (2552°F)) and thermal cycling tests demonstrate the stability and reliability of the INL-developed design (typically, less than 2% drift was observed). Tests in INL's Advanced Test Reactor (ATR) are underway to demonstrate the in-pile performance of these thermocouples.

Although the currently-pursued INL thermocouple design appears promising, the literature suggests that there are several options that have the potential to enhance thermoelement ductility and the temperature resolution of the INL-proposed thermocouple. Hence, a joint University of Idaho (UI) and INL project was initiated to extend INL efforts by evaluating three options: alternate materials that aren't commercially available, alternate thermocouple diameters, and alternate fabrication techniques. Initial results from these evaluations are reported in this paper.

2. BACKGROUND

When INL initiated this thermocouple development effort, various types of instrumentation that might be employed for in-pile, high temperature applications were reviewed (Rempe and Wilkins, 2005). For temperatures above 1100°C (2012°F), specialized thermocouples were deemed to be the simplest and most economic approach for in-pile high temperature measurements. Table I lists commercially-available materials initially considered for thermocouple components based on their high temperature thermal properties, nuclear properties, and cost.

Table I: Candidate Thermocouple Component Materials

Component	Candidate Materials
Thermoelements	Molybdenum,* Zircaloy-4, Titanium-45% Niobium, Niobium-1%Zirconium
Insulators	Aluminum Oxide, Hafnium Oxide, Magnesium Oxide
Sheaths	Titanium, Zircaloy-4, Niobium-1%Zirconium

*Evaluations considered several types of Molybdenum: undoped Mo, Mo-1.6% Nb, KW-Mo (doped with tungsten, silicon and potassium), and ODS-Mo (containing lanthanum oxide).

Materials interaction tests were completed by heating representative thermocouple samples in gettered argon at 1300 and 1600°C (2372 and 2912°F). As shown in Figure 1(a), 1300 °C (2372°F) tests indicated significant materials interactions occurred with samples containing Zr-4 thermoelements, Al_2O_3 insulators, and Zr-4 sheaths. However, 1600°C (2912°F) results for Nb-1%Zr and Mo thermoelement wires and Nb-1%Zr sheaths indicate that no discernible materials interactions occurred between these materials and HfO_2 insulators (see Figure 1(b)).

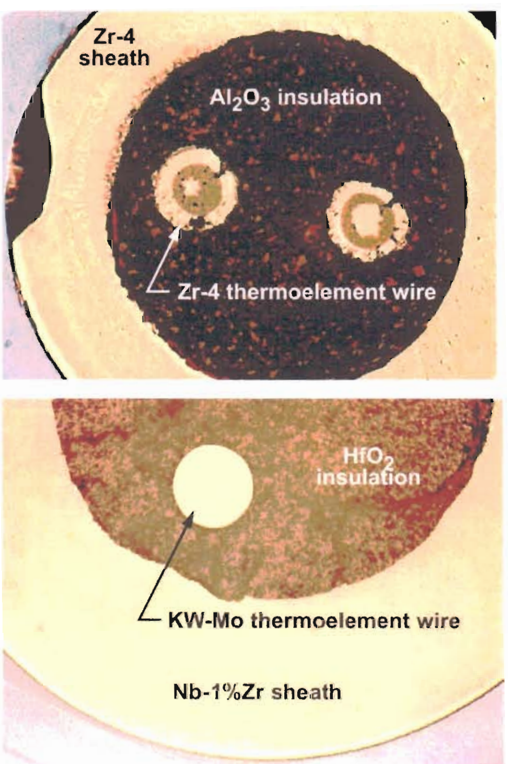
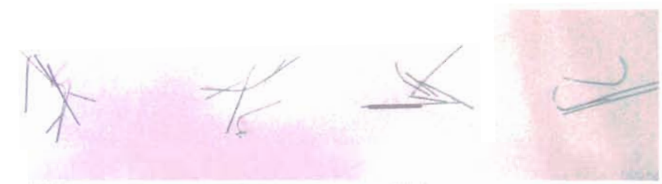
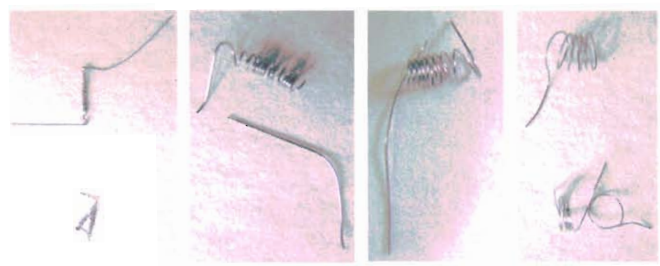


Fig. 1. Materials interaction test results (wire-insulator-sheath) using representative thermocouple samples.

Mandrel-wrap tests on wires exposed to temperatures up to 1600°C (2912°F) provided insights about thermoelement embrittlement. Wire samples from each of the thermoelement materials listed in Table I were wrapped on mandrels of two, five, ten, and twenty times the wire diameter. Those metals that wrap without damage on a small-diameter mandrel after high-temperature exposure are better candidates from the standpoint of embrittlement. Most Table I thermocouple wire materials exhibited suitable ductility. The one exception, undoped Mo wire, recrystallized at 1200°C (2192°F). As illustrated in Figure 2(a), this wire was brittle after heating at 1300°C (2372°F). However, other tested Mo wires (e.g., KW-Mo, ODS-Mo, and Mo-1.6%Nb) remained ductile even after heating at 1600°C (2912°F) (see Figure 2(b)).



(a) Un-doped Mo wire after heating at 1300 °C (2372°F)



(b) Doped Mo-KW wire after heating at 1600 °C(2912°F)

Fig. 2. Ductility test results (0. 254 mm (0.010") diameter wire).

Calibration tests were also completed for candidate thermocouple combinations. Results (see Figure 3) indicate that the thermoelectric response is single-valued and repeatable for the candidate thermoelements considered. In addition, results indicate that the high temperature resolution is acceptable for all thermocouple element combinations considered [although some combinations are limited due to materials interactions at temperatures below 1600°C (2912°F)].

Several candidate low neutron cross-section thermocouple component materials experienced minimal interactions and remained ductile at high temperatures. Tests indicated that the thermoelectric response for several candidate thermoelement combinations is single-valued and repeatable with acceptable resolution. The selection of thermocouple materials will depend on the desired peak temperature and accuracy requirements. If thermocouples are needed for temperatures of 1600°C (2912°F) or higher, the doped Mo / Nb-1%Zr and Mo-1.6% Nb / Nb-1%Zr combinations are recommended.

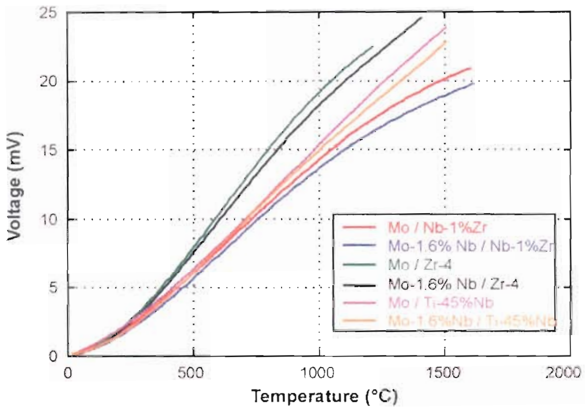


Fig. 3. Calibration curves for candidate thermocouples.

Historically, industry has employed the use of high temperature alloys (e.g, W/Re alloys and Pt/Rh alloys) rather than pure metals in thermocouples to improve their performance with respect to ductility, stability, and reliability. (Zysk and Robertson, 1974) Prior experience with Mo/Nb thermocouples (e.g.., Wilkins, 1988; Greenslade, 1992; Wilkins, 1995) suggests that similar efforts are warranted.

D. A. Prokoshkin and E. V. Vasil 'eva (1964) indicate that the addition of small amounts (less than 1%) of zirconium to niobium has been found to raise its recrystallization temperature by 25°C (45°F). The addition of molybdenum (up to 4%) may delay recrystallization by 75°C (135°F) (up to 1200 °C (2192°F)). Investigations by Schley and Metauer (1982) show that the addition of small amounts of molybdenum (less than 10%) to niobium will improve its temperature resolution.

Efforts have also been completed to improve the ductility and resolution of molybdenum. D. A. Prokoshkin and E. V. Vasil 'eva (1964) indicate that the recrystallization temperature of molybdenum is increased if it is alloyed with small amounts of niobium. To control molybdenum crystal structure during recrystallization, suitable "dopants" are added to molybdenum. In the case of molybdenum, the dopant is typically tungsten and potassium silicate. In more recent years, lanthanum oxide has been used as a dopant for molybdenum. Furthermore, investigations by Schley and Metauer (1982) suggest that the addition of small amounts of niobium (less than 5% to molybdenum) will improve its thermoelectric properties.

Table II lists molybdenum and niobium alloys being evaluated in this project. Several types of doped molybdenum, two alloys of molybdenum with small amounts of niobium, and four alloys of niobium with small amounts of molybdenum will be evaluated. Ultimately, three types of high temperature testing will be completed: (1) Ductility evaluations, (2) resolution evaluations, and (3) long duration testing with transients. However, this paper discusses results only for the molybdenum alloys because niobium alloy production has been delayed.

Table II: Molybdenum and Niobium Alloys Evaluated

Designator	Description
+ wire	
KW-Mo	Molybdenum doped with W, K, and Si
Doped Mo	Molybdenum doped with LaO
Mo-1.6% Nb	Molybdenum-1.6% Niobium alloy
Mo-3% Nb	Molybdenum-3% Niobium alloy
- wire	
Nb-1Zr	Niobium-1% Zirconium alloy
Nb-4Mo	Niobium-4% Molybdenum alloy
Nb-6Mo	Niobium-6% Molybdenum alloy
Nb-8Mo	Niobium-8% Molybdenum alloy

Initial INL efforts focused on swaged thermocouples fabricated from 0.254 mm (0.010") diameter thermo-element wires. Typically, thermocouple reliability increases with increasing thermo-element diameter. (Ludka, et al.; 1984) This paper discusses results from initial investigations of the influence of thermo-element wire diameter on Mo-Nb thermocouple performance.

3. DUCTILITY EVALUATIONS

Applying the approach initially used by INL for commercial materials (see Section 2), 0.254 mm (0.010") diameter wires for Table II candidate materials were tested for ductility after being exposed to high temperatures (1400, 1600, and 1800°C (2552, 2912, 3272°F)) for various durations (2, 5, and 12 hours). Wire ductility was then tested by wrapping the samples tightly around mandrels of 2, 5, 10, and 20 times the wire diameter.

After heating at 1400°C (2552°F) for 2, 5, and 12 hours, none of the samples show reduced ductility. All samples were capable of being wound for several turns without breaking, and are shown in Figures 4 through 6.

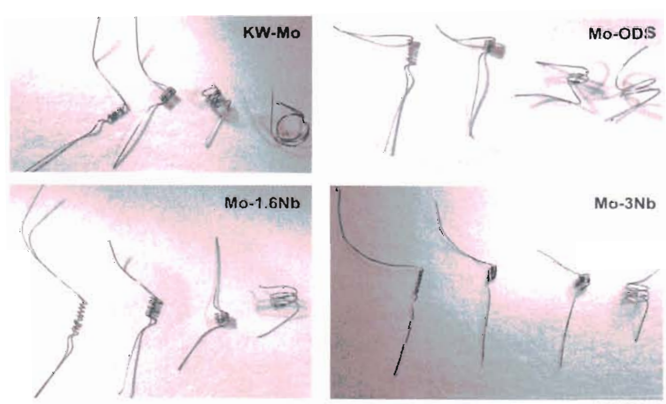


Figure 4. Samples heated for 2 hours at 1400°C (2552°F)

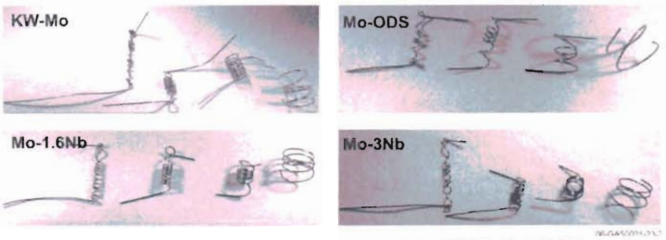


Figure 5. Samples heated for 5 hours at 1400°C (2552°F)

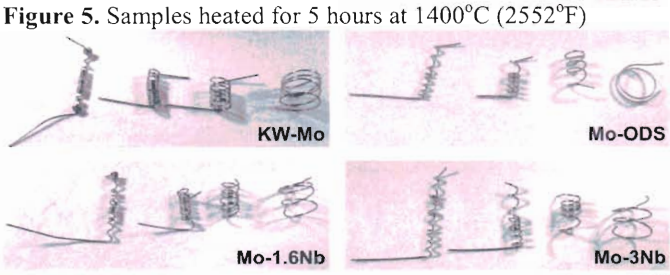


Figure 6. Samples heated for 12 hours at 1400°C (2552°F)

At 1600°C (2912°F), some samples began to embrittle (see Figures 7 through 9). The ODS-Mo remained ductile, for all mandrels and heating durations. Although a failure occurred when the KW-Mo was wrapped on the 0.508 mm (0.020") mandrel, this may have been caused by a flaw in the sample. That wire still wrapped many times without additional breakage. The Mo-1.6%Nb samples broke during wrapping on the 0.508 mm (0.020") mandrel for each heating test and on the 1.27 mm (0.050") mandrel after the 2 and 12 hour heating tests. This consistent result suggests embrittlement, rather than material flaws. However, several wraps could still be completed for each sample. The Mo-3%Nb samples remained ductile after 2 and 5 hours; but after 12 hours, the wires were brittle.

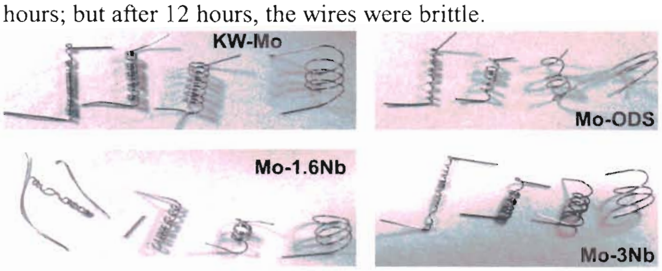


Figure 7. Samples heated for 2 hours at 1600°C (2912°F)

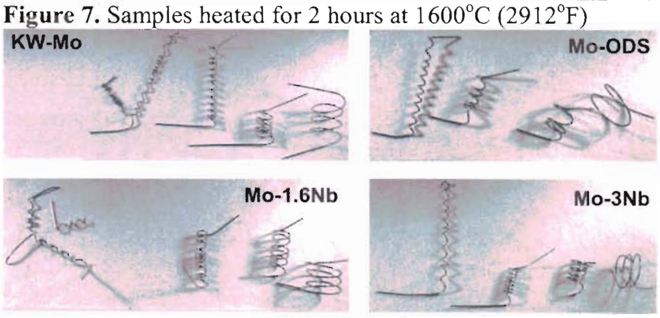


Figure 8. Samples heated for 5 hours at 1600°C (2912°F)

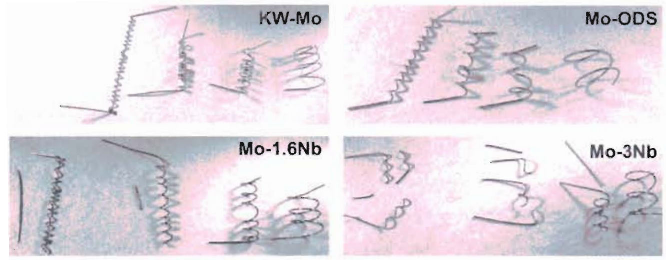


Figure 9. Samples heated for 12 hours at 1600°C (2912°F)

Results from mandrel wrap tests for wires heated at 1800°C (3272°F) are shown in Figures 10 through 12. The ODS-Mo wires remained ductile for all mandrels and durations. The KW-Mo wire experienced some breaks when being wrapped on the 0.508 mm (0.020") mandrel after 2 hours and 12 hours. However, these samples could still be wound for many turns. The Mo-1.6%Nb samples failed for each mandrel and heating duration with the exception of the 5.08 mm (0.20") mandrel after 2 hours. The Mo-3%Nb samples performed in a manner nearly identical to the Mo-1.6%Nb.

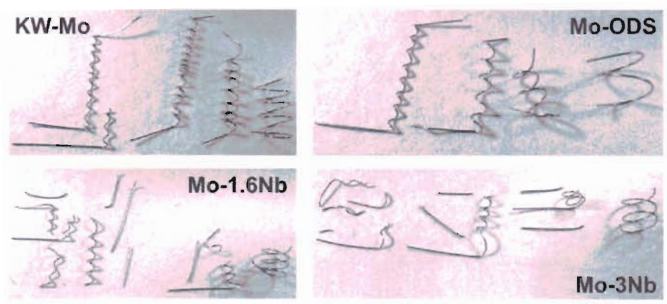


Figure 10. Samples heated for 2 hours at 1800°C (3272°F)

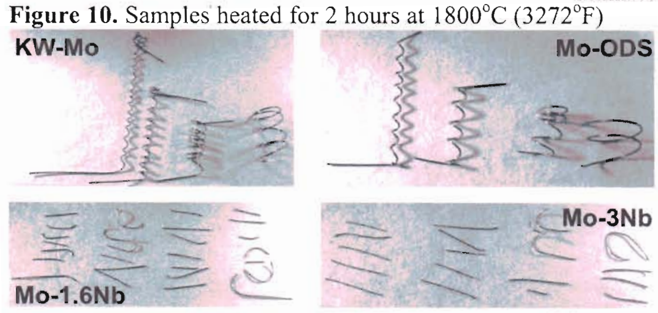


Figure 11. Samples heated for 5 hours at 1800°C (3272°F)

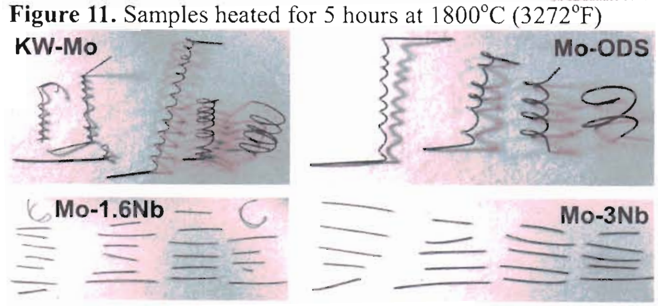


Figure 12. Samples heated for 12 hours at 1800°C (3272°F)

For all heating tests at 1400°C (2552°F), 1600°C (2912°F), and 1800°C (3272°F), the ODS-Mo and KW-Mo samples exhibited suitable ductility. However, the Mo-1.6%Nb samples became brittle after heating for 5 and 12 hours at 1800°C; and the Mo-3%Nb samples became brittle after 12 hours at 1600°C (2912°F) and for heating durations at 1800°C (3272°F).

4. RESOLUTION EVALUATIONS

Calibration tests were performed on prototype thermocouples in order to compare their temperature resolution. Three types of thermocouples were fabricated for testing: as received bare wire (ARBW), as received swaged (ARS), and swaged then heat treated (SHT). Thermocouples were fabricated using the thermoelement wire materials listed in Table II. However, the ODS-Mo was only included in the ARBW evaluations because of the limited supply available. Tests were only completed for thermocouples with a niobium/1% zirconium negative thermo-element wire because of production delays on non-commercial niobium alloys.

ARBW thermocouples were fabricated by threading hard-fired hafnia onto thermoelement wires and loading into an alumina tube, with the hot junction open to the environment. ARS thermocouples were prepared using crushable hafnia insulation and loading into a niobium/1% zirconium sheath which was then swaged and sealed. The SHT thermocouples were made in the same way as the ARS thermocouples but were heat treated for five hours at 1700°C (3092°F.) This time period was selected based on results from tests reported in Section 5.

The prototypes, with a reference thermocouple, were tested in gettered high purity argon. The furnace was programmed to increase the temperature to 1600°C (2912°F), incrementally, holding at selected temperatures while calibration data were obtained. The furnace temperature then dropped incrementally to 1000°C (1832°F), then increased incrementally to 1600°C (2912°F), then dropped back incrementally to 1000°C (1832°F). The reference junction of each thermocouple was immersed in an ice point cell, which maintains a temperature of 0°C (32°F). The voltage output of each thermocouple, and the reference Type S thermocouple data, were recorded every two seconds. This allowed for a minimum of 1000 data points at each temperature for each thermocouple after the output had stabilized. The average values at each temperature were used to generate calibration curves for each prototype.

Calibration curves produced during the first heating cycle of the ARBW calibration tests are shown in Figure 13. Data in this plot show that the ODS-Mo thermocouple has slightly greater sensitivity than the others. The plots of subsequent test cycles (not shown) reveal that the emf produced by each thermocouple decreases after the first heating and then stabilizes. The emf shift for each thermocouple was approximately equal. This loss of sensitivity is caused by grain growth in the wires, which is accelerated by elevated temperature.

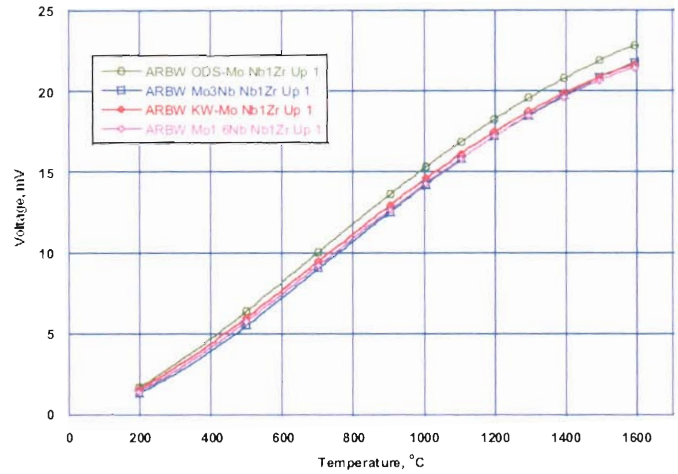


Figure 13. Calibration curves for ARBW thermocouples

Calibration curves produced for the ARS thermocouples are shown in Figure 14. These curves are similar to those of the ARBW thermocouples, except the ARS curves lose some resolution at higher temperatures. Data from subsequent cycles show that the ARS thermocouples experience a similar emf shift to the ARBW thermocouples, but to a greater degree. The thermoelectric response is stable after two cycles.

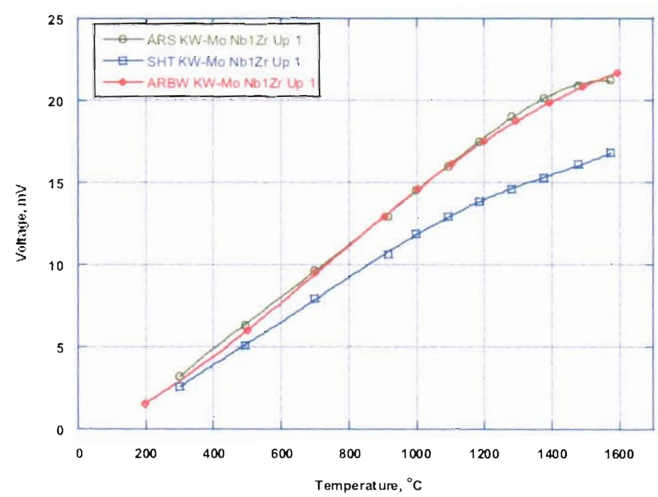


Figure 14. Calibration curves for ARS thermocouples

Figure 15 shows the calibration curves for the SHT prototypes. The Mo1.6Nb thermocouple deviates from the other thermocouples at higher temperatures; the cause of this is unclear. The resolution of the SHT is significantly reduced when compared to the ARBW and ARS prototypes. It is suspected that this reduction in resolution is due to the grain growth caused by heat treating. The calibration of all of the SHT thermocouples remained stable for all cycles.

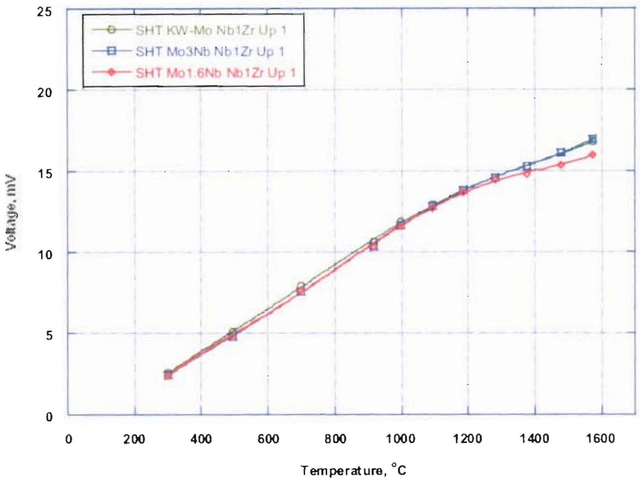


Figure 15. Calibration curves for SHT thermocouples

The Mo3Nb and Mo1.6Nb wires exhibited considerably less ductility than thermocouples with doped molybdenum. Thermocouples constructed from these materials have also been shown to have reduced resolution compared to KW-Mo and ODS-Mo prototypes. Due to lack of ODS-Mo wire, only one thermocouple was constructed from this material. However, ductility and calibration test results for this material are promising. The KW-Mo material performed similarly to the ODS-Mo, and was superior to the Mo3Nb and Mo1.6Nb, in terms of ductility and resolution. KW-Mo is therefore the recommended positive thermo-element material. Figure 16 shows the calibration curves for the three thermocouples constructed with the KW-Mo positive thermo-element wire.

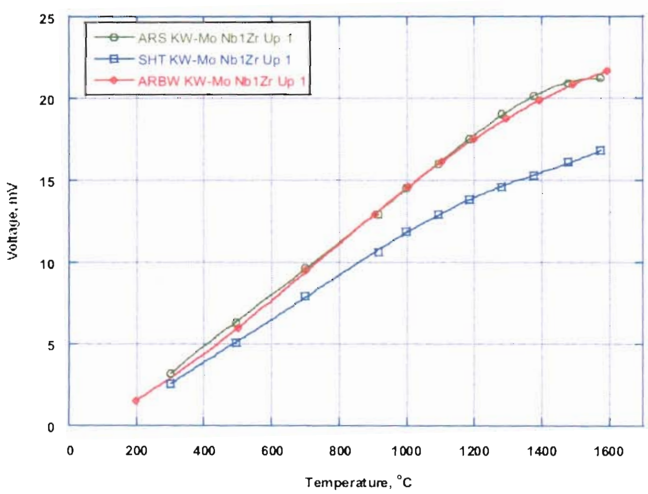


Figure 16. Calibration curves for KW-Mo thermocouples

5. SIZE VARIATION

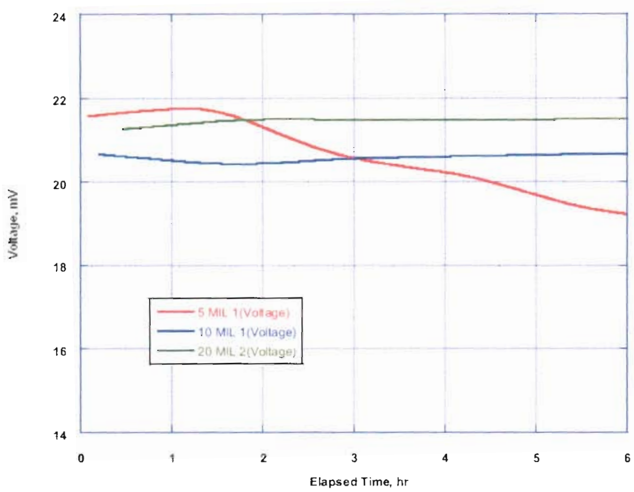
The reference INL design is a 1.5748 mm (0.062") diameter thermocouple fabricated from thermoelement wires that are initially 0.254 mm (0.010") in diameter. However, it is anticipated that some applications will require larger or smaller diameter thermocouples. This section reports results from initial evaluations to develop designs for alternate diameter thermocouples and assess the impact of size on thermocouple performance.

Prototype thermocouples were fabricated with thermo-element wires of three different diameters. Thermocouples 5 MIL 1 and 5 MIL 2 were constructed from 0.127 mm (0.005") wire, 10 MIL 1 and 10 MIL 2 from 0.254 mm 0.010" wire, and 20 MIL 1 and 20 MIL 2 from 0.508 mm (0.020") wire. Note that commercially available materials (doped KW-Mo and Nb-1%Zr) were used for this task because these materials are less expensive to obtain. For each size of thermocouple, sheath tubing and insulator materials were obtained and an appropriate process was developed for swaging reductions.

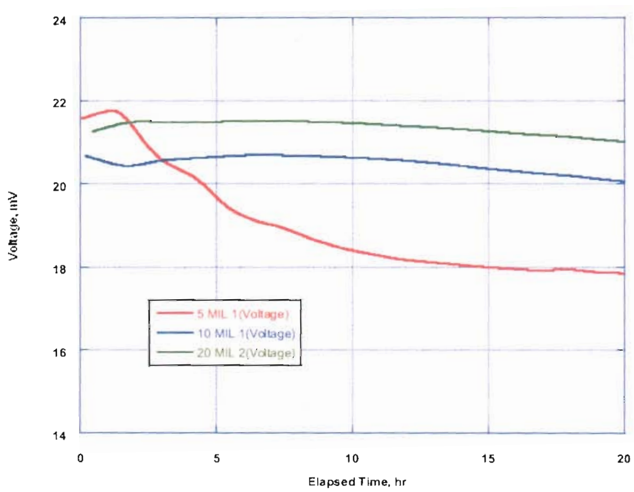
Two thermocouples of each size were prepared (e.g., 5 MIL 1 and 5 MIL 2; 10 MIL 1 and 10 MIL 2, 20 MIL 1 and 20 MIL 2). However, one of the thermocouples containing 0.127 mm (0.005") wire, 5 MIL 2, failed during fabrication. The remaining five thermocouples were heat treated for 20 hours at 1700°C (3092°F) to gain insights about appropriate time periods required for grain growth stabilization.

Representative data obtained during heat treatment are plotted in Figure 17. Thermocouples were inserted into a tube furnace at a time corresponding to 0 hours and immediately connected to the data acquisition system. As shown in Figure 17, the smaller diameter thermocouple is more susceptible to "noise" induced by the tube furnace. Figure 17a data indicate that all three thermocouples stabilized within the first 4 hours of heat treatment. In fact, the emf response suggests that a heat treatment be limited to 5 hours.

During the cool down period, the remaining smaller thermocouple (with 0.127 mm (0.005") thermoelement wires) experienced an open circuit failure. Transient tests were performed on the remaining thermocouples to examine thermocouple stability. Changes in emf were minimal (less than 1%) for the larger diameter thermocouple after two cycles. Larger drifts (up to 4%) were observed in the standard-sized thermocouples, which were fabricated with 0.254 mm (0.010") wires. Data reported in Section 4 show that a stable thermocouple response is obtained for operating temperatures of up to 1600 °C when heat treatment times of five hours at 1700 °C are selected.



(a) Response during first 6 hours of heating.



(b) Response during entire 20 hours of heating

Figure 17. Comparison of various diameter thermocouples during 1700 °C (3092°F) heat treatment.

Efforts are underway to develop a fabrication process that will yield more robust small diameter thermocouples. Then, additional samples for each thermocouple size will be prepared in which the heat treatment duration is limited to 5 hours; additional evaluations will be completed to assess the effect of diameter on thermocouple performance.

SUMMARY

Several options have been identified that could further improve the lifetime and reliability of recently INL-developed thermocouples for in-pile testing, allowing their use in higher temperature applications (up to at least 1700 °C (3092°F)). A joint UI/INL project was initiated to extend INL efforts by evaluating three options: alternate materials not commercially available, various diameters, and alternate fabrication techniques, to ultimately provide recommendations for an improved molybdenum/niobium alloy thermocouple design. Initial results from this UNERI are presented in this paper.

Evaluations indicate that doped molybdenum alloys, either ODS molybdenum or KW-Mo, retain ductility better than the two non-commercial, molybdenum niobium alloys (Mo1.6% Nb and Mo3%Nb) evaluated. Thermocouples containing doped molybdenum were also observed to have better high temperature resolution. When non-commercial niobium molybdenum alloys are received, similar evaluations will be completed.

Initial evaluations indicate that molybdenum-niobium alloy thermocouples containing larger diameter thermoelement wires are even more stable than thermocouples containing 0.254 mm (0.010") diameter thermoelement wires. Several difficulties were experienced in initial efforts to develop thermocouples containing smaller diameter thermoelements (0.127 mm (0.005")). Efforts are underway to improve the fabrication process for these miniature thermocouples so that they will be more robust.

ACKNOWLEDGMENTS

This research is funded as part of a DOE-NE University Nuclear Energy Research Initiative (Grant DE-FC07-06ID14738). INL work supported by the U.S. Department of Energy, Office of Nuclear Energy, Science, and Technology, under DOE-NE Idaho Operations Office Contract DE AC07 05ID14517.

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