



## Characterizing Drift Behavior in Type K and N Thermocouples After High Temperature Thermal Exposures

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### ABSTRACT

Although of the widespread use of base metal thermocouples in the industry, many previous relevant researches have shown that the accuracy and stability of thermocouples are clearly influenced by any physical or chemical changes in their thermoelements. Among the most important of these changes are the inhomogeneity, pollution, oxidation and microstructure changes of the thermoelements, all of these changes and more leads to thermocouples drift after a prolonged thermal exposure. To study how these changes affect the drift and thermoelectric properties of thermocouples, in this work we subjected the base metal thermocouples of types K and N to successive thermal exposure periods at their maximum temperatures. Scanning electron microscopy (SEM) and Energy Dispersive X-ray (EDX) systems were used to monitor the change in the crystal structure and chemical composition of the thermocouple wires after each stage of the thermal heating, and then we studied the changes in the thermoelectric properties of thermocouple wires. The results showed type N thermocouples are more stable at high temperatures (up to 1050° C), even if used for long periods (for more than 1200 hours) at those temperatures, but K type thermocouples showed a rapid drift with first exposure to high temperatures and completely failed after 600 hours due to devastating corrosion.

## 1. Introduction

As a matter of fact, temperature measurements play a very important role in different fields of industrial applications that directly affects the quality, effectiveness and safety of the final product [1]. Since the improvement in any control system requires that the models that the system based on must be accurate, where different temperature sensors are basic for it, [2] so it is necessary to choose a suitable temperature sensor with high precision, stability and repeatability [1]. Thermocouples consider as the most widely thermometer used in temperature measurements, which include base-metal thermocouples as Types E, J, K, N, T and etc. [3]. As known the Seebeck effect is the change in the electric potential (voltage) along the wire accompanying the

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redistribution of the electrons. It happens only where there is heat flow, so is strictly a temperature gradient effect. The Seebeck coefficient depends on the electronic properties of the conductor so is different for every metal and alloy, and varies with defect or contaminant concentration [4].

Mechanical stress, metallic structure changes, chemical diffusion, contamination and oxidation can affect in the inhomogeneity, stability and drift of thermocouple wires [3] also thought to be the reasons for Seebeck coefficient changes in thermocouples. Since, the various forms of contamination result in the wire is no longer homogeneous along its length, and the degree of wire homogeneity is directly influencing the degree of uniformity in the thermoelectric Seebeck coefficient, all of these leads to instability of the output voltage of the thermocouple and thus the temperature measurement accuracy [5].

As the problem that has been rising is the deterioration of the thermocouple along the time due to defects, so calibration should be conducted in a certain period of time according to the regular standard to maintain the quality of thermocouple. In practice, mechanical impact almost happens every day and cannot be simply avoided i.e. during measurement or handling process. This, in turn, gives significant defects to the thermocouple micro- or even macroscopically. The defect can often reduce and even in some cases ruin the performance of the thermocouple [6].

Due to the large demand for accurate temperature measurements, especially in the range from 500 °C to 1100 °C and a wide using types K and N thermocouples in many of different industrial applications not only for temperature measurement but also for temperature controlling processes [7] so, we have chosen them to make this study. There are also some other reasons; they are the most commonly used base metal thermocouples at high temperatures, have comparable range and price and have a large Seebeck coefficient which increase their sensitivity to any change in electromotive force (emf). So, we can quantify the change in the thermocouple output by calculating the drifts in regular calibrations over long periods of use and studying the change in the resulting error [8].

Despite many studies on the stability of thermocouples, there are no enough studies on the effect of changes in microstructure and chemical composition on the thermoelectric properties of thermocouple wires following thermal exposure.

Since all Ni- based thermocouples are supersaturated solid solutions which are unstable substances and consequently have the natural tendency of the excess solute to precipitate causing the emf drift at elevated temperatures due to solute concentration change and since the drift rate of emf depends on the precipitation rate [4], so in this research, we study the thermoelectric behaviour and microstructure evolution of types K (Nickel-Chromium / Nickel-Alumel) and type N (Nicrosil / Nisil) thermocouples after thermal exposure in multiple stages. Microstructure analysis was done using FEI Quanta 250 FEG Scanning electron microscopy (SEM) which is a very versatile tool in materials characterization integrates successfully with Energy Dispersive X-ray (EDX) detectors that make it ideal for compositional and micro-structural imaging and analysis.

## **2. Experimental Techniques**

In this study, we chose two types of base metal thermocouples (types K and N) to study. In order to test the thermocouple's thermoelectric properties and related microstructure changes, it was required to test them before and after use in different thermal stages. Our experimental work consists of four repeated stages in order to investigate how the base metal thermocouples affected by the long thermal exposure periods at high temperatures, and when each type of thermocouples should recalibrate or thermally re-anneal or do not use again if needed. Each stage represented in the following processes respectively; Microstructure and elemental composition analysis,

homogeneity, calibration, and then exposing the thermocouples at its maximum temperature for 300 hours. Procedures of thermo electrical properties study of type K and N thermocouples can be summarized in the following

- i. Prior of any test or thermal exposure, a sample of each thermocouple wire under test (nearly 4 cm) was analyzed using FEI Quanta 250 FEG system (SEM and EDX) to obtain the initial compositional characterization of the samples,
- ii. Testing the homogeneity of the tested thermocouples at 100 °C,
- iii. Pre-calibration of the assembled thermocouples (type K from 50 °C up to 850 °C) and (type N from 50 °C up to 1050 °C), using high quality temperature standards calibrated on ITS-90,
- iv. Thermal exposure for the thermocouples and thermocouples wires samples at its maximum temperature for 300 hours,
- v. Steps "A" to "D" are repeated after each stage of heat exposure,
- vi. The calibration drift is determined as the emf change (or its temperature equivalent) between the first calibration and the final calibration (re-calibration).

### 2.1 Preparation of Specimens

The thermocouples used in this study were supplied from OMEGA USA (class 1) according to international standards of manufacturing [9]; their details and specifications are given in Table 1. For preparing the thermocouple, nearly about 200 cm from each type of thermocouple wires was cut, and then welded their hot junctions, also for preparation of thermocouple samples, about 4 cm were cut to be suitable for "intermediate" microstructure analysis, where two samples of each thermocouple type from the same pulley were studied. Prior to starting the test, thermoelements or thermocouples must not be subject to any heat treatment, e.g. calibration or annealing.

**Table 1**

Thermocouples description for type K and N thermocouples

Thermocouples Specification	Thermocouple Type	
	K	N
Wires diameter, mm	0.5	1.6
Range, °C	-270 to 870	-270 to 1090
Insulation (sheath)	Nextel Braid	Silica
Thermocouple diameter, mm	2.4	2.4
ASTM (E 230) tolerance (Class 1) @ 1000 °C	0.4 %	0.4 %

### 2.2 Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX) Analysis

All thermoelements of thermocouples under test were analyzed as received and after each heat treatment using FEI Quanta FEG-250 Scanning Electron Microscope (micro analysis technique) with high resolution imaging and elemental composition analysis at the National Research Centre (NRC-Egypt), to obtain the compositional characterization of the samples. EDX analysis was done also for all the samples before and after each thermal exposure period to detect the elemental composition changes.

## 2.3 Calibration of Thermocouples

To assess the drift and homogeneity of thermocouples, we calibrated them as-received and after each stage of thermal exposure; (type K from 50 °C up to 850 °C) and (type N from 50 °C up to 1050 °C). The reference standard used for the calibration was thermocouple type S. All measurements were carried out when thermocouples reference junctions were in ice point. Electromotive force (emf) readings were taken in ( $\mu\text{V}$ ) using Keysight 34420A Nanovoltmeter.

### 2.3.1 Thermal drift

In order to evaluate the thermocouple drift, we calculated the change between the resulting EMF (or its equivalent temperature) of the first calibration and its corresponding after each thermal exposure stage of thermocouple [8].

### 2.3.2 Thermoelectric homogeneity

Homogeneity tests were made for the thermocouples at a fixed temperature 100 °C (as received and after each thermal exposure period for 300 hours). We used a uniform dry well furnace with stability  $\pm 0.005$  °C (Fluke 9170 with immersion depth 16 cm) in which the insertion/withdrawal technique was carried out. After reaching the target temperature and being stable, the thermocouple was inserted slowly into the furnace and its measuring junction was positioned 2 cm below the top surface of the furnace. The thermocouple was held in this position for 15 min and then its electromotive force (emf) was measured. The immersed depth of the thermocouple was then increased by 2 cm and after 5 min its emf was measured again. This procedure was repeated until the thermocouple was fully immersed in the furnace, and then withdrawn from the bottom of the furnace at the same insertion rate by measuring the resultant emf [10].

## 3. Results and Discussion

### 3.1 Effect of Thermal Exposure on Microstructure and Chemical Composition of Thermocouples

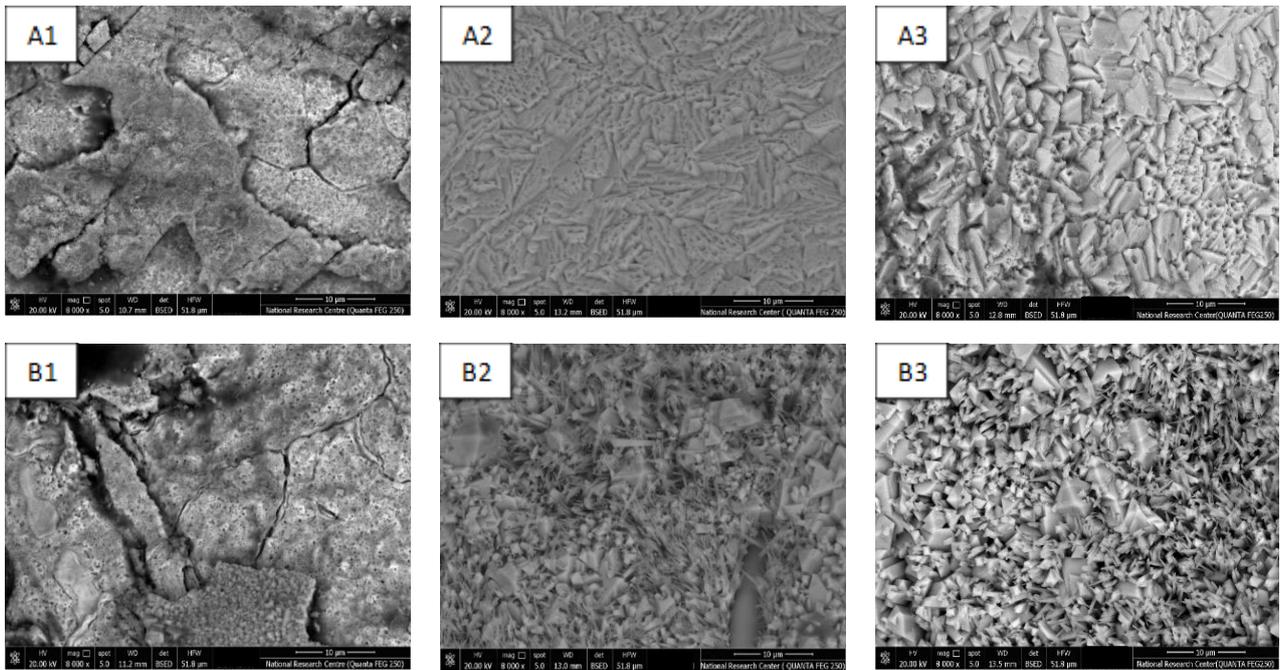
#### 3.1.1 For thermocouple type (K)

Figure 1(A), (B) and Table 2 and 3 represent the results of analyzing type K thermocouple samples by SEM and EDX respectively. Figure 1 exhibits the changes in its microstructure before and after each thermal exposure stage of 300 hours at 850°C. Table 2 shows the EDX analysis of positive (K+) and negative (K-) thermoelements of type-K thermocouple as received and after different thermal exposure periods at 850°C in weight percent (%). Table 3 summaries the rate changes in chemical composition during thermal exposure periods.

In Figure 1, a completely deformation appears on the surface of both K+ and K- thermoelements especially after first thermal exposure for 300 hours at 850°C. Where, both A1 and B1 show large grain boundaries beside cracks at the surface in the as received state (just after welding). After 300 hours of thermal exposure, K+ and K- showed "agglomeration" of short-range ordering (SRO) as shown in Figure A2 and B2. But after 600 hours of thermal exposure, no significant changes in the surface structure as shown in Figure A3, B3 except a slight enlargement in the grain size and appearance of some oxidation.

Table 2 shows that, both thermoelements of K+ and K- are clearly oxidized, but it increases more in K-, and simultaneously the amount of Cr decreases in K+ with increasing the amount of Al

and Mn in K- which cause harsh internal oxidation, plus making K+ toxic via chemical diffusion at high temperatures [11]. Table 3 clarified that K- is more sensitive to oxidation 3 times than K+. The total thermal exposure of type K was only 600 hours because one of its thermoelements (may be K- ) was cut after 600 hours and this may be due to destructive corrosion at about 850 °C, where wires started failing at only 800 °C [12] and also embrittlement along grain boundaries which is a result of thermocycling—Alumel embrittles at lower temperatures than Chromel [13]. The decreasing of Cr to about 9% is in accordance with KRIŪKIENĖ *et al.*, [14] which enhance the oxidation rate in K+.



**Fig. 1.** Microstructure changes of K+ (A) and K- (B) after different periods of thermal exposure at 850 °C. As received (A1, B1), after 300 hours (A2, B2) and after 600 hours (A3, B3)

**Table 2**

Chemical composition changes in positive (K+) and negative (K-) thermoelements of type-K thermocouple before and after different thermal exposure periods in Weight Percent (%)

Terminal	Heat exposure Stage (hours)	Ni	Cr	Al	O	C	Mn	Si	Ca
K+ (Ni-Cr)	As received	50.24	12.02	-----	14.46	21.1	0.96	0.52	0.7
	After 300	71.15	0.92	-----	19.58	8.35	-----	-----	-----
	After 600	78.54	2.54	-----	18.92	-----	-----	-----	-----
K- (Ni-Al)	As received	64.55	-----	0.82	10.86	21.17	1.28	1.32	-----
	After 300	59.31	-----	0.5	23.15	8.87	8.17	-----	-----
	After 600	52.56	-----	0.96	28.44	15.83	-----	1.15	1.06

As a recommendation, 0.5 mm Type K should not be used above 750 °C as suggested in the ASTM manual (used at a temperature well below the 870 °C) as well as over 700 °C, the user should ensure sufficient clean air to avoid green rot [12].

**Table 3**

Conclusion of chemical composition changes in type K thermocouple thermoelements over all thermal exposure periods

EDX analysis of positive leg,			EDX analysis of negative leg,		
Element	Weight Change (%) after all thermal exposure (600 h)		Element	Weight Change (%) after all thermal exposure (600 h)	
C	- 21.1 %	↓	C	- 5.34 %	↓
O	+ 4.46 %	↑	O	+ 17.58 %	↑
Cr	- 9.48 %	↓	Al	+ 0.14 %	↑
Ni	+ 28.3%	↑	Ni	- 11.99 %	↓

### 3.1.2 For thermocouple type (N)

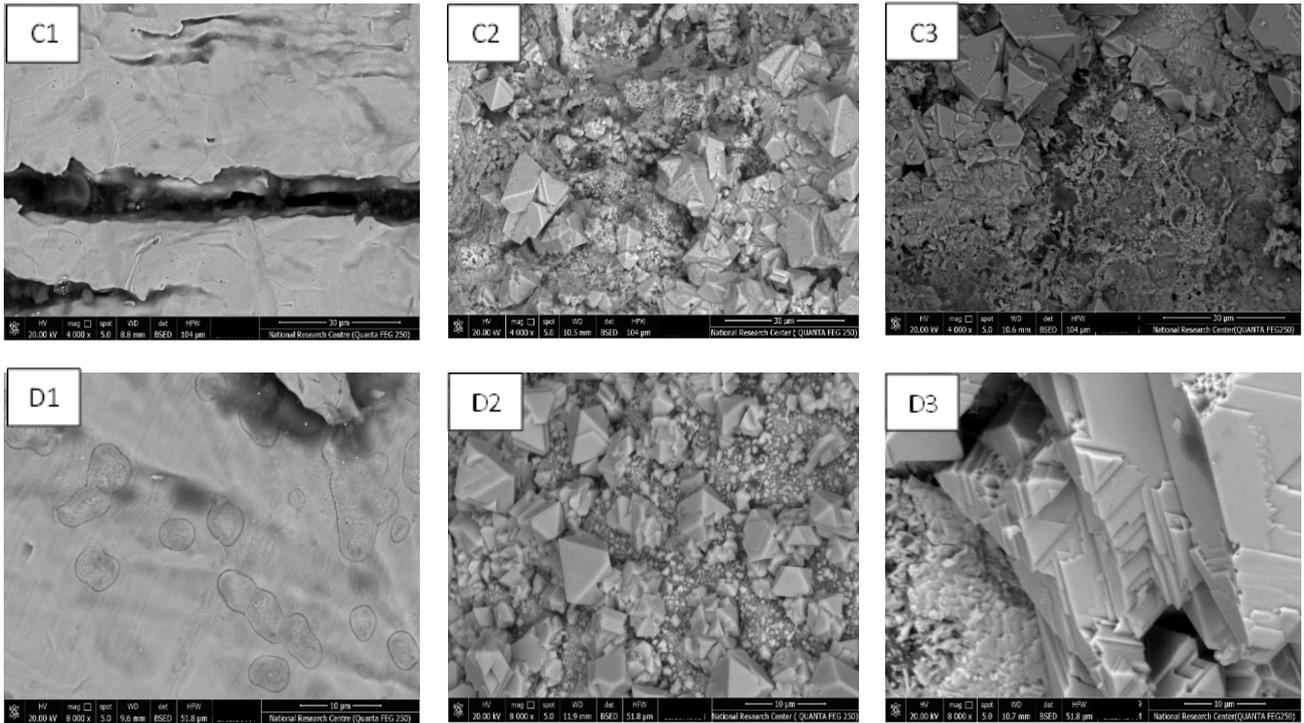
Figure 2(C), (D) and Tables 4 and 5 represent the results of analyzing type N thermocouple samples by SEM and EDX respectively. Figure 2 exhibits the changes in its microstructure as received and after 300 hours and 1200 hours of thermal exposure at 1050°C. Table 4 shows the EDX analysis of positive (N+) and negative (N-) thermoelements of type-N thermocouple as received and after different thermal exposure periods at 1050°C in weight percent (%). Table 5 summaries the rate changes in chemical composition during heat exposure periods.

In Figure 2, the structure of both N+ and N- thermoelements is affected by thermal exposure at high temperatures for long periods (1200 hours). Where, N+ is affected only after first 300 hours of thermal exposure at 1050 °C as occurred in K+ at 850 °C and after that there is no significant change till 1200 hours, whereas N- is affected after each stage of thermal exposure leading to accumulative large grain size and oxidation with time exposure.

Before thermal exposure, there are some cracks in the N+ surface as shown in C1, whereas N- indicates some SRO with nearly smooth surface as shown in D1. After 300 hours, both N+ and N- are subjected to change with the formation of SRO as indicated in C2 and D2. After final exposure of 1200 hours; N+ nearly not affected while N- showed enlargement in the grain size with time exposure as well as oxidation in both N+ and N- as shown in C3 and D3.

Table 4 shows increasing both Cr and Si quantities by about 12% and 4.5% respectively after 300 hours in the N+, this led to the formation of oxidation resistors but N- terminal is oxidized with decreasing the amounts of Si and Mg [3]. In all subsequent heating stages, the N+ nearly not affected, unlike the N- which shows increasing and decreasing oxidation moreover the presence of other elements like Si, Mn, Mg, C....etc. Table 5 investigates that Nisil (N-) is sensitive to oxidation than Nicrosil (N+).

Type N thermocouple is preferable than type K thermocouple because the higher Cr content of the Nicrosil thermoelement makes it far less susceptible to green-rot than K+ which prefers Cr oxidation especially above 700 °C [3].



**Fig. 2.** Microstructure changes of N+ (C) and N- (D) after different periods of thermal exposure. As received (C1, D1), after 300 hours (C2, D2) and after 1200 hours (C3, D3)

**Table 4**

Chemical composition changes in positive (N+) and negative (N-) thermoelements of type-N thermocouple before and after different thermal exposure periods in Weight Percent (%)

Terminal	Heat exposure Stage (hours)	Ni	Cr	Si	Mg	O	C	Mn	Na
N+ (Ni-Cr-Si)	As received	68.55	11.91	1.71	1.62	-----	16.2	-----	-----
	After 300	44.88	24.27	6.31	0.98	-----	21.47	2.08	-----
	After 600	59.13	13.05	1.57	-----	20.42	5.83	-----	-----
	After 900	64.13	13.15	1.29	0.87	19.13	-----	1.41	-----
	After 1200	48.16	20.36	3.59	1.15	24.87	-----	1.87	-----
N- (Ni-Si-Mg)	As received	69.68	0.6	4.17	1.78	7.17	14.43	0.2	1.97
	After 300	68.75	2.87	1.98	-----	22.31	4.09	-----	-----
	After 600	65.67	-----	9.12	0.23	18.08	6.89	-----	-----
	After 900	88.39	2.19	-----	0.45	7.8	-----	1.16	-----
	After 1200	76.83	2.55	0.56	-----	15.79	3.39	0.89	-----

**Table 5**

Conclusion of chemical composition changes in thermocouple (type N) thermoelements over all thermal exposure periods

EDX analysis of positive leg,			EDX analysis of negative leg,		
Element	Weight Change (%) after all thermal exposure (1200 h)		Element	Weight Change (%) after all thermal exposure (1200 h)	
C	- 16.2 %	↓	C	-11.04 %	↓
O	+ 24.87%	↑	O	+ 8.62 %	↑
Si	+ 1.88 %	↑	Mg	- 1.78 %	↓
Cr	+ 8.45 %	↑	Si	- 3.61 %	↓
Ni	- 20.39%	↓	Ni	+ 7.14 %	↑

### 3.2 Effect of Thermal Exposure on Thermoelectric Properties of Type K and N Thermocouples

#### 3.2.1 Drift of thermocouples

Thermal drift was measured for thermocouples (types K and N) held at a fixed immersion in a 150 mm long, of the dry block furnace. All thermocouples were calibrated as-received and after each thermal exposure period at different calibration points covering the temperature range of each type using dry block furnaces, (type K from 50 °C up to 850 °C) and (type N from 50 °C up to 1050 °C). Table 6 shows the drift of thermocouples at their maximum calibration point.

##### 3.2.1.1 For type K thermocouple

Although an early cut occurred in the thermocouple type K, it showed a rapid drift before cutting. The maximum drift is observed at mid-range ~ 400 °C (equivalent to -3.5 °C) and at the maximum temperature 850 °C (~-3.4 °C). This is due to the fact that, when the type K thermocouple is exposed at temperature above 600 °C, a portion of the positive thermoelements will undergo the short range order transformation and drift will take place [15] as described before in (3.1.1).

Therefore, care should be taken when using the thermocouple type K at high temperature (above 1000 °C), because you may have to replace it quickly. Alumel, Nicrosil, and Constantan are thought to be the individual thermoelements responsible for most of the thermal drift.

**Table 6**

Drift of thermocouples at their maximum calibration temperature

Heat exposure stage	T/C drift = (Error after each heat exposure stage - Error in initial state)	
	Type-K @ 850 °C	Type-N @ 1050 °C
Initial state	0	0
After 300 hours	-3.4	3.9
After 600 hours	---	3.3
After 900 hours	---	4.3
After 1200 hours	---	3.9

##### 3.2.1.2 For type N thermocouple

The drift of thermocouple type N is occurring only after the first thermal exposure for 300 hours at 1050° C and it showed a significant stability in all following thermal exposure stages after that. An earlier study by Bentley and Russel [16], in which 750°C and 1050 °C thermal treatments were used, found the drift could be reduced by a factor of 2-4, but was manufacturer dependent.

Therefore, we recommend a heat treatment for type N thermocouple at 1050° C for 300 hours before use. Type N thermocouple is the most stable base metal thermocouple up to 1000° C. One of the most important causes of thermocouple drift is contamination of one or two of the thermocouple legs. Contamination can be built in, acquired in service or originate from the thermocouple alloy. Another source of contamination is heating elements or insulation materials of the furnaces. So, it is recommended more frequently recalibration of thermocouples to define the drift rate and hence thermocouples life expectancy.

#### 3.2.2 Inhomogeneity profile of thermocouples

##### 3.2.2.1 For thermocouple type (K)

Figure 3(a) and (b), shows the inhomogeneity profile for type K thermocouple at 100 °C as received and after 300 hours of thermal exposure, respectively. The deviation of the

thermal emf relative to the reference function is plotted as a function of immersion depth. The inhomogeneity of the type K thermocouple changed from  $0.4 \mu\text{V}$  ( $\sim 0.01^\circ\text{C}$ ) to  $3.1 \mu\text{V}$  ( $\sim 0.07^\circ\text{C}$ ) after 300 hours (from the first time of thermal exposure periods) as shown in Figure 3(a) and (b). Where, thermocouple type K was only tested after 300 hours exposure because one of its thermoelements (may be K-) was cut after 600 hours.

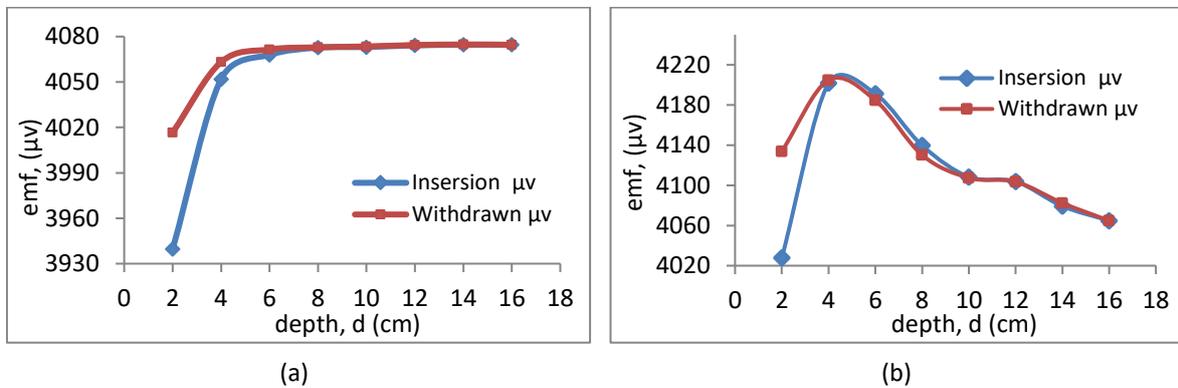


Fig. 3. Immersion profile of type K. (a) "as received" and (b) "after 300 hours thermal exposure"

### 3.2.2.2 For thermocouple type (N)

Figure 4(a) and (b), shows the inhomogeneity profile for type N thermocouple at  $100^\circ\text{C}$  as received and finally after 1200 hours of thermal exposure, respectively. The deviation of the thermal emf relative to the reference function is plotted as a function of immersion depth. The maximum inhomogeneity of type N thermocouple is  $11.8 \mu\text{V}$  which is equivalent to  $0.39^\circ\text{C}$ .

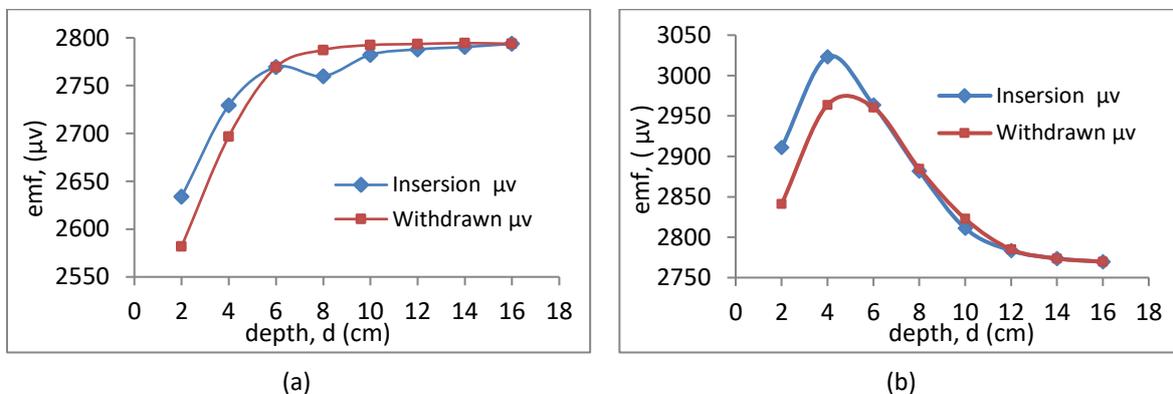


Fig. 4. Immersion profile of type N. (a) "as received" and (b) "after 1200 hours thermal exposure"

### 3.2.3 Seebeck coefficient changes of thermocouples due to thermal exposure

As mentioned earlier the Seebeck coefficient stability is the base where the thermocouple work is dependent on it. Where, the change in Seebeck voltage,  $S$ , occurs only where there is a temperature gradient and in proportion to the temperature gradient.

Seebeck voltage,  $S$ , is defined from Eq. (1)

$$\Delta E = S \times \Delta T, \quad S = \Delta E / \Delta T \quad (1)$$

where,  $S$  is the Seebeck coefficient,  $\Delta E$  is the average EMF generated in thermocouple wires and  $\Delta T$  is the temperature difference between the measuring and reference junctions of thermocouple.

The percentage error in Seebeck coefficient  $\Delta S\%$  is calculated from Eq. (2)

$$\Delta S\% = \frac{\Delta S}{S} = \frac{S_{meas} - S_{ref}}{S_{ref}} \times 100 \quad (2)$$

where,  $S_{meas}$  is the average Seebeck coefficient inferred from the measured voltage and the temperatures measured either side of the scanner gradient, and  $S_{ref}$  is the corresponding Seebeck coefficient inferred from the thermocouple reference tables. Consequently, the results reflect the thermocouples' adherence to the reference functions as well as their inhomogeneity and drift [17].

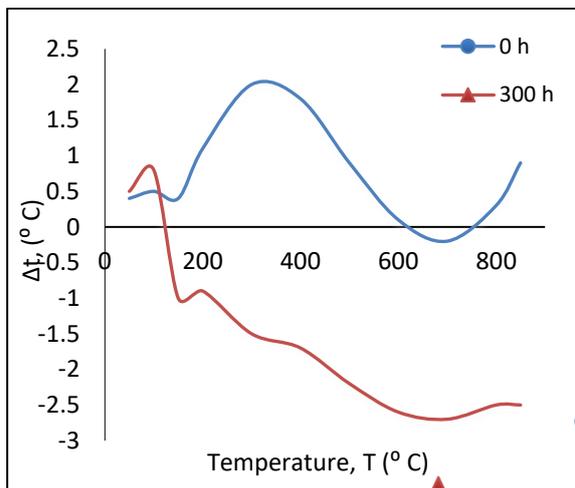
### 3.2.3.1 For thermocouple type (K)

As shown in Figure 5(a), there is a clear change in the calibration results after 300 hours of thermal exposure at 850 °C from that in the as received state (sine wave) while after 300 hours, there is a drift in one direction (negative) which increased with increasing temperatures, the temperature correction ( $\Delta t$ ) is calculated from Eq. (3)

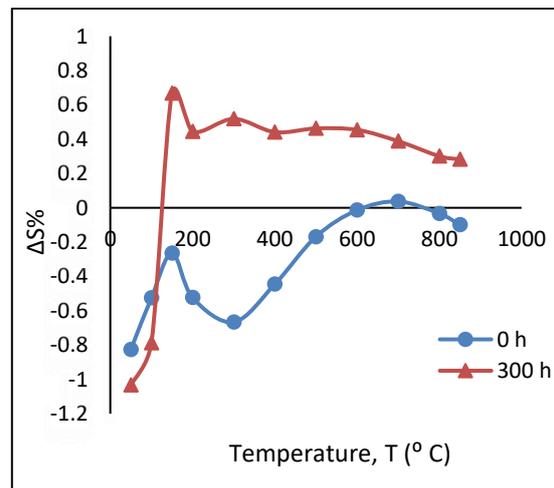
$$\Delta t = t_s - t_m \quad (3)$$

where,  $t_s$  is the standard temperature and  $t_m$  is the measured temperature.

Seebeck coefficient changes are presented in Figure 5(b), where the maximum change is observed at 300 °C (~0.5%) after first exposure for 300 hours and it is nearly constant along the range after that (~0.4%). As the time exposure increased the drift increased with significant value, especially at mid-range and maximum temperatures, while below 150 °C the drift is low ~10 $\mu$ v whereas above 150 °C, drift ranged from 50 $\mu$ v up to 145 $\mu$ v.



**Fig. 5. (a)** Temperature correction as a function of temperature between 50 °C and 850 °C for Type K thermocouple in the first state and after 300 hours of thermal exposure



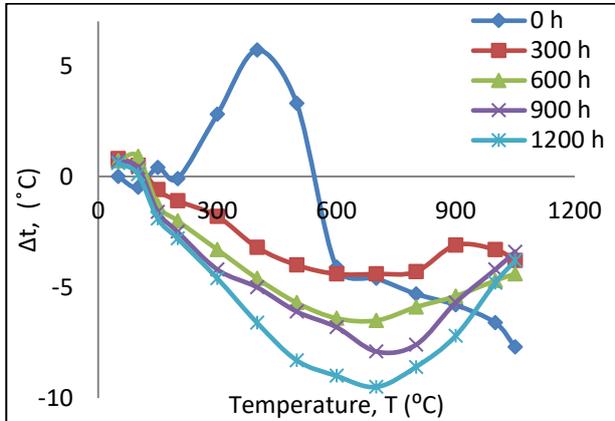
**Fig. 5. (b)** Change in Seebeck coefficient from the reference functions for Type K thermocouple in the first state, and after 300 hours of thermal exposure

### 3.2.3.2 For thermocouple type (N)

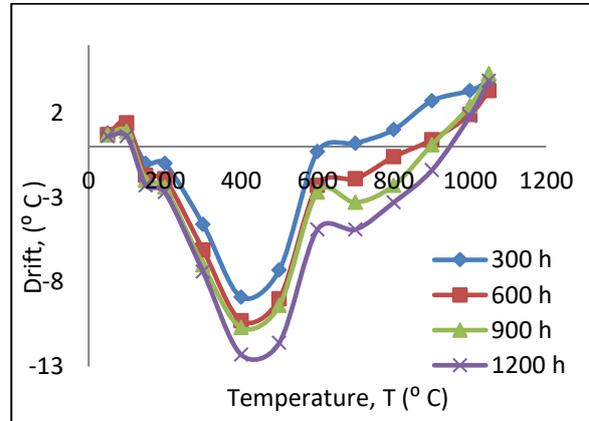
Figure 6(a), (b), (c) and (d) present the calibration results and drifts after periodically heat treatments for Type (N) at 1050 °C. In Figure 6(a) a clear change is observed in the calibration results after the first thermal exposure of 300 hours at 1050 °C. After different periods of heat exposure (600, 900 and 1200) hours, they are showed a nearly constant behaviour of correction in one direction (negative) as in type (K) which increased with increasing temperatures until reached the maximum correction after 1200 hours (~-9.5 °C at 700 °C) and decreased with temperature after that (~-3.8 °C at 1050 °C). Figure 6(b) exhibits the temperature drifts of type N thermocouple after different periods of heat exposure at 1050 °C (300, 600, 900 and 1200) hours where the maximum drift is recorded at the mid-range ~400 °C (~-12.3 °C) and at the maximum temperature 1050 °C (~+3.9 °C) at 1200 hours.

Seebeck coefficient changes are presented in Figure 6(c) where  $\Delta S\%$  is increased with time exposure and the maximum change is observed at 400 °C (~2%) after 1200 hours at 1050 °C. The behaviour is the same in all periods of 300, 600, 900 and 1200 hours with maximum change at 400 °C and decreased after that till reaching a minimum value at 1050 °C ~0.4% after thermal exposure of 1200 hours. With increasing thermal exposure times, the drift increases with significant value ~100  $\mu\text{V}$  especially @ range (400 to 600 °C) after 1200 hours, at 700 °C and 800 °C after 900 hours.

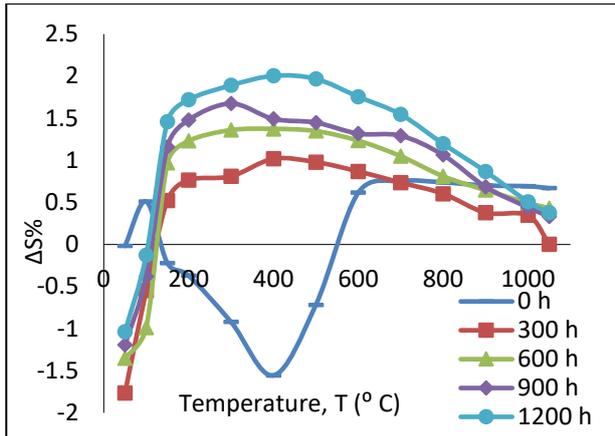
Below 150 °C the drift is ranged from ~7 $\mu\text{V}$  up to 93 $\mu\text{V}$  and at 1050 °C it is nearly constant ~100  $\mu\text{V}$  at all periods of thermal exposure 300, 600, 900 and 1200 hours as in Figure 6(d). The maximum positive shift in Figure 6(c), (d) at 400 °C may be due to the SRO of Nicrosil thermoelements as a result of impurities precipitation and the negative shift in Figure 6(d) which occurred at 1000 °C and 1050 °C may be attributed to oxidation accumulated with SRO also.



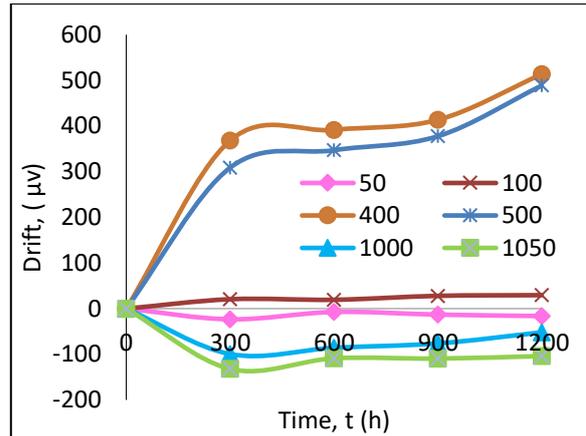
**Fig. 6. (a)** Temperature correction as a function of temperature between 50 °C and 1050 °C for Type N thermocouple after time periods of 0, 300, 600, 900 and 1200 hours



**Fig. 6. (b)** Temperature drifts of type N thermocouple after 300, 600, 900 and 1200 hours of thermal exposure at 1050 °C



**Fig. 6. (c)** Change in Seebeck coefficient from the reference function for Type N thermocouple in the first state, and after different periods of thermal exposure for time periods of 0, 300, 600, 900 and 1200 hours



**Fig. 6. (d)** Emf drifts as a function of time along the range of Type N thermocouple

#### 4. Conclusion

Base metal thermocouples (types K and N) were chosen to study the changes occurring after long thermal exposure periods. The study included observing the changes in the thermoelectric properties of thermocouples over different thermal exposure periods and then studies the change in microstructure in those same stages of thermal exposure, in order to investigate the effect of crystalline change on the metrological performance of thermocouples.

Although various changes happened in type K and N thermocouples microstructure and their test results of homogeneity, drift and Seebeck coefficient, but type N thermocouples appear stable at high temperature up to 1050 °C for long periods (about 1200 hours), where the drift is appeared in a significant value (3.9 °C) after the first time of thermal exposure and later become nearly stable.

However, the results are in good accordance with the results presented elsewhere [3,18,19,20] but thermocouples type K showed a rapid drift for the first time of thermal exposure (~3.4 °C) and its life is timed out at 600 hours of thermal exposure. Where, its alloys undergo oxidation at high temperatures when exposed to oxygen and other oxidizing agents forming the oxide film on their surface. This film is brittle and less strength than metal itself, which reduces the mechanical strength of thermoelements.

Microstructure analysis appears that, one of the most important causes of thermocouple drift is contamination of one or two of the thermocouple legs. Alumel and Nicrosil thermoelements are responsible for most of the thermal drift of type K and N thermocouples respectively.

It is recommended to anneal types K and N thermocouples before beginning their use for 300 hours at their maximum temperatures (850 °C and 1050 °C respectively) and then calibrate them, also recalibrate more frequently in order to define the rate of drift and reduce it and hence increase the base metal thermocouple's life expectancy.

Users of type K and N thermocouples should also take into account the cleanliness of the environment surrounding the thermocouples by using ultra-pure ceramic insulators and avoid any stress or mechanical damage on the wires.

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