

## Limits of noble and base metal thermocouples

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### Abstract

In the following paper we will focus on one of the most frequently used temperature sensors, which are thermocouples. As these sensors are used in a variety of applications reaching from simple industrial ones up to aerospace measurements the knowledge of their behaviour and limitations is of high importance. Within this paper we will be focusing on both base and noble metal thermocouples which have their specific areas of use. The emphasis of the presented studies is on the hysteresis and homogeneity effects within base metal type K and N thermocouples and on the effect of new construction of noble metal type Au/Pt thermocouples.

### Introduction

Temperature measurement directly affects the quality, effectiveness and safety of industrial manufacturing processes. In many of these processes base metal thermocouples are used for monitoring and control. Because of their wide use as a temperature sensor, it is necessary to understand their behaviour under various influences and in different conditions. Two factors that contribute considerably to the uncertainty in temperature measurement using such sensors are the hysteresis effect and the homogeneity effect: many publications such as [1-11] have shown the need for further investigation into these effects.

For this investigation the two commonly used Type N and Type K thermocouples were investigated. The typical composition of the alloys should be consistent but it can vary by manufacturer. The thermocouples were all mineral insulated metal sheathed (MIMS) format. Strain measurements were performed on thermocouples of diameter 1.0 mm and 1.5 mm, whilst hysteresis measurements on 3.0 mm diameter thermocouples. In all cases the sheath material was Inconel 600. The overall objective of this part of the study is to better quantify these effects to enable realistic values to be included in uncertainty budgets for this type of thermocouple.

The problematic of noble metal thermocouples is more related to high precision measurements and therefore the investigation focuses on a different aspects. According to the International Temperature Scale of 1990 (ITS-90), HTSPRT are used as interpolation instruments in the temperature range between the freezing points of aluminium (660.323 °C) and silver (961.78 °C) [12]. Their sensitivity to contamination, lack of stability, and poor repeatability are well-known problems [13-15]. Au/Pt thermocouples are considered to be an alternative to the HTSPRTs in this temperature range. As further improvements in manufacturing processes of Au/Pt thermocouples have been made, an increase of their thermoelectric stability and homogeneity has been achieved. This has resulted in excellent measuring properties which allow reproducible measurements of high accuracies which are better by about one order of magnitude compared with Pt/Rh alloyed thermocouples. The task group for the mise en pratique for the definition of the kelvin of the Consultative Committee for Thermometry (MeP-K, CCT) has recommended further research on Au/Pt thermocouples as a potential candidate for a new interpolation instrument within the ITS-90 for the temperature range between the aluminium and silver fixed points [11].

Two Au/Pt thermocouples constructed by using only quartz glass insulation materials were investigated. The quartz glass tubes had a higher purity of 99.995% compared to typical purities of 99.7% of alumina. Furthermore, quartz glass insulation tubes were used to have a smoother surface of the inner holes of the insulation tubes compared to alumina tubes allowing the easier movement (caused by thermal expansion) of the thermoelements. In this way the creation of stress relieving coil in hot junction which is normally used to compensate the effect of the different expansion coefficients of gold and platinum is no longer required.

Instead, the unhindered movement of the thermoelements inside the smooth quartz glass tubes avoids the formation of mechanical stress in the thermoelements. The Au/Pt thermocouples marked Au/Pt 12-01 and Au/Pt 13-03 were made of pure platinum wire (99.997 %) supplied by Alfa Aesar. The diameter of the wire was 0.5 mm and its length was 2 m. The gold wire for Au/Pt 12-01 had a nominal purity of 99.999% and was supplied by M&K GmbH. The gold wire for Au/Pt 13-03 had an unknown purity and was supplied by Berliner Metallhüttenund Halbwerkzeuge (BMHW). Both gold thermoelements had a diameter of 0.5 mm with a length of 2 m.

### **Measuring procedures**

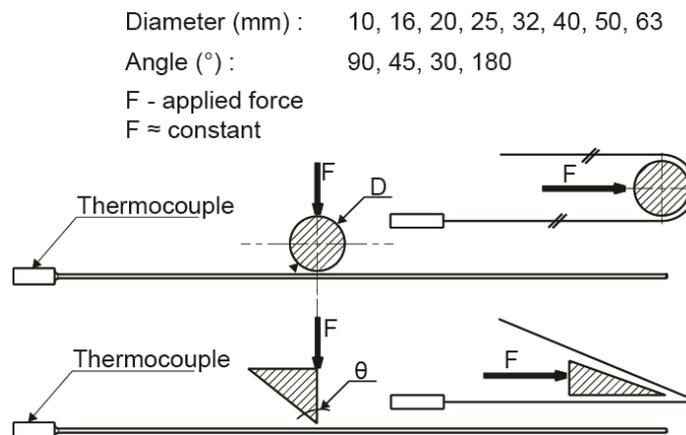
The hysteresis effect was investigated for Type K and Type N base metal thermocouples (, three of each type). These were 700 mm in length and 3.0 mm in diameter. As the hysteresis is caused by changes in the Seebeck coefficient, the dimensions of the thermocouples do not have any influence on the hysteresis [4]. A single zone horizontal tube furnace was used for the experiment. The furnace uniformity was measured and the tested thermocouples were positioned alongside each other in the furnace, with their measurement junctions located at the centre ensuring that they experienced very similar thermal conditions. A low uncertainty Type R thermocouple was used as a reference to obtain the temperature in the vicinity of the measurement junctions of the tested Type N and Type K thermocouples.

Seven complete measuring cycles were performed, where each cycle typically lasted for more than 34 hours. This represents around 300 hours of exposure above 200 °C for each thermocouple. Each cycle consisted of initially heating the collection of thermocouples from 200 °C to 1000 °C, and then cooling back to 200 °C. Increasing and decreasing the temperature was completed in 50 °C temperature steps with one hour for stabilization at each temperature before commencing measurements (this process was automated to ensure the highest possible level of repeatability and comparability of the measured data).

For the homogeneity measurements four 800 mm long MIMS Type N and Type K thermocouples, with an outer diameter of either 1.0 mm or 1.5 mm were tested. The thermocouples each contained thermoelement diameters 0.18 mm (outer diameter 1.0 mm) and 0.27 mm (outer diameter 1.5 mm). One of each diameter and type of thermocouple was exposed to a series of bends around a circular and angular surface (values listed in Fig.1). These bends were applied in turn along the length of the thermocouple. All of the bends were separated from each other by a segment of the thermocouple on which no mechanical strain was applied. The scheme of the strain application and the used diameters and angles of bending are illustrated in Fig. 1. After each bend application, the thermocouples were then straightened and the homogeneity re-measured. To determine the effect of mechanical strain on these thermocouples, the initial state of their homogeneity was determined. This was measured by the one-gradient method which was realized by immersing the thermocouple in an isothermal stirred oil bath and then incrementally raising it via a linear rail system. The oil temperature was set to 150 °C, high enough to see the changes in homogeneity along the thermocouple but low enough so as not to introduce any thermally generated inhomogeneity.

The possible effect of horizontal and vertical positions on the electromotive force (emf) was performed on two Au/Pt thermocouples. Furthermore, a calibrated Au/Pt thermocouple with alumina inner insulation made of the Standard Reference Material® (SRM®) 1749 [16] was used as a reference. The investigations were performed in the temperature range from 600 °C to 950 °C. The thermocouples' homogeneity, as one of the most important uncertainty contributions affecting their accuracy, was measured, before starting the position testing and fixed point measurements, after the measurements in horizontal position and at the end after the vertical position measurements. In addition fixed-point measurements at the freezing points of Al and Ag were performed before and after the position testing to check the thermoelectric stability of the Au/Pt thermocouples. The homogeneity measurements were performed in a salt bath at a stable temperature of 300 °C and over a length of 16 cm of the thermocouples. Additional homogeneity scanning procedure was performed at the freezing

point of silver. The homogeneity was measured by withdrawing the fully emerged thermocouple from the fixed-point cell. The thermoelectric homogeneity was measured over a length of 12 cm with steps of 2cm.



**Fig. 1** Illustration of the bending procedure using circular (upper panel) and angular (lower panel) surfaces together with the values of diameter and angle applied.

Further measurements using an additional Pt20%Rh outer protection tube, in which the thermocouples Au/Pt 12-02 and Au/Pt 13-03 were inserted successively, have been performed in the two furnaces (in vertical and horizontal position) and at fixed points. The purpose of this protective tube was to create a typical measuring condition in which this tube is used for contamination protection. The final investigation was focused on the leakage effect possibly caused by the inner quartz glass insulation tube. This investigation was performed by repeated measurements at the fixed points of Zn, Al and Ag using Au/Pt thermocouples constructed with quartz glass inner insulation tubes and furthermore repeated after the insulation replacement for alumina inner insulation tubes.

### **Summary**

Homogeneity measurements show that a small impact on the homogeneity of Type K thermocouples (up to 0.09 °C) is measurable. Type N thermocouples, on the other hand, are found to be insensitive to the effects of mechanically induced strain through bending (no effect was measurable). This test provides a useful method to inform a practical uncertainty budget, when bending of thermocouples is performed and therefore must be accounted for in the measurement uncertainty. The measured hysteresis show similar behaviour as in publications [17-21] with slight differences in the temperature range where the hysteresis occurs. This is most likely caused by the different source of material used for the thermocouple thermoelements. The hysteresis tests shown a tendency for both types to give a repeatable deviation of up to 4 °C from the reference function but the measurement uncertainty has to be taken into account.

The investigation of the Au/Pt thermocouples which were made by using solely quartz glass as an insulation material exhibited considerable deficiencies when used at temperatures higher than about 650 °C (independent from measuring position). This unsatisfying performance is suspected to be caused by the reduced insulation resistance of the single thin walled quartz glass tubes used. Taking into account the different wall thickness, the alumina insulation tube maintains its higher resistance at a value of  $8.5 \times 10^7 \Omega$  at 1000 °C in comparison to quartz glass which only has a resistance of  $3.6 \times 10^7 \Omega$  at the same temperature [22]. This is furthermore supported by the measuring results from Table 1 where calibration in fixed point with quartz glass insulation and alumina insulation replacement was done. If it were possible to use quartz glass tubes with thicker walls resulting in higher insulation resistance, the benefit of the unhindered movement of the thermoelements in the smooth quartz glass holes might compensate effects of the reduced insulation resistance found in this work.

**Table 1** Emfs measured at fixed points when using the Au/Pt thermocouples 12-01 and 13-03 with quartz glass and alumina insulation tubes.

Fixed point	Au/Pt 12-01				Au/Pt 13-03			
	emf / $\mu\text{V}$ (Quartz)	emf / $\mu\text{V}$ ( $\text{Al}_2\text{O}_3$ )	$\Delta\text{emf}$ / $\mu\text{V}$	$\Delta t$ / $^\circ\text{C}$	emf / $\mu\text{V}$ (Quartz)	emf / $\mu\text{V}$ ( $\text{Al}_2\text{O}_3$ )	$\Delta\text{emf}$ / $\mu\text{V}$	$\Delta t$ / $^\circ\text{C}$
<b>Ag</b>	16084.9	16091.7	6.8	0.273	16101.6	16113.1	11.5	0.462
<b>Al</b>	9299.3	9300.6	1.3	0.065	9312.6	9315.0	2.4	0.119
<b>Zn</b>	4933.2	4934.0	0.8	0.050	4942.6	4943.0	0.4	0.025

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