

Thermal Recovery from Cold-Working in Type K Bare-Wire Thermocouples

A. D. Greenen¹  · E. S. Webster²

Received: 2 August 2016 / Accepted: 7 October 2017 / Published online: 23 October 2017
© Springer Science+Business Media, LLC 2017

Abstract Cold-working of most thermocouples has a significant, direct impact on the Seebeck coefficient which can lead to regions of thermoelectric inhomogeneity and accelerated drift. Cold-working can occur during the wire swaging process, when winding the wire onto a bobbin, or during handling by the end user—either accidentally or deliberately. Swaging-induced cold-work in thermocouples, if uniformly applied, may result in a high level of homogeneity. However, on exposure to elevated temperatures, the subsequent recovery process from the cold-working can then result in significant drift, and this can in turn lead to erroneous temperature measurements, often in excess of the specified manufacturer tolerances. Several studies have investigated the effects of cold-work in Type K thermocouples usually by bending, or swaging. However, the amount of cold-work applied to the thermocouple is often difficult to quantify, as the mechanisms for applying the strains are typically nonlinear when applied in this fashion. A repeatable level of cold-working is applied to the different wires using a tensional loading apparatus to apply a known yield displacement to the thermoelements. The effects of thermal recovery from cold-working can then be accurately quantified as a function of temperature, using a linear gradient furnace and a high-resolution homogeneity scanner. Variation in these effects due to differing alloy compositions in Type K wire is also explored, which is obtained by sourcing wire from a selection of manufacturers. The information gathered in this way will inform users of Type K thermocouples about the potential consequences of varying levels of cold-

Selected Papers of the 13th International Symposium on Temperature, Humidity, Moisture and Thermal Measurements in Industry and Science.

✉ A. D. Greenen
adam.greenen@npl.co.uk

¹ National Physical Laboratory (NPL), Teddington, UK

² Measurement Standards Laboratory (MSL), Lower Hutt, New Zealand

working and its impact on the Seebeck coefficient at a range of temperatures between $\sim 70^\circ\text{C}$ and 600°C . This study will also guide users on the temperatures required to rapidly alleviate the effects of cold-working using thermal annealing treatments.

Keywords Base metal · Cold-work · Inhomogeneity · Thermocouple · Type K

1 Introduction

The Type K thermocouple formulated by Hoskins in the early twentieth century [1] is still widely used, particularly in industrial settings, even with the advent of arguably more stable base metal thermocouples such as Type N [2]. Due to the popularity of the Type K thermocouple, it has been extensively characterized such that a number of contributing factors causing larger-than-expected temperature measurement errors are known due to drift in the Seebeck coefficient. These include short-range ordering (SRO), oxidation and the introduction of defects caused by cold-working along with any subsequent thermal recovery. Drift due to cold-working and thermal recovery will be the focus of this paper. A review of the first two mechanisms is presented by Webster [3] (and crossreferences within).

The first dedicated investigations into the effects of cold-working on thermal recovery where quantifiable amounts of strain were applied to Type K thermocouples was in the early 1960s [4, 5]. Strain was typically applied linearly, or by swaging to obtain wire extensions of over 20 % or so. Potts and McElroy opted to use a homogeneity scanner based on the designs by White [6] which became the de facto standard methodology for quantifying inhomogeneity in thermocouples, and it appeared that cold-working was accelerating SRO in the positive Type K thermoelement. Further research [7] suggested that high mobility grain defects also caused changes in the Seebeck coefficient. Developments in spectroscopic techniques allowed the characterization of the high mobility grain defects caused by cold-working [8]. The resolution of identifying inhomogeneities also improved, largely thanks to improved homogeneity scanners developed by Fenton [9].

The aim of this paper is primarily to look at the effect of thermal recovery between $\sim 7^\circ\text{C}$ and 600°C in bare-wire Type K thermocouples that have been subject to cold-working. The recent development of a high-stability linear gradient furnace and high-resolution homogeneity scanner at MSL has enabled the recovery processes to be studied in far greater detail than in earlier studies.

2 Experimental Details

Three key pieces of equipment were used in the experimental work described in this paper: the tensional loading apparatus, gradient furnace and steam heat-pipe scanner. The gradient furnace (GF) has ten temperature-controlled zones, producing a linear temperature gradient from 100°C to 1000°C , with a standard deviation of $\pm 1.5^\circ\text{C}$ with respect to variation from linearity [10]. The steam heat-pipe scanner consists of a very steep, but well-controlled temperature gradient between a steam and acetone

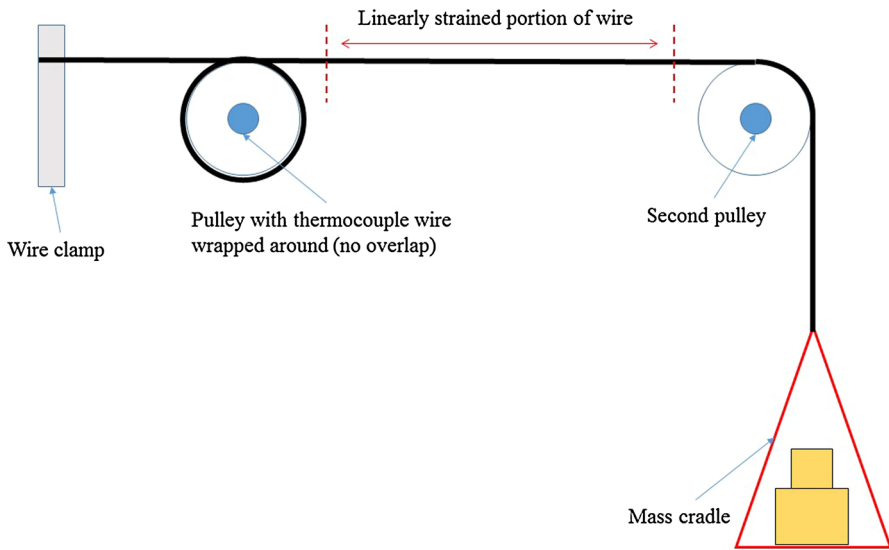


Fig. 1 Tensional loading apparatus

heat pipe, allowing for localized, high-resolution (down to several mm) measurement of the Seebeck coefficient along thermocouple wires [11]

2.1 Tensional Loading Apparatus

Winding and unwinding thermoelements around a mandrel, bending and swaging are common methods of applying cold-working [9, 12]. In practice, it is difficult to reproducibly apply the same amount of cold-working to different wires using these processes, due to uncontrolled and unquantifiable stresses and strains applied to the wire. In order to produce a repeatable, quantifiable level of cold-working in thermocouple wires, a tensional loading apparatus depicted in Fig. 1 is used in this study allowing known yield displacement, or strain, to be applied linearly across the length of wire. The engineering strain (e) is defined by the extension from a prestrained (L) to a post-strained (l) portion of wire as shown.

$$e = \frac{l - L}{L} \%$$

The linearity of the strain applied between the two pulleys was verified by marking a test piece of wire at regular intervals and applying strain. By adding small increments of weight to the mass cradle while slowly lowering it under a controlled fashion, the amount of strain applied could be tightly controlled. All of the straining was performed in a temperature-controlled room.

Table 1 EDS analysis showing the major constituents of Type K wire from a range of manufacturers

Thermoelement manufacturer and type	Element content in wire % (\pm to $k = 1$)						
	Nickel	Chromium	Silicon	Aluminum	Manganese	Iron	Copper
Hoskins 0.5 mm Chromel	89.63 (2.02)	8.89 (0.32)	–	–	0.66 (0.17)	0.82 (0.17)	–
Hoskins 0.5 mm Alumel	93.75 (2.01)	–	2.31 (0.28)	2.70 (0.34)	1.24 (0.19)	–	–
M4 0.5 mm K+	87.84 (2.02)	9.21 (0.31)	2.96 (0.21)	–	–	–	–
M4 0.5 mm K–	92.72 (2.07)	–	2.42 (0.30)	3.27 (0.42)	1.6 (0.25)	–	–
M6 0.5 mm K+	88.70 (1.95)	9.40 (0.34)	1.28 (0.18)	–	–	0.61 (0.17)	–
M6 0.5 mm K–	97.06 (1.95)	–	1.32 (0.17)	–	0.79 (0.14)	0.84 (0.14)	–
M8 0.5 mm K+	86.10 (1.73)	7.79 (0.23)	0.19 (0.07)	–	–	4.39 (0.20)	1.53 (0.25)
M8 0.5 mm K–	90.58 (1.74)	0.20 (0.12)	2.48 (0.22)	2.54 (0.25)	1.66 (0.19)	2.53 (0.16)	–

2.2 Wires Under Test

The thermocouple wires were sourced from a variety of manufacturers in order to see how different alloys of Type K wire are affected. Table 1 shows each positive and negative thermoelement for each Type K wire manufacturer in this study, and the relative composition of each wire was obtained by energy-dispersive X-ray spectroscopy (EDS). Apart from the original Hoskins, the name of each manufacturer of Type K wire has not been disclosed.

2.3 Thermocouple Assembly/Experimental Procedures

In all instances, a strained portion of wire (700 mm long) is butt-welded to a short, 50 mm section of new wire ‘off the reel,’ serving as a reference inside the gradient furnace and also an idea of how much the Seebeck coefficient changes after cold-working (prior to thermal treatment). Another new piece of wire is also butt-welded to the other end of the strained wire to create a thermoelement approximately 1.5 m long. This wire is then fed into one bore of an alumina twin-bore sheath (4 mm diameter, 1 mm bores and 1 m long). Into the other bore is fed the opposing thermoelement, which is not subject to any cold-working, to create a bare-wire Type K thermocouple. The inhomogeneity of the thermocouple is then assessed using the steam heat-pipe scanner to provide information on the initial state. A representative scan of the first 750 mm of a scanned thermocouple wire is shown in Fig. 2 (the Relative Seebeck change is explained in the next section), with different amounts of strain; the transition from the 50 mm section of unstrained wire can be shown. It should be noted the 50 mm

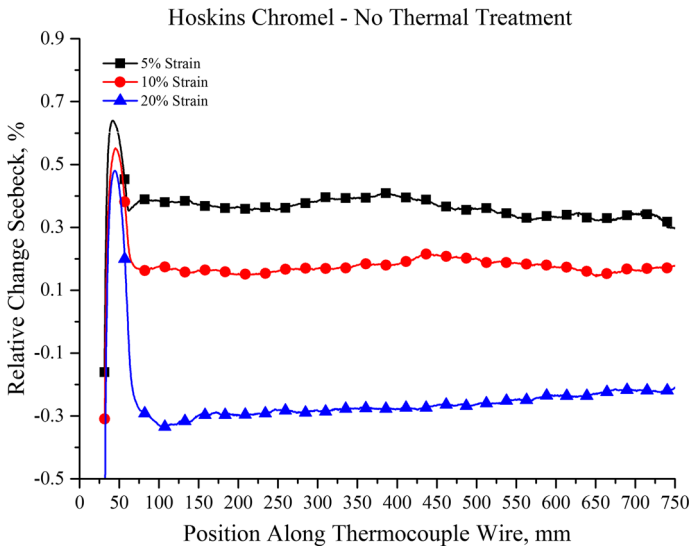


Fig. 2 Hoskins Chromel wire in the initial strained state

wire section will usually be consistent, but not necessarily identical to the reference function (a Hoskins thermocouple typically has a Seebeck coefficient 0.7% higher than the reference function in this state for example). The rapid change in the Seebeck coefficient from 0 mm to 50 mm can be attributed to conduction errors when the tip of the thermocouple is very close to the top of the heat pipe.

The three key procedures in this study are listed below.

Procedure 1.1/1.2: For procedure 1.1, the strain in the Chromel/K+ thermoelements was investigated at 0%, 5%, 10% and 20%. A strain of 20% was found to be the maximum that could be applied before the failure point was reached. The Hoskins sample was aged in the gradient furnace for periods of between 5 min and 20 h to build a detailed picture of the thermal recovery as a function of temperature and time. Other manufacturer's samples were only aged for a single 4 h period, as it was found this was sufficient to indicate the thermal recovery process. For procedure 1.2, the strain and thermal recovery tests detailed above are also conducted on Hoskins Alumel and K- thermoelements.

Procedure 2: Comparisons were made between linear stretching and nonlinear bending to induce strain in Hoskins Chromel. Mandrel diameters of 1.5 mm and 5.2 mm were used to bend the wire samples. Samples were again aged in the GF for a period of 4 h to gauge the thermal recovery process.

Procedure 3: Investigating the thermal recovery from 0% and 20% cold-working for a Hoskins Chromel and two other K+ thermoelements from different manufacturers in two different thermal states is carried out. The first is the as-received anneal state (AR) with unknown thermal and cold-work history from the manufacturers. The second is the 900 °C quench anneal state, known as the 'ground state' (GS). The 900 °C quench anneal involves isothermally heating the entire (unstrained) sample to 900 °C

for 5 min, followed by a rapid removal from the furnace. The thermoelement is then allowed to air quench to room temperature within the alumina sheath, placing it in the ground state [2,8] in which the crystal lattice structure will be in a disordered state, with any preexisting cold-work removed. Cold-work is then applied to the annealed wires and GF aging of 4 h conducted followed by scanning.

3 Results

3.1 Cold-Worked Chromel/K+ Thermoelements (Procedure 1.1)

Initially, the Chromel thermoelement from Hoskins is strained to provide a baseline for other wire manufacturers. The thermal recovery as a function of temperature is captured at set time intervals to capture the rate of the recovery process for wires with a varying amount of strain applied. Figure 3a–c displays the thermal recovery at 5 %, 10 % and 20 % strain and additionally, a repeat at 20 % strain to validate the repeatability of the tensional loading apparatus and gradient annealing procedure.

By displaying the inhomogeneity of each thermocouple as a *relative* ($\Delta S/S$) change from the Seebeck coefficient, the results can be uncoupled from the scanning temperature used. With some caution, the relative error for any given temperature can be inferred for thermocouple types where the Seebeck coefficient remains approximately constant with temperature (thus, $\Delta S/S$ is similar to $(\Delta T/T)$, where T is temperature in $^{\circ}\text{C}$) [10]. The calculated % change in Seebeck coefficient is based on the *average* measured Seebeck coefficient, $\bar{S}(x)$ occurring over the gradient region of the homogeneity scanner and \bar{S}_{ref} , the Seebeck coefficient calculated from the reference function [13]. The temperature spatial resolution of the steam heat-pipe scanner is approximately 5.5°C for a bare-wire thermocouple in a 4 mm alumina sheath. The resolution of the scanner, the linearity of the gradient furnace, and the derivation of $\bar{S}(x)$ are discussed in further detail in [10,14].

$$\text{Relative Change Seebeck}(\%) = \frac{\bar{S}(x) - \bar{S}_{ref}}{\bar{S}_{ref}} \%$$

As shown in Fig. 3a–c, there is a clear correlation between strain and relative changes to the seebeck coefficient. Changes of 0.4 %, 0.6 % and 1 % occurred, respectively, after 5 %, 10 % and 20 % linear strain, which is similar to values reported elsewhere [5]. Figure 3a–c reveals that as the amount of cold-work strain is increased, the temperature at which SRO becomes active decreases, similar to the findings of Kollie et al. [15]. Partial thermal recovery from cold-working can start at temperatures well below 200°C , in agreement with observations made in a recent study [3], but not necessarily shown in earlier studies [7,9]. For 20 h of exposure to temperatures above the ‘peak SRO’ value at around 400°C – 450°C , the relative seebeck coefficient is consistent regardless of strain applied, and the value of this ‘peak’ is similar to those reported by Bentley for unstrained Type K elements [16]. Figure 3d shows there was good repeatability when using the tensional loading apparatus and gradient furnace, with changes in Seebeck coefficient typically within 0.05 % of each other, even after long

periods of temperature exposure. After 20 h of exposure, the Alumel was replaced to check for any artefacts introduced into the measurement. It is clear that the changes in Seebeck coefficient above are dominated by the strained Chromel wire.

Figure 4a–c decouples the SRO in thermally exposed wires regardless of the amount of cold-working applied in K+ wire to highlight the effects of applied cold-working on the Seebeck coefficient. By comparing strained and unstrained portions of wire for a fixed time period, the thermal recovery rate can be investigated as the strain is increased, in order to quantify the magnitude of the cold-work effect. Firstly, regardless of the amount of strain applied the effects of SRO and cold-work are separate, and thermal recovery from cold-work to a consistent state is completed by around 500 °C regardless of the amount of strain applied, and this supports findings made by Fenton [9]. It should be noted, however, that this state is *not* the AR state; hence, there is no evidence of the 50 mm unstrained section of wire as it is exposed to temperatures above 500 °C. Below around 500 °C, however, the different thermocouple compositions (as shown in Table 1) display different thermal recovery rates. It appears that compositional variation in the Type K wires plays a role in the strain sensitivity of the thermal recovery. The M8 K+ wire for example clearly has a poorer thermal recovery when the strain is increased beyond 10%.

When comparing the effects of strain and thermal recovery between different manufactures of wire, one needs to be aware that manufacturing procedures can leave behind residual cold-work (RCW). In some cases, the wire can be in an extremely work hardened state leading to large inhomogeneities when strained. For example, the M4 K+ wire could not be strained more than ~15% before failure, unlike any of the other wires which could be strained to beyond 20%, suggesting that a higher proportion of RCW was present. Applying strain below 10% showed no repeatability and sometimes the relative change in Seebeck coefficient dropped as low as –25%. This effect is due to ‘necking’ of the wire at locations weakened by the drawing process.

3.2 Cold-Worked Hoskins Alumel (Procedure 1.2)

Cold-working tests on Hoskins Alumel strained to 5%, 10% and 20%, had a negligible effect the inhomogeneity or deviations from the reference function, as shown in Fig. 5. The amount of strain applied had little effect on the inhomogeneities developed as a function of temperature when aged in the GF. These results are in good agreement with previous studies, which also showed Alumel to be far less sensitive to cold-working than Chromel; see, for example, [7]. To obviate the effects of Chromel GF aging in the Alumel, a new section of AR Chromel wire was paired with the GF aged Alumel. However, as the strain-induced deviations in the Alumel wire were small, some of these changes were masked by apparent variations in the AR Chromel, which are suspected to be caused by variations in RCW. This variation prevents substantial claims about the effect of cold-working on the homogeneity of Alumel wire. In any case, the drift due to GF aging in Alumel is also small and leads to a negative trough at about 300 °C, regardless of the amount of cold-working. Tests on K– wire from other manufacturers are not presented here as the results were similar to those of the Hoskins Alumel sample. The conclusion from this section of work also indicates that

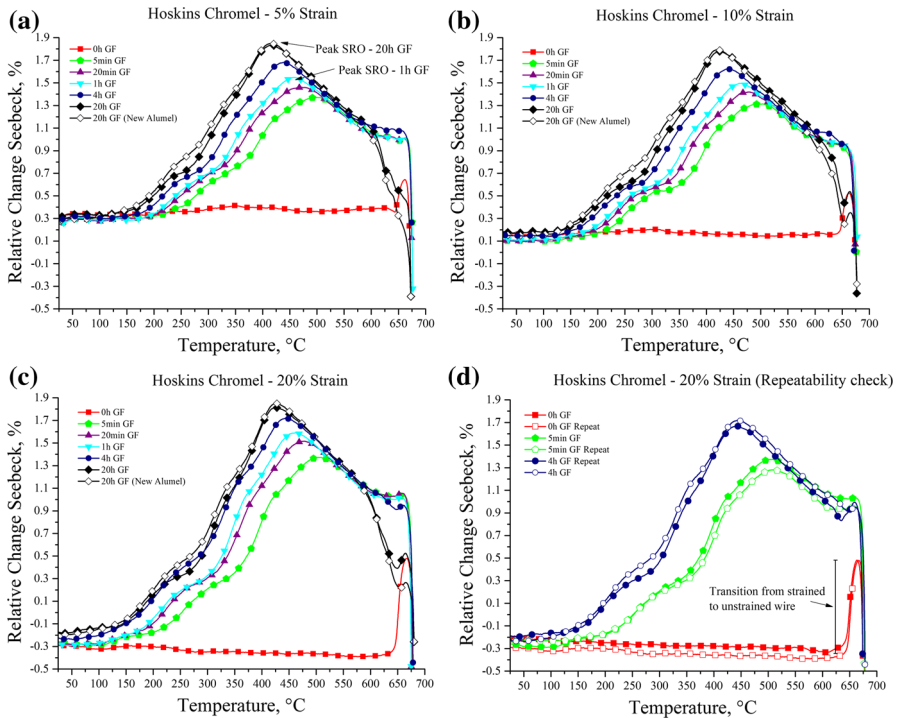


Fig. 3 Plots showing the thermal recovery of K+ wire sourced from Hoskins at a variety of different strain rates

the effects reported on K+ wire in this study are nominally representative of Seebeck changes for the whole thermocouple.

3.3 Comparison of Linear to Nonlinear Cold-Working (Procedure 2)

The vast majority of studies employ a method of cold-working to thermocouples that involves a combination of bending or swaging [5, 12]. This process is normally adopted because it replicates the application of cold-working during manufacturing or normal use; however, this is difficult to quantify due to the nonlinear nature of the forces applied in these processes. Fenton suggested that wrapping a 0.5 mm conductor around a 1.5 mm mandrel produced the same amount of cold-working as 10 % to 20 % strain, but did not provide any evidence [9]. In this study, the claims by Fenton were replicated along with a larger 5.2 mm mandrel winding with the Hoskins Chromel as shown in Fig. 6.

The inhomogeneities caused by winding the wires around 1.5 mm and 5.2 mm mandrels compare remarkably well with the 5 % and 20 % strained pieces of wire, respectively. This is a key result, as it highlights that the application of strain is quantifiable and analogous to bending, which often occurs during typical usage of Type K thermocouples.

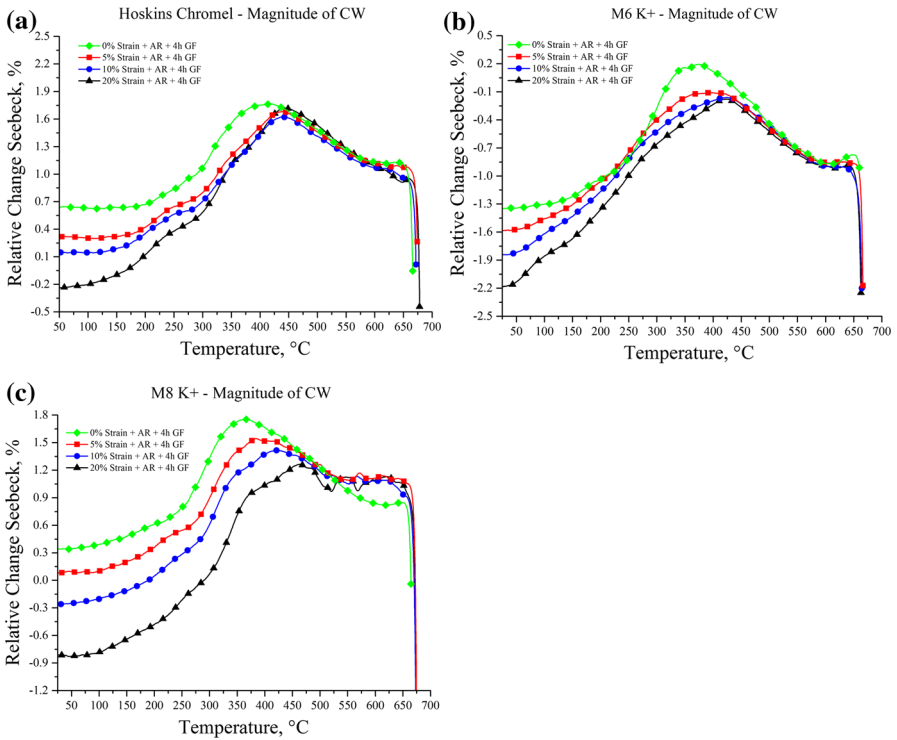


Fig. 4 ‘Cold-work effect’ for the different manufacturers of K+ wire

Strain to Hoskins Alumel Wire

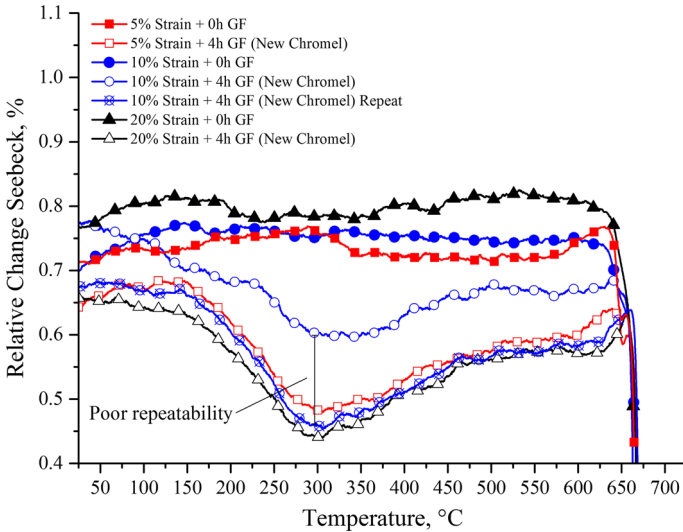


Fig. 5 Thermal recovery in Alumel, after 4 h annealing at 5%, 10% and 20% strain

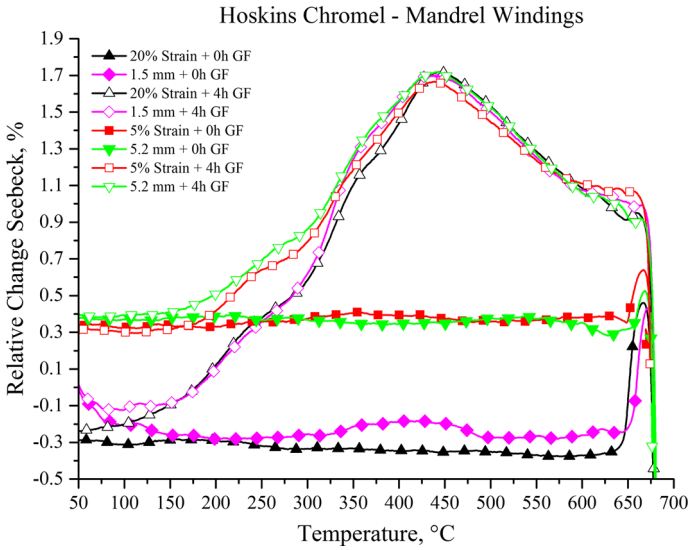


Fig. 6 Linear and nonlinear cold-working comparison with Hoskins Chromel wire

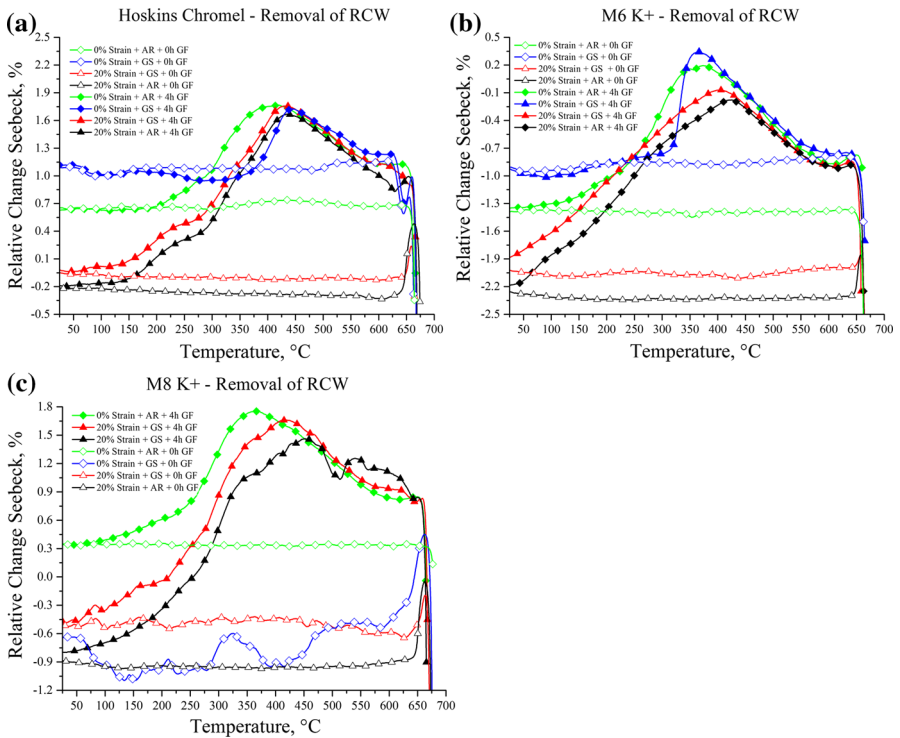


Fig. 7 Removal of RCW for three of the manufacturers

3.4 Heat Treatment of K+ Wires to Remove Residual Cold-Work (Procedure 3)

In order to investigate the thermal recovery process independently from residual cold-work (RCW), especially between manufacturers, the Chromel/K+ thermoelements are placed in the 'ground state' (GS) as part of procedure 3 (described in earlier sections). A short annealing time is used limiting the oxidation of the bare elements. This technique allows an assessment of the RCW introduced into the thermoelements by the manufacturer in the as-received state (AR). Additionally, information on the rate of thermal recovery can be investigated for thermoelements containing 20 % strain, when in either the AR or the GS condition. Figure 7a–c shows the difference in K+ thermoelements after 4 h GF aging in either the AR or GS and when there has been either no applied strain or after 20 % strain. The differences in behavior are attributed to variations in composition (Table 1). The Hoskins and M6 K+ wires have similar responses to GS anneals and the subsequent thermal recovery. The M8 wire on the other hand could not be reliably annealed into the GS, as shown in Fig. 7c. This could be due to the increased quantity of iron in the M4 K+ wire, as changes in the lattice structure of iron at around 900 °C cause the iron to contract [16], which could induce nonuniform strain along the wire. This would go some way to explaining why then applying a uniform strain to the M8 wire appeared to 'smooth' out the inhomogeneities left by the GS anneal. The differences in Seebeck coefficient between the AR and GS for these wire samples, after 20 % strain, are likely due to the formation of oxides during the GS anneal. Comparing strained thermoelements in the AR and GS annealed states, up to 5 % cold-working is left over from the manufacturing process, also observed by Potts and McElroy [5]. Any additional strain applied by the tensional loading apparatus appears to be additive to any RCW in the wires.

4 Conclusions

A number of experiments have been carried out, involving the application of varying amounts of cold-work strain in a controlled, quantifiable fashion to identify the effects of thermal recovery in Type K thermoelements, specifically the K+ element. The key findings are identified below:

1. Thermal recovery from any amount of applied strain to K+ wires, by *manual* cold-working (i.e., not swaging) is achieved at around 500 °C, regardless of the composition. This state, albeit consistent with and without strain applied, is not the same as the AR state before thermal exposure has taken place.
2. Below 500 °C, relatively small variations in wire composition influence the rate of thermal recovery and the threshold temperature at which thermal recovery starts.
3. Further strain applied to K+ wires containing an excessive amount of residual cold-work, generated during manufacturing, leads to highly inconsistent changes in the Seebeck coefficient, regardless of the wire composition. In some instances, these changes could be as large as 25 %. Additionally, for these wires physical yield failure also occurred at far lower strains.
4. Claims made by Fenton [9] about the equivalences of applying cold-working, either by using mandrels or by applying linear strain, have been replicated in this

study, thus suggesting that the method of applying strain does not play a significant role in the creation of defects within the thermoelements.

5. Any additional cold-working applied on top of residual cold-work in the wires appears to simply be additive.

It is hoped the findings presented here can help inform users of Type K thermocouples wire about the potential impact of applying cold-working to portions of thermocouples that contribute to the signal (i.e., occupy a temperature gradient), at low (< 500 °C) temperatures. The changes in the Seebeck coefficient can be of such a magnitude that the thermocouples can be unknowingly taken outside of their as-supplied tolerances. There is also value demonstrated in preconditioning Type K thermocouples with an unknown history at a temperature of around 500 °C, which places the thermocouples in a known state and thus a suitable condition to calibrate Type K thermocouples. This is discussed in further detail by Webster [17].

Acknowledgements This work was carried out during a 2 month secondment by Adam Greenen to the Measurement Standards Laboratory of New Zealand (MSL). Adam acknowledges funding from the UK National Measurement System, the NPL International Secondments Scheme and MSL in order to make this secondment possible. The authors would also like to acknowledge the efforts of Jonathan Pearce in regard to facilitation the secondment, and to MSL for being excellent hosts during Adam's secondment.

References

1. Hoskins Manufacturing Company, *Hoskins Chromel-Alumel Thermocouple Alloys, Catalog M-61* (Hoskins Mfg. Co. Detroit, Mich., 1961)
2. N.A. Burley, R.M. Hess, C.F. Howie, J.A. Coleman, The nicrosil versus nilsil thermocouple: a critical comparison with the ANSI standard letter-designated base-metal thermocouples. *Temp. Meas. Control Sci. Ind.* **6**, 1159–1166 (1982)
3. E.S. Webster, Low-temperature drift in MIMS base-metal thermocouples. *Int. J. Thermophys.* **35**, 574–595 (2014)
4. D.D. Pollock, D.I. Finch, The effect of cold-working upon thermoelements. *Temp. Meas. Control Sci. Ind.* **4**, 237 (1962)
5. J.F. Potts Jr., D.L. McElroy, The effect of cold working, heat treatment, and oxidation on the thermal emf of nickel-base thermoelements. *Temp. Meas. Control Sci. Ind.* **4**, 243–264 (1962)
6. W.P. White, The constancy of thermoelements. *Phys. Rev. (Series I)* **23**, 449–474 (1906)
7. M. Campari, S. Garribba, The behavior of Type K thermocouples in temperature measurement: the chromel P-alumel thermocouples. *Rev. Sci. Instrum.* **42**, 644–653 (1971)
8. R.W. McCulloch, J.H. Clift, Lifetime improvement of sheathed thermocouples for use in high-temperature and thermal transient operations. *Temp. Meas. Control Sci. Ind.* **6**, 34 (1982)
9. A.W. Fenton, The travelling gradient approach to thermocouple research. *Temp. Meas. Control Sci. Ind.* **5**, 1973–1990 (1972)
10. E.S. Webster, D.R. White, H. Edgar, Measurement of inhomogeneities in MIMS thermocouples using a linear-gradient furnace and dual heat-pipe scanner. *Int. J. Thermophys.* **36**, 444–466 (2015)
11. D.R. White, R.S. Mason, A thermocouple homogeneity scanner based on an open pressure-controlled water heatpipe. *Int. J. Thermophys.* **31**, 1654–1662 (2010)
12. P. Pavlasek, C.J. Elliott, J.V. Pearce, S. Duris, R. Palencar, M. Koval, G. Machin, Hysteresis effects and strain-induced homogeneity effects in base metal thermocouples. *Int. J. Thermophys.* **36**, 467–481 (2015)
13. ASTM, *Standard Specification and Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples*, (ASTM International, 2012)
14. E. Webster, D.R. White, Thermocouple homogeneity scanning. *Metrologia* **52**, 130 (2015)

15. T.G. Kollie, J.L. Horton, K.R. Carr, M.B. Herskovitz, C.A. Mossman, Temperature measurement errors with Type K (Chromel vs. Alumel) thermocouples due to short-ranged ordering in Chromel. *Rev. Sci. Instrum.* **46**, 1447–1461 (1975)
16. R.E. Bentley, *The Handbook of Temperature Measurement Vol. 3: Theory and Practice of Thermoelectric Thermometry* (Springer, Singapore, 1998)
17. E.S. Webster, Thermal preconditioning of MIMS Type K thermocouples to reduce drift. *Int. J. Thermophys.* **38**, 5 (2016)