Effect of Electrical Annealing to the Inhomogeneity Improvement of Type-S Thermocouples

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Abstract. The thermoelectric inhomogeneity as a function of position along wires is one the significant uncertainty of measurement using thermocouples. Here we report development of an electrical annealing system for thermoelectric inhomogeneity treatment. Two inhomogeneous type-S thermocouples, which had the inhomogeneity greater than 0.04%emf, are successfully recovered using the system. An improvement on thermocouple performance as large as 0.28 °C (at temperature of 1000 °C) can be obtained using the system. This article provides detailed information and may help the reader to obtain a quick grasp about the system.

Keywords: Uncertainty; thermocouple; Inhomogeneity treatment;

1. Background
Fast response, good stability, and very broad of measurement range, these are the reasons why thermocouples are widely used as secondary reference thermometers. Thermocouples also serve as precise and accurate contact thermometer for high-temperature applications (1-5). Despite of their versatility, thermocouples also have some limitations because of their physical condition. The Seebeck coefficient of the thermocouple wire can become non-uniform due to the cold work and the use for the elevated temperatures. Seebeck coefficient variations along thermocouple wire result thermocouple inhomogeneity (6). The attempt to use emf corrections from thermocouple inhomogeneity measurements is impractical if not impossible because the effect of inhomogeneity may be either subtractive or additive to the emf (7). By far the most convenient way is to include the thermocouple inhomogeneity as one source of uncertainty.

Thermocouple inhomogeneity is one of the dominant uncertainty sources of thermocouple thermometry, however it is sometime reversible and can be restored simply by isothermal annealing. Isothermal annealing can be done by exposing the thermocouples to a long, uniform, and fixed high-temperature furnace (furnace annealing) or by hanging the wires between two electrodes in air and passing an electric current through them (electrical annealing). Among these methods electrical annealing offers effective and direct-treatment to the thermocouple wire (7, 8).

The existing literatures on annealing of thermocouples are dominated by furnace annealing because the electrical annealing is rather difficult technique. The electrical annealing requires the then annealed thermocouple wires to be inserted into an insulator, thereby exposing the wires to further cold-work. Normally, the electrical annealing is combined with the furnace annealing to remove small mechanical strain during the mounting, thus limiting the analysis of the electrical annealing itself.

In this article, we develop alterations to the wire electrical annealing system and propose a new electrical annealing procedure. A procedure we propose is a 1000 °C Quenched State (QS*) for 90 mins followed by a 750 °C Annealed Stated (AS*) for 30 mins. Wire inhomogeneity assessments are conducted before and after electrical annealing (without furnace annealing). Two Type S thermocouples are used in the experiment. Our report aims to build on previous studies and provides new data on the recovery of thermocouple inhomogeneity by electrical annealing. This work may also be important for those trying to improve the calibration and measurement capability (CMC) of thermocouple calibration by reducing the value of thermocouple inhomogeneity (9-12).
2. Experimental Details

We develop an electrical annealing system that consists of two main components: an annealing circuit and a non-contact wire temperature measurement as shown in Fig. 1. The annealing circuit gives a flow of electric current into thermocouple wire. It is placed in an open enclosure (78 cm length by 53.5 cm width and 175 cm deep). The longest wire that may be annealed using this system is about 270 cm. The thermocouple wire is hung between two separated electrodes made of conductive copper. A voltage regulator (Volt Slider S-260-10B, Yamabishi Electric, Tokyo, Japan) controls the amount of current going to the wire. An ampere meter and a voltmeter measure the system current and voltage, respectively. The surface temperature of wire \( t_{\text{wire}} \) is determined using a variable focus radiation thermometer, linear pyrometer LP2 (Type IKE LP2, PTB, Stuttgart, Germany). The output current of LP2 is read by Pico ammeter (6485 Pico ammeter, Ohio, USA) and is recorded using a personal computer. All meters are traceable to the SI units.

Pt10Rh/Pt wires are withdrawn from two type S thermocouples (Model 5650, Fluke Hart Scientific, Utah, USA), hereafter referred to as TC-1 and TC-2. The wire length is about 260 cm, suitable for use with our system. Thermocouple inhomogeneity assessment is performed before and after electrical annealing to study effectiveness of our proposed annealing procedure. The procedure of inhomogeneity assessment is similar to that of APMP Regional Comparison of Type R (Pt-Pt13%Rh) Thermocouples from 0 to 1100°C, APMP.T-S1 (13).

The annealing procedures of TC-1 and TC-2 are similar in term of the operating temperature and the duration. Moving the slider of the regulator to increase the amount of current, the wire temperature, \( t_{\text{wire}} \), is increased from room temperature to 1000 °C at a rate not exceeding 40 °C/min. After \( t_{\text{wire}} \) reaches 1000 °C, \( t_{\text{wire}} \) at first is kept at this condition for 90 min; \( t_{\text{wire}} \) is then decreased to 750 °C and kept constant for 30 min. Afterward the passive air quenching is conducted by moving the slider to the minimum position, then the current decreases to zero and \( t_{\text{wire}} \) cools down to room temperature. The cooling rate is thus only determined by natural convection between the wire and the air. The wire is removed from the electrodes after \( t_{\text{wire}} \) reaches room temperature.

3. Results and Discussion

A prolonged use of thermocouple at high-temperatures has been reported (6, 14, 15) to be a major cause thermocouple inhomogeneity. It is also observed in our heavily-used reference standards. TC-1 had been used for more than 1000 hours in a horizontal furnace (Model 9112B, Fluke Hart Scientific, Utah, USA) with installed thermal block (406 mm of immersion depth), to measure temperatures up to 1100 °C. Initial scan of thermoelectric signature as exemplified by Fig. 2 (black dash line) clearly shows that TC-1 has become inhomogeneous. At an immersion depth of about 328 mm to 376 mm, the initial scan of TC-1 exhibits a negative change in emf, dropping by about -0.3 μV. On the other hand, at immersion depths of about 168 mm to 328 mm, the initial scan of TC-1 shows positive drift of the emfs with the magnitude as high as 0.7 µV. The initial scan further shows that the inhomogeneity of TC-1, before the annealing, is found to be within ± 0.042 % (±0.35 °C, given that the type S sensitivity is 11.539 μV•°C\(^{-1}\) at 1000 °C), measured from 160 mm to 400 mm.
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200
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160
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160

Fig. 2 Inhomogeneity scanning graph of TC-1.

TC-2 had also been used for more than 1000 hours at a temperature range up to 1100 °C; however, it subjects to many different immersions depths because it was used in various calibration media such as horizontal furnace (the same furnace used in TC-1), dry-block (Model 9141, Fluke Hart Scientific, Utah, USA), and salt-bath (Model 6055, Fluke Hart Scientific, Utah, USA). Initial scan of the thermoelectric signature (between tip and 400 mm) as shown in Fig. 3 (black dash line) indicates that TC-2 has a significant region of inhomogeneity about the tip. This deviation in the emf is far greater than observed in TC-1, and most likely caused by the different immersion depth. The inhomogeneity of TC-2, before the annealing, is calculated to be within ± 0.046 % (±0.38 °C at 1000 °C), measured from 160 mm to 400 mm of immersion depth.

The reduction of emfs in the thermoelectric scans indicates the reduction of Seebeck coefficient along those regions. The oxidation of Pt alloy has been strongly attributed to this behaviour. When a type S thermocouple is used at a temperature range of 500 °C to 900 °C, oxidation of the Rh in the Pt10Rh leg should lead to the presence of rhodium oxide layer. If the temperature is held within this range, then it can cause the depletion of Rh and the growth of an oxide layer (16, 17). As Seebeck coefficient is proportional to the Rh concentration, the Seebeck coefficient also decreases along the depleted region which results in a reduction in emf. The increase in emf, seen in the first 250 mm of TC2, after annealing (Fig. 3) suggest rhodium oxide has been dissociated in this region and converted back to metallic rhodium. This process has been previous reported on (18).

Many of the emf changes in TC-1 and TC-2 were shown to be by the combination of rhodium oxide, vacancies, and defects, which were removed by appropriate annealing (solid blue lines in Figs. 2 and 3). TC-2 suffers the reduction of emf by approximately -0.08% emf measured at the immersion depth of 400 mm before and after the electrical annealing. Pt and Pt10%Rh are annealed together which may cause the formation and liberation of rhodium oxide from the Pt10%Rh leg to the Pt leg. However the electrical annealing appear to show higher restoration effect on the Seebeck-coefficient uniformity than the segregation drift of the Seebeck-coefficient caused by the liberation of rhodium oxide.

The final inhomogeneity of TC-1 is calculated to be within ±0.008% (about ±0.07 °C at 1000 °C). The value is about five times less than its initial value and signifies only a small inhomogeneity component, with more typical values being about ± 0.02% (19). The final inhomogeneity of TC-2 is found to be ±0.036% (about ±0.30 °C at 1000 °C). The value seems statistically not significant but if we take a look at the scan (Fig. 3), there is significant increase in Seebeck coefficient at a range of immersion 168 mm to 360 mm. The increasing in Seebeck coefficient presumably because the annealing at 1000 °C results grain growths, rhodium oxide removal, and reduction in defect concentrations.

Fig. 3 Inhomogeneity scanning graph of TC-2.
4. Conclusions
In summary, our annealing procedure recovers the inhomogeneities of Type S thermocouples. Significant improvements were seen in two tested thermocouples. The origin of improvements can be explained using existing theories about oxidation in Pt-Rh based alloys. We also suggest that by using a 750 °C anneal, the required vacancy annealing time can be reduced from 24 hours at 450 °C (20) to half an hour, while not exposing the Pt10%Rh leg to substantial growth of rhodium oxide. The system, procedure, and mechanism aid understanding of electrical annealing processes.

Acknowledgments
This work is supported by Ministry of Research, Technology and Higher Education of the Republic of Indonesia through the scheme of Incentive Research Program for the National Innovation System (04/INS-2/PPK/E/E4/2017). It is a pleasure to acknowledge the technical supports provided by A. Hermana and Effendi, and insights gained while discussing about the system with Y.G. Kim, S.N. Park.

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