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Investigation of Temperature Drift with Thermocyclic Oxidation on K-Type Bare Thermocouples

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Abstract: The irrevocable variations in the thermoelectric properties arise in normally used bare type-K thermocouples that are subjected to high temperatures. Over an extended period of time, these long-term modifications may manifest themselves as a systematic shift in the thermocouple's characteristics. This paper investigates the temporal instability of thermocouple output at temperatures over 800 °C. This investigation focuses on the change in temperature output that occurs in thermocouples of varying diameters after they have been subjected to high temperatures for more than 365 hours. This data was used to assess the relationship between the amount of temperature drift with different lead diameters and the degradation of the material compositions of thermocouple thermoelements. It is critical to determine the pace at which the thermocouple material degrades over time. This paper concentrates in detail on the drift analysis of thermocouples of different diameters which were tested at high temperatures with their segmental material compositions. Within the scope of this investigation, the elemental analysis of the thermocouple could be carried out using scanning electron microscopy in conjunction with an energy dispersive X-ray (SEM-EDAX) both before and after the thermocouple had degraded. The results show that the thermoelectric response decreases dramatically and the highest drift reaches to about 196.4 μV for (Ø0.51 mm), about 118.6 μV for (Ø1.29 mm) and 79.2 μV for (Ø2.05 mm) thermocouples, respectively at 800 °C after prolonged usage of 365 hours. The highest thermal drift was achieved with [90-10] vol% Ni-Cr / [95-05] vol % Ni-Al, Mn, Si composite thermocouples, which related to the increased amount of the 2-3 oxide layers that were formed in the composite of bare thermoelements.

Keywords: drift analysis; EDAX; seebeck coefficient; SEM; thermocouple

1 INTRODUCTION

Thermoelectric sensors, often known as thermocouples, are one of the most prevalent forms of temperature sensors used in industries due to its sturdy structure and ability to tolerate high temperatures and tough environments. Though thermocouples are a common form of temperature sensor, there are still effects that occur in them under various settings that need to be investigated more. At higher temperatures, temperature readings are more prone to inaccuracy, resulting in a drift [1] in the thermocouple output. The power plant industry is one of the most severely affected by thermocouple oxidation and erosion issues. Oxidation and corrosion caused by coal combustion are connected to certain contaminants in the fuels that cause the creation of non-protective scales or may disturb the typically protective oxide scales that are present in the fuel [2]. Generally, external oxidation issues [3] in a thermal power plant occur in three broad locations: i) near the fire zone in the water wall or boiler tubes, ii) in the superheater and reheater tubes, and iii) in the duct work that transports the combustion flue gases. To understand the behavior under boundary constraints, this testing and study method are required.

To properly manage the impacts of measurement errors, it is essential to have a solid understanding of the many factors that might play a role in causing them, as well as the solutions that are at one's disposal for reducing or removing them. Among the causes of errors are: i) the initial deviation of the conversion characteristics caused by inhomogeneity in the thermocouple [4], ii) the degradation of the thermocouple under the influence of high temperature and long operation time [5], and iii) the error caused by the cold junction effect in the thermocouple. The oxidation of thermocouple material [6] is caused by the overexposure of thermocouple material to high temperatures for an extended length of time, as well as the presence of particular contaminants in the thermocouple material.

Experimentally calculated thermocouple error with controlled temperature profile by [7] was compared to theoretical estimates by [8]. Neural network approaches were used to repair errors caused by inhomogeneity of thermocouple electrodes over a long experiment. At a 5% significance level, the error is 0.46 °C. [9] used a thermal model to compute the exponentially decreasing thermal drift of a single junction thermal converter based on a thermocouple's cold side temperature. [10] proved that the reported thermal voltage is governed by the temperature of the thermocouple along its whole length. This is the case even if the temperatures at the hot and cool junctions are different. In their study, [11] showed that thermocouple inhomogeneity acquired during operating conditions under the influence of time and higher heating rate affects the thermoelectric output up to 10 °C for K-type thermocouples, and that the length of the thermocouple wire, heat flux inside the furnace, and length of insertion all play a significant role in determining the output voltage. Voltage output is an important indicator of thermocouple accuracy and durability [12]. This shift in stability is caused by changing the material constant Seebeck coefficient. This coefficient has an impact on temperature-dependent voltage. Mixing materials results in unique designs. This constant influences the voltage output temperature of the thermocouple. These modifications may have an impact on the quality of life, longevity, and safety [13]. Physical and chemical changes in the material can induce variations in the thermocouple lead. This material constant might change, either momentarily or permanently. Over 600 °C, annealing and hysteresis cause thermocouple reversible changes [14]. When thermocouples are exposed to high temperatures without being protected by a sheathing, the voltage output rises. Positive drift occurs only at temperatures between 600 °C and 900 °C when utilizing nickel-based thermocouples with metal sheathing and mineral insulation [15]. As the temperature increases, the voltage reduces slowly and considerably. This behavior has been described in depth by [16], with the same conclusions being reached for thermocouples of type N and K by [17].

A number of recent articles have shown how particle movement between thermocouple thermoelements, mineral separation material, and metal sheath characterises the drift process. According to [18] and [19], manganese (Mn) is the most significant source of pollution since it is the primary element that produces drift in the measurements. Despite the fact that this element is present in the thermocouple's sheathing material, it contaminates the thermocouple thermoelements at temperatures more than 1100 °C, affecting the Seebeck coefficient [20]. Temperature decrees of type N and K MIMS thermocouples with an external width of 3 mm are shown in publications [21]. At 1100 °C, the recorded temperature variance from the initial state was 10 °C, and at 1200 °C, the recorded temperature variance from the original condition was 24 °C. These results were obtained after an experimental session that lasted 1000 hours. In thermocouples wires, temperature between 600 °C and 1000 °C cause permanent alterations. Pollution depletes cell membrane electromotive power (EMF) which is demonstrated for the Type N thermocouple where the Seebeck coefficients were permanently altered [22]. One thermocouple branch uses a metal alloy with 14.4% chromium, 1.4% silicon, and 0.1% magnesium. The other branch uses a metal alloy with 95.6% nickel, 4.4% silicon, and 0.1% magnesium. The coated steel substrates were investigated [23] using XRD, SEM-EDAX, and other techniques. Cr, Ni, and their oxides may prevent oxidising species from penetrating coated surfaces. [24] examined the surface layer generated on uncontaminated titanium after plasma electrolytic oxidation (XPS). This research examined the degeneration of thermocouple material using SEM-EDAX analyses method, induced by continuous contact to high temperatures, which causes surface oxidation. SEM is an effective technology for investigating a material's surface and cross-section and EDAX provides SEM's chemical components [25]. Several studies discuss thermocouple drift and temporal stabilities [26], with one study stating that a large degree of long-term stability of chromel-alumel based thermocouples is a problem that warrants more investigation.

One of the most difficult issues as previously stated that each kind of thermocouple has is maintaining the voltage output's stability over time. This study will use chemically induced adjustments to investigate a permanent change in the Seebeck coefficient. These irrevocable variations occur in nickel-based thermocouples when exposed to temperatures over 800 °C. In this work, we are concerned with the drift that happens in bare Type K thermocouples exposed to temperatures of 800 °C for more than 365 hours.

2 MATERIALS AND METHODS

2.1 Thermocouple Drift Analysis

Measurements were made on type K thermocouples with bare configuration to determine the relationship between drift voltage and the diameter of the thermocouple [27]. There were three thermocouples with a length of 28 cm examined, each of which belonged to the class 3 with ±2.5 °C accuracy available for the [28] thermocouple. The outer dimensions, as well as the corresponding AWG standard specifications, are shown in Tab. 1.

Table 1 AWG Specification and Outer diameters of tested type K thermocouples

Diameter of thermocouple with AWG standard	Outer diameter (*Ø*) of Type K thermocouples / mm	Resistance per unit length / mΩ/m
12	2.053	5.211
16	1.291	13.17
24	0.511	84.22

In materials, the Seebeck coefficient is a structural characteristic that governs the voltage produced within a material as a result of heat conduction. This property of a material may be determined by examining the slope of the thermoelectric voltage (E) with a temperature difference ($\delta T = T_1 - T_2$) graph for that material. Thermocouple sensors [29] work by comparing the Seebeck coefficients of two electrically dissimilar materials to determine temperature (S_A and S_B). The thermoelectric voltage generated is indicated by the change between their Seebeck coefficients $S_{AB} = S_A - S_B$, which is represented by:

$$E = \int_{T_1}^{T_2} S_{AB} \times dT = \int_{T_1}^{T_2} S_A - S_B \times dT \tag{1}$$

$$E = \lim_{\delta T \to \infty} \frac{\delta V_{AB}}{\delta T} \tag{2}$$

where δV_{AB} is the electric potential across the interface of two dissimilar conductors with a thermal gradient, δT.

The Fluke temperature furnace (Model No. 9150) having a range of 150-1200 °C with well's diameter of 1.25 inches and a depth of 5.5 inches is used. A Rhodium thermocouple was used to test the furnace's temperature stability and uniformity. The furnace temperature was steady between 0.1 °C to 2 °C for 365 hours. Aluminum oxide Dry Blocks provide a stable and exact temperature for thermocouples. The Thermocouple Furnace's microprocessor-based controller makes its set-point steady and accurate. During the drift test, the controller monitors the thermocouple junction. The furnace is set to warm up for 10 minutes after reaching 800 °C before stability.

Furthermore, Thermocouples in dry-temperature wells have a hole-to-hole consistency of 0.05 °C. Inaccuracies in industrial temperature sensors are common. Temperature stability was measured using the same type R thermocouple. A temperature discrepancy of 0.06 °C was discovered during a 5-hour stability test. A calibrated type R thermocouple validated the temperature stability of the furnace as an added precaution. All early research, testing, and operation took place at 800 °C. The temperature stability of the reference point impacts the thermocouple's voltage output, making drift detection more difficult. A dry block cell with long-term stability of 0.010 °C was utilised as the reference point.

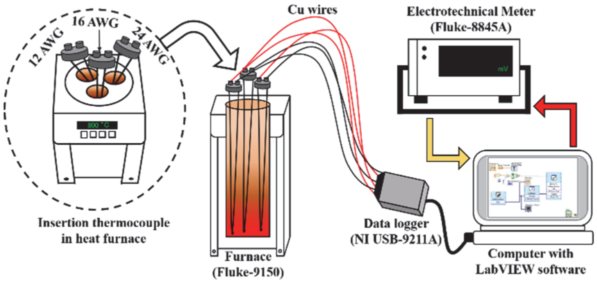
Table 2 The experiment's uncertainty budget with $k = 2$

Uncertainty source	Uncertainty values / °C
Furnace stability	0.5
Type K thermocouple	1.1
Reference point stability	0.006
Electro-technical meter	0.0042

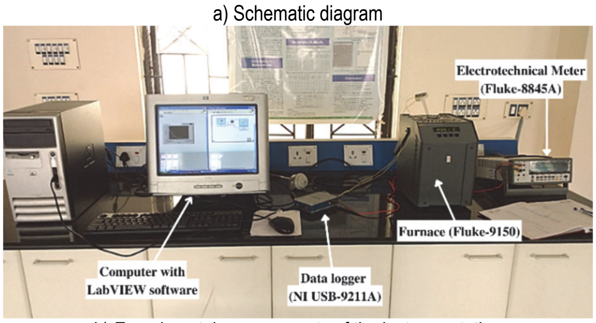
Tab. 2 illustrates that the experiment's uncertainty budget takes into account the uncertainties of each component. The uncertainty figures for each source are indicated in °C [30]. The total uncertainty value is calculated as 1.208 °C with $k = 2$ using RSS Method.

2.2 Thermocouple Inhomogeneity Test

An automated recording system is used, which featured a Data logger (NI USB- 9211A), an instrument for electro-technical measurement (Model No.8845A) and a computer equipped with recording software; the information provided was recorded as shown in Fig. 1.



a) Schematic diagram



b) Experimental arrangements of the instrumentation

Figure 1 Schematic diagram and experimental arrangements of the instrumentation

It was decided that the recording interval for all voltage output sensors should be one minute. The equivalence of the data from each sensor was ensured by employing this quick and automated recording since the data recording times were almost identical. There are some critical outliers in the collected data, which has been minimized by the use of elementary statistics method, for the statistical rejection of outlying data the critical values of Dixon's "Q" parameter and related subrange ratios are considered, at the 95% confidence level [31]. Following this initial filtering, an average value is calculated for each five-hour period, which is used to aid in the evaluation of the extent of drift in the system.

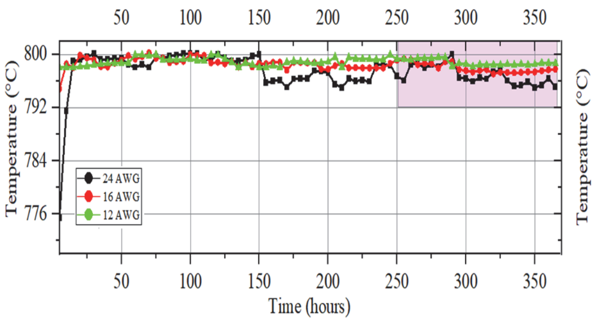


Figure 2a Temperature deviations of three different diameter thermocouples (12 AWG, 16 AWG & 24 AWG)

Results of measurements made at a temperature of 800 °C are shown in Fig. 2, while results of measurements performed at a temperature of 800 °C are shown in Fig. 3. In this graph, we can see the temperature difference of type K thermocouples with varied diameters in relation to their beginning voltage value.

The smallest thermoelement diameters that have the biggest voltage decreases are shown clearly as voltage output in Fig. 2.

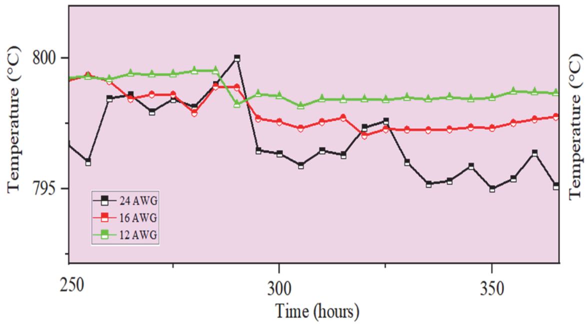


Figure 2b Thermocouples detailed drift characteristics after 250 hours of prolonged usage at 800 °C

An outer diameter of Ø0.5 mm thermocouple demonstrates this maximum degree of decrease. At the beginning of the experiment with a temperature of 800 °C, the recorded data exhibited identical values. Following a duration of 250 hours at a temperature of 800 °C, the discrepancy in temperature decreases to 5 °C from the initial state. Subsequently, after a period of 350 hours at the same temperature of 800 °C, the temperature disparity reached 25 °C. As you can see in Tab. 3, various diameters of thermocouples have varied maximum levels of decrease. Also, as observed, the rate of drift varies with temperatures. There was an earlier drift as observed in Fig. 2a and Fig. 2b, as a consequence of the increased temperature. The findings show that nickel-based thermocouples lose voltage over time when exposed to high temperatures. These findings also demonstrate a connection between voltage output levels and the diameter size of thermocouple thermoelements [32, 33].

Table 3 Maximum temperature difference from the initial state for various thermoelement diameters at 800 °C

Thermo element wire diameter Ø / mm	Temperature difference from the initial state after 365 hours at 800 °C
2.053	–2
1.291	–3
0.511	–5

To construct a drift function for type K thermocouples, the temperature decreases values as well as the wire diameters were used. The data presented in Fig. 2a and Fig. 2b demonstrate that there is a significant increase in the drift rates, measured in °C/hour, for thermoelements with smaller diameters.

The results indicate a significant decrease in the thermoelectric response, with the maximum drift reaching around 196.4 μV for a thermocouple with a diameter of 0.51 mm, 118.6 μV for a thermocouple with a diameter of 1.29 mm, and 79.2 μV for a thermocouple with a diameter of 2.05 mm, respectively, after 365 hours of continuous operation at a temperature of 800 °C.

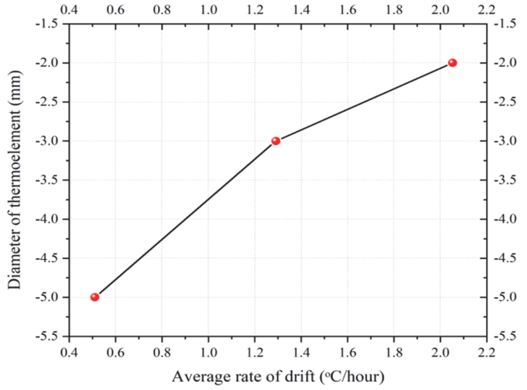


Figure 3 Diameter of type K thermocouple thermoelements and average temperature decree after one hour (measured at 800 °C)

In Fig. 3, each of the red points refers to individual thermocouple's temperature drops by one hour and how the diameter of the thermoelement changes when thermocouples are heated to 800 °C.

3 PREPARATION OF THERMOCOUPLE SAMPLES FOR SEM/EDAX TESTS

Following the completion of drift analysis, visual observations are taken to determine the color, luster, or any other physical characteristics of the oxide scales that are formed. The thermocouple samples obtained as a result of the oxidation process are subjected to SEM/EDAX analysis before being evaluated. Hitachi SEM/EDAX is used for atomic spectroscopy (Model No. S3400N), which has high resolution and 5X-300000X magnification range that are ideal for elemental analysis (10 KV HV mode). It is necessary to establish the repeatability of the testing techniques for a few additional samples. SEM and EDAX analysis may be done on conductive/non-conductive/magnetic materials, and the samples utilised for the analysis can be in powder form, pellet form, film form, or biological form, depending on the application. The deterioration of the sample is investigated in this research using pellets as a type of analysis. As seen in Fig. 4, to perform the analysis, samples are obtained at 5 cm and 10cm above the tip of the thermocouple from both the positive and negative terminals of the thermocouple. When using SEM and EDAX, the sample size is limited to a few millimeters at most. As a result, a sample ranging in size from 0.5 millimetre to 0.7 millimetre is created. Sample A is obtained from the thermocouple's tip. Sample B is collected 5 cm above the thermocouple's positive terminal (Chromel), whereas Sample C is obtained 5 cm above the thermocouple's negative terminal (Alumel). Sample D is 10 cm above the thermocouple tip in the Chromel terminal, whereas Sample E is 10 cm above the thermocouple tip in the Alumel terminal.

4 STRUCTURAL AND THERMOELECTRIC CHARACTERIZATION OF THE THERMOCOUPLES

The present research seeks to determine the extent to which a K-type thermocouple's surface has degraded as a result of extended exposure to very high temperatures. The nominal composition of a standard K-type thermocouple is shown in Tab. 4. The existence of a chemical component in the typical K-type thermocouple that was employed for the experimental investigation is shown in Tab. 5.

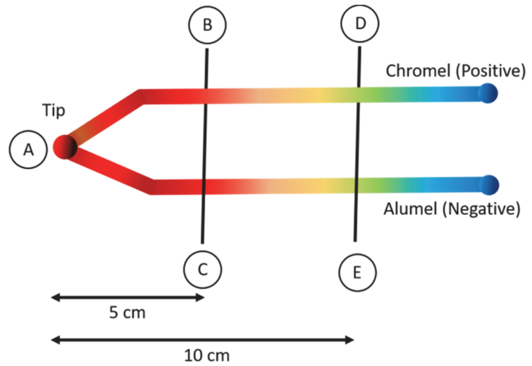


Figure 4 Basic framework for the sample preparation of thermocouple for structural analysis

In Fig. 4, a SEM picture is shown together with the appropriate elemental analysis or chemical characterization done using EDAX at the thermocouple tip, as well as the positive and negative sides. According to the basic premise that each element has a unique atomic structure that allows it to produce a distinct set of peaks on its electromagnetic emission spectrum as follows, the characterisation of the elements in K-type thermocouples is based on the composition mentioned in Tab. 4.

Table 4 Normal composition for chromel and alumel thermoelements

Elements	Chromel / wt%	Alumel / wt%
Ni	90	95
Cr	10	-
Mn	-	3
Al	1	2^1

Table 5 Composition analysis (wt%) of type-K thermocouple

Position	O	Mg	Al	Si	Ca	Cr	Ni
Tip*	17.58	1.23	8.07	1.71	-	3.18	62.94
Chromel	14.79	0.86	1.43	4.17	4.06	3.75	70.93
Alumel	20.10	1.78	0.78	4.81	0.88	6.12	65.54

The structure of a SEM picture of a standard K-type thermocouple is illustrated in Fig. 5, which exhibits a uniform surface at the tip, a positive terminal, and a negative terminal, as well as a uniform surface at the tip.

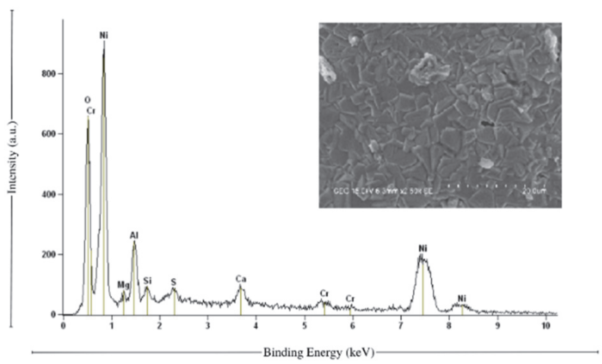


Figure 5a SEM Cross-sectional images of thermoelements subjected to 800 °C for 365 hours, together with thermocouple elemental maps Tip

As seen in Fig. 5a, the bright and dark regions within these microstructures refer to the Ni-Cr and Ni-Al particles, respectively. The microstructures were found to be highly similar to those of the Chromel Alumel composites based on the homogeneity and grain sizes. The well-developed per collation paths of the conductive Mn phase can be clearly seen for all Ni-Cr composites.

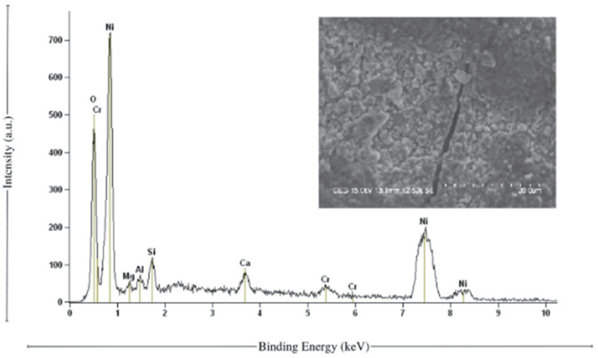


Figure 5b SEM Cross-sectional images of thermoelements subjected to 800 °C for 365 hours, together with thermocouple elemental maps Chromel material (positive)

However, it is important to note that metal silicide secondary phases formed during sintering were observable in the composite microstructures, which is attributable to the effect of wire drawing, which is conducted to get the appropriate wire diameter. The average size of the Ni-Cr grains was measured as 3-5 µm. The K-type thermocouple is placed in a furnace at 800 °C for 1 hour, after which it is quenched in air for 15 minutes. This procedure is done for a total of ten times. A thermocouple voltage is measured at a constant temperature of 800 °C after the thermocyclic oxidation process is completed.

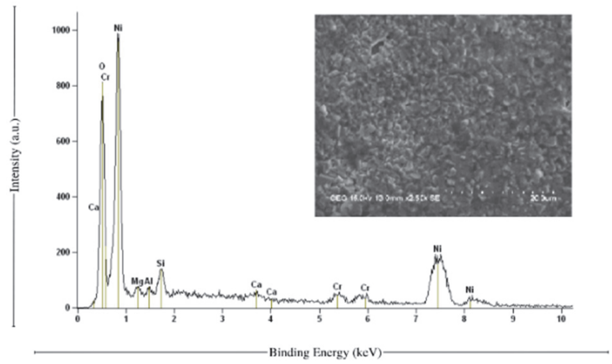


Figure 5c SEM Cross-sectional images of thermoelements subjected to 800 °C for 365 hours, together with thermocouple elemental maps Alumel Material (negative)

It is essential to carry out the entire process a total of seven times. Fig. 5 shows the results of 7 repetitions of the procedure where 5 distinct samples are collected and the corresponding SEM pictures are acquired at a size of 20 µm as shown in Fig. 6.

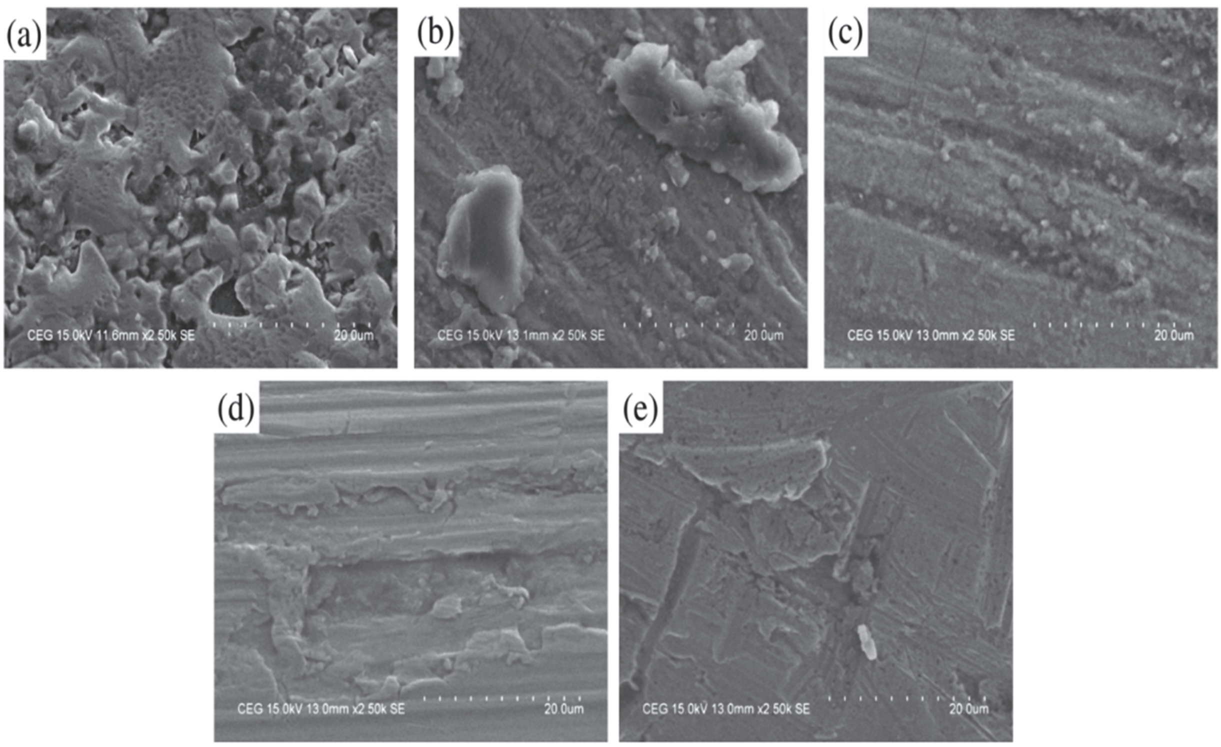


Figure 6 Surface Morphology examined with scanning electron microscope for different position of thermocouple (a) Sample A; (b) Sample B; (c) Sample C; (d) Sample D; and (e) Sample E after sintering at 800 °C for 365 h

The highlights of observation from the SEM image of degraded thermocouple are as follows:

Sample A has been severely deteriorated as a result of the exposure to high temperatures at the thermocouple's tip;

Due to the proximity of this sample to the thermocouple's tip, deterioration will be more rapid than in samples D and E, respectively;

Samples D and E exhibit minor oxide layer formation, adhesion loss, and surface roughening, all of which are indicative of corrosion.

EDAX is used to perform an elemental analysis on a deteriorated thermocouple, and the findings are displayed in Fig. 7. EDAX indicates the net count of the material for each sample as the accelerating voltage increases in the range of 1 to 10 keV for each sample. Depending on the properties of each element in the thermocouple, peaks are recorded, which are sharp lines in the construction of the thermocouple. Brehmstrahlungen (Baseline/Backdrop) is a flat, featureless background with no discernible pattern. Ni, Cr, and Al are the most important elements in a K-type thermocouple.

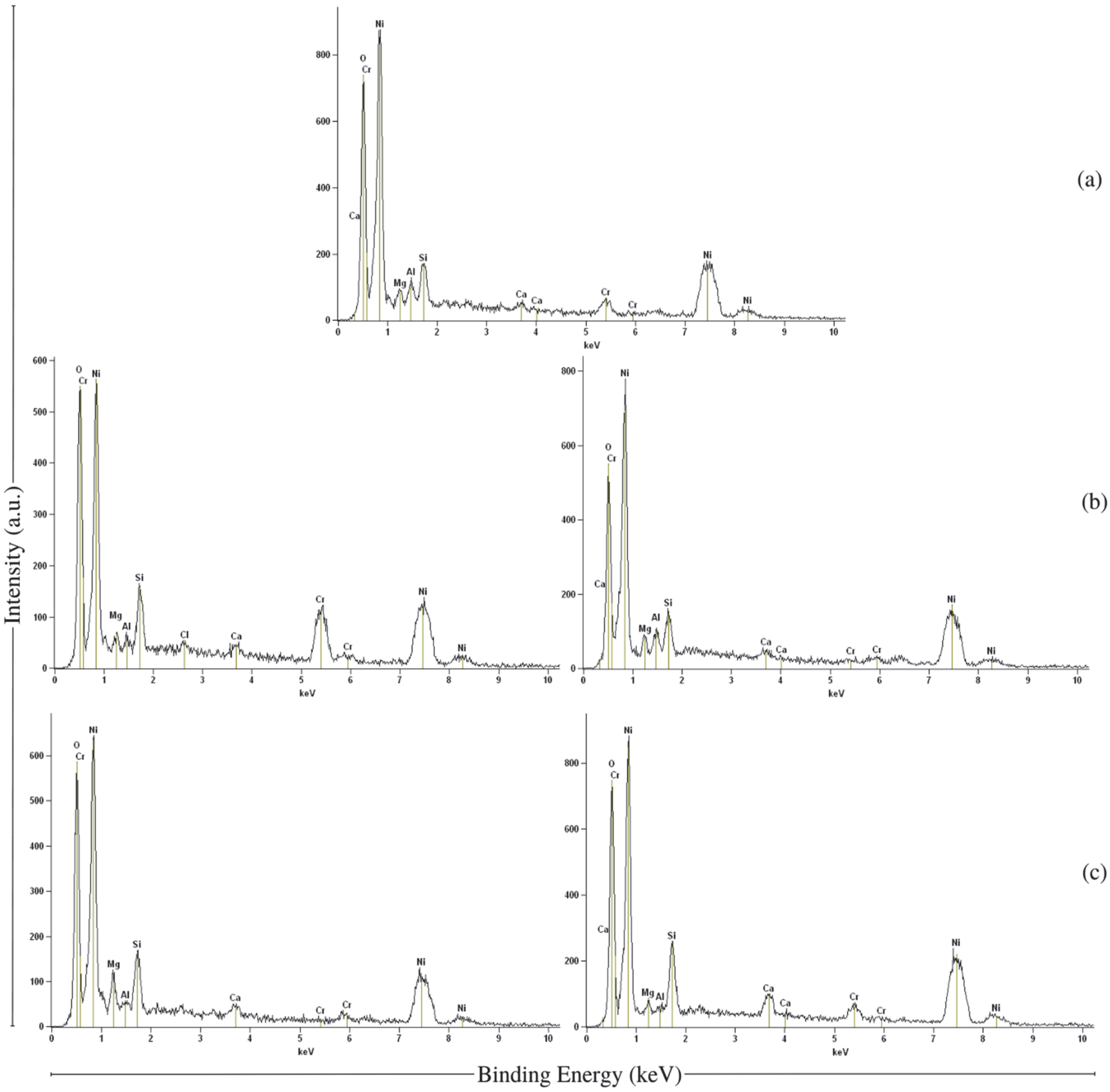


Figure 7 EDAX results in K-type thermocouple (a) Sample A; (b) Sample B & C; (c) Sample D & E

During the degrading process, nickel oxide (NiO_2) was created, chromium oxide (CrO_2) was generated, and aluminium oxide (AlO_2) was formed. On the surface of the thermocouple, these oxides combine to create an oxide layer.

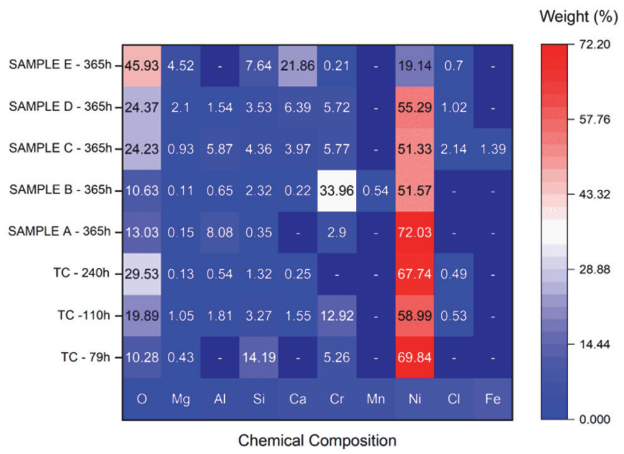


Figure 8 Weight (%) of Chemical composition of 12AWG Thermocouple for different operating time at 800 °C

As seen in Fig. 8 and Fig. 9, compared to a standard thermocouple, the oxygen content of all samples increases up to 45.93%. Hence, it can be concluded that continuous measurement at high temperature followed by air quenching will result in an increasing oxide layer, which will eventually become brittle and break down, resulting in the formation of another layer.

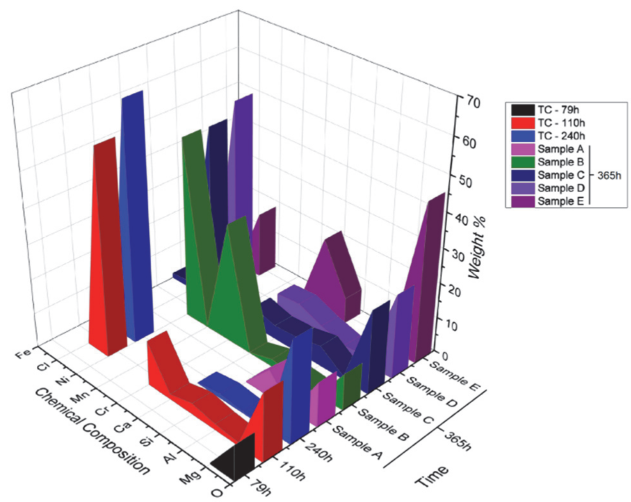


Figure 9 Chemical compositions weight (%) analysis results of 12AWG thermocouple for different operating time at 800 °C

5 CONCLUSION

The collected data indicate a significant correlation between the thermal drift and the diameter of the thermocouple thermoelements. Similar drift behavior was seen at 800 °C. The varied temperature decreases patterns and average degrees Celsius per hour dips for various thermoelement sizes are observed from the results. Further, SEM-EDAX testing is used to examine the deterioration of K-type thermocouples exposed in high temperatures. This work provides a comprehensive investigation of the driftphenomenon observed in thermocouples of varying diameters when subjected to high temperatures, taking into account the composition of their segmental materials. The main causes of the drift data are summarized as follows: The thermoelectric characteristics of a Nicrosil thermoelement changed because chromium migrated out of the thermoelement, deposited on its surface, or was somehow chemically altered. Drift can also be caused by the presence of manganese in the thermocouple material, which is a chemical contamination of the thermoelement material When temperatures reach more than 500 °C and continue at higher degrees, contamination begins. Based on an examination of drift analysis and structural analysis, the levels of drift for different thermoelement diameters are governed by the amount of contaminated material present. Mn naturally dopes chromel thermoelements more quickly than alumel thermoelements. As a consequence of manganese influence on thermocouple thermoelement, the test results indicated that the formation of a significant oxide layer is due to the deterioration of the material.

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