National Physical Laboratory

NPL REPORT MAT 110

ETMT DIGITAL CALIBRATION FOR TEMPERATURE AND RESISTIVITY

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AUGUST 2022

Department for Business, Energy & Industrial Strategy

FUNDED BY BEIS

ETMT Digital Calibration for Temperature and Resistivity

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ABSTRACT
An exercise was conducted to establish uncertainties in measurement practice for temperature and resistivity in dilute Ni alloys using the ETMT system, thus providing a digital route for underpinning applications using the ETMT instrument.

ISSN 1754-2979

DOI ADDRESS: https://doi.org/10.47120/npl.MAT110

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This work was funded by the UK Government's Department for Business, Energy and Industrial Strategy (BEIS) through the UK's National Measurement System programmes.

Approved on behalf of NPLML by
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CONTENTS

1 INTRODUCTION

The NPL ETMT system allows miniature testpieces of conductive materials to be examined for their mechanical and electrical properties as a function of temperature. Interpretation of the temperature dependence of properties can be enhanced by measuring the electrical resistivity as a function of temperature, where phase or magnetic transformations are well known. This report examines some of the calibration issues with a view to providing digital data regarding current, voltage and temperature with a supporting understanding of uncertainties. In due course it is anticipated that this calibration will be used by Instron for digital circulation to current and future users of the Instron ETMT. The exercise was conducted on three dilute Ni alloys coded Ni 201, Ni 125 and Ni 541. EDM/Diamond Ground ETMT testpieces, of nominal dimensions of 40 x 2 x 1 mm, were extracted from a larger alloy piece by NPL workshop.

1.1 MATERIALS FOR CALIBRATION STUDIES

The compositions of the Ni alloys were:

Ni 201: Nominally pure Ni (99%); Low carbon version of Ni 200.
Ni 125: Al (1 to 2%); Si (1 to 2%); Y (0.1 to 0.2%); Balance Ni
Ni 541: Al (2%); Cr (2%); Si (2%); Balance Ni

The rationale for investigating these three alloys is that Ni 201 has a well defined Curie point, Ni 125 is a dilute Ni alloy but with a different Curie point and Ni 541, also a dilute alloy, has a smooth resistivity curve up to 800 °C without a Curie Point. These differences were thought to be useful for highlighting variations in behaviour.

1.2 RT RESISTIVITY

The room temperature resistivity of each alloy testpiece was measured using a NPL internal procedure [1] in which a constant current of 1 A is passed through the sample in both directions to obtain a mean value of the voltage drop over a length of 4.2 mm (between two needle points) measured with a sensitive digital voltmeter. The results for ten samples of each alloy are shown in Table 1 (Ni 201). Table 2 (Ni 125) and Table 3 (Ni 541). The individual values can be ranked and the rank data is shown in Figs 1, 2 and 3 for Ni 201, Ni 125 and Ni 541 respectively. Rank provides some information on the uniformity of composition.

Table 1 RT resistivity of Ni 201

Ni 201	Length L	width w	Thickness t	Current, I	pd sep	mV	Resistivity	Conductivity	
	mm	mm	mm	A	mm	mean	nohm.m	MS m^{-1}	%IACS
ETMT									
DRNi201a	40.00	2.00	0.98	1.00	4.20	0.200	93.3	10.71	18.44
DRNi201b	40.00	2.00	0.99	1.00	4.20	0.192	90.5	11.05	19.02
DRNi201c	40.00	1.99	0.99	1.00	4.20	0.192	90.1	11.10	19.11
DRNi201d	40.00	2.00	1.00	1.00	4.20	0.202	96.2	10.40	17.89
DRNi201e	40.00	2.00	0.97	1.00	4.20	0.197	91.0	10.99	18.91
DRNi201f	40.00	1.97	0.98	1.00	4.20	0.194	89.2	11.21	19.30
DRNi201g	40.00	2.00	0.99	1.00	4.20	0.202	95.2	10.50	18.07
DRNi201h	40.00	2.00	0.99	1.00	4.20	0.201	94.8	10.55	18.16
DRNi201i	40.00	2.00	0.98	1.00	4.20	0.197	91.9	10.88	18.72
DRNi201j	40.00	2.00	0.96	1.00	4.20	0.196	89.6	11.16	19.21

Table 2 RT resistivity of Ni 125

Ni 125	Length L	width w	Thickness t	Current, I	pd sep	mV	Resistivity	Conductivity	
	mm	mm	mm	A	mm	mean	nohm.m	MS m^{-1}	%IACS
ETMT									
DRNi125a	40.00	1.98	0.99	1.00	4.20	0.452	211	4.74	8.16
DRNi125b	40.00	1.99	1.00	1.00	4.20	0.458	217	4.61	7.93
DRNi125c	40.00	1.98	1.00	1.00	4.20	0.447	211	4.75	8.17
DRNi125d	40.00	1.98	0.99	1.00	4.20	0.461	215	4.65	8.00
DRNi125e	40.00	1.98	0.99	1.00	4.20	0.453	211	4.73	8.14
DRNi125f	40.00	1.99	0.98	1.00	4.20	0.456	212	4.72	8.13
DRNi125g	40.00	1.99	1.00	1.00	4.20	0.461	218	4.58	7.88
DRNi125h	40.00	1.98	0.99	1.00	4.20	0.457	213	4.69	8.07
DRNi125i	40.00	1.99	0.99	1.00	4.20	0.473	222	4.51	7.76
DRNi125j	40.00	1.99	0.98	1.00	4.20	0.459	213	4.69	8.08

Table 3 RT resistivity of Ni 541

Ni 541	Length L	width w	Thickness t	Current, I	pd sep	mV	Resistivity	Conductivity	
	mm	mm	mm	A	mm	mean	nohm.m	MS m^{-1}	%IACS
ETMT									
DRNi541a	40.00	1.99	1.01	1.00	4.20	1.036	496	2.02	3.47
DRNi541b	40.00	1.97	0.99	1.00	4.20	1.060	492	2.03	3.50
DRNi541c	40.00	1.98	0.98	1.00	4.20	1.064	492	2.03	3.50
DRNi541d	40.00	2.00	1.01	1.00	4.20	1.027	494	2.02	3.48
DRNi541e	40.00	1.99	1.00	1.00	4.20	1.039	492	2.03	3.50
DRNi541f	40.00	1.99	0.98	1.00	4.20	1.061	493	2.03	3.49
DRNi541g	40.00	1.99	1.00	1.00	4.20	1.046	496	2.02	3.47
DRNi541h	40.00	2.00	1.00	1.00	4.20	1.070	510	1.96	3.38
DRNi541i	40.00	1.99	0.99	1.00	4.20	1.056	495	2.02	3.47
DRNi541i	40.00	2.00	1.00	1.00	4.20	1.052	501	2.00	3.44

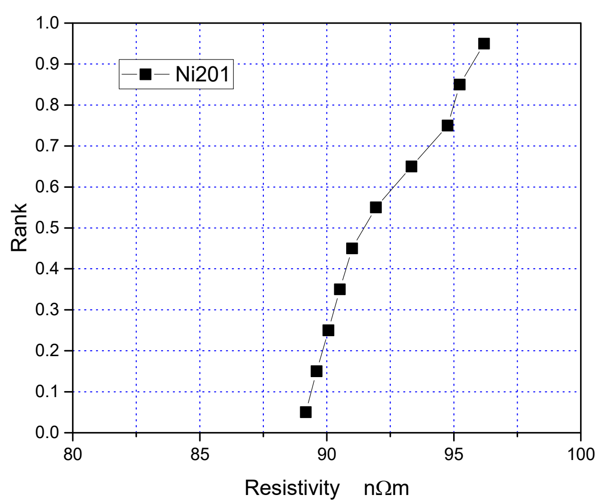

Figure 1 Rank for RT resistivities of Ni 201

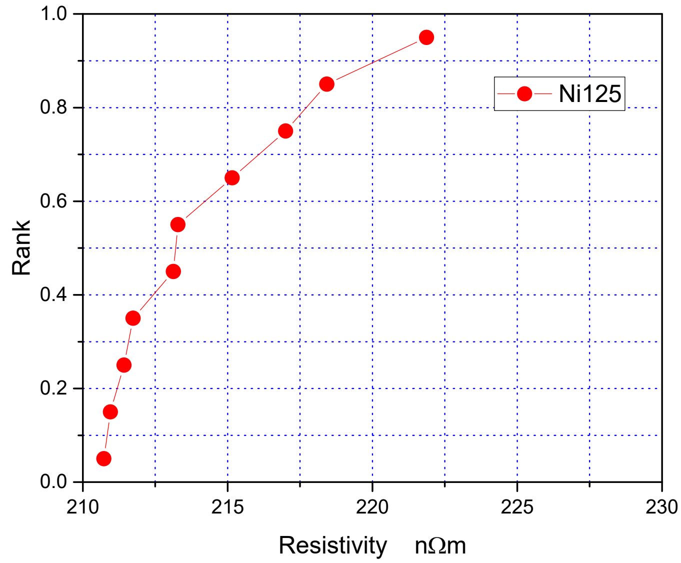

Figure 2 Rank for RT resistivities of Ni 125

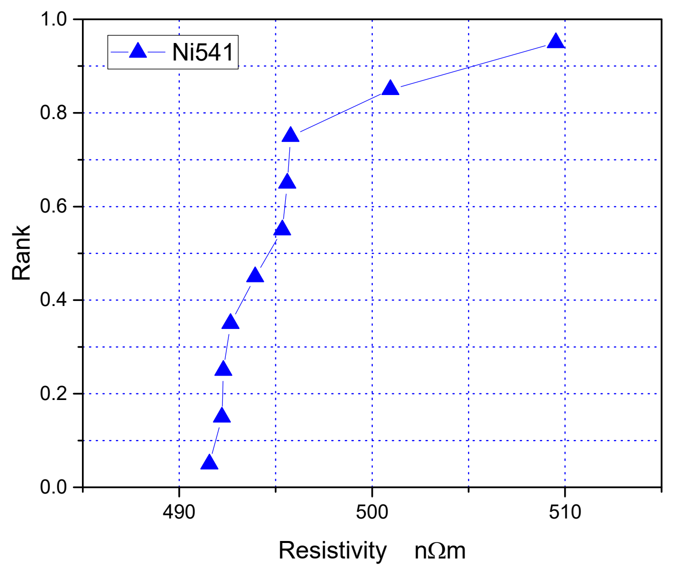

Figure 3 Rank for RT resistivities of Ni 541

The RT resistivity value is then used to calculate a length, L, over which voltage, V, is measured in the ETMT. Changes in size due to expansion on heating are small and are ignored in the calculation. In the ETMT, the temperature is increased by increasing the current, I, applied to the sample of cross sectional area A. The measured voltage across the length between the potential contacts increases as the testpiece gets hotter.

The measured voltage as a function of temperature then allows the resistivity, *R*, to be calculated from:

$$R = V\,A\,/\,I\,L$$

The uncertainty in resistivity at each individual measurement is about 1.2%, which largely arises in the measurement of the distance between the potential probes (estimated to be 1%), the dimensional measurements of the testpieces (0.5%) and the current and voltage measurements. The latter two values have uncertainties less than 0.1%. [1].

Detailed background information on the NPL ETMT system can be obtained from a NPL Good Practice Guide [2]. The following tests were performed on three testpieces of each alloy in the ETMT. The samples are held between grips that are moved apart to maintain zero load as the sample expands as it is heated by a direct current.

Heat to 800 °C at 2 °C/s

Cool to RT at 2 °C/s

During these cycles the following data was acquired and stored:

Time; Load; Temperature; system Grip Displacement; Current; and Voltage. Resistance is calculated from the latter two measurements.

2 NICKEL 201

Pure Ni has a handbook magnetic Curie point of 358 °C. The resistivity data in the first heating cycle on the first testpiece (coded Ni201a) is shown in Fig 4. The inflection in the curve corresponds to the Curie point. The region around the Ni201a Curie point of about 340 °C is shown expanded in Fig 5. The cooling curve is shown plotted with the heating curve in Fig 6 indicating excellent correspondence for the applied rates of 2 °C/s. The second heating and cooling cycles were equally consistent but are not plotted. The full resistivity plots on the first heating cycle for all three testpieces (N201a, b, and c) are shown in Fig 7 while the expanded region around the Curie point is shown in Fig 8.

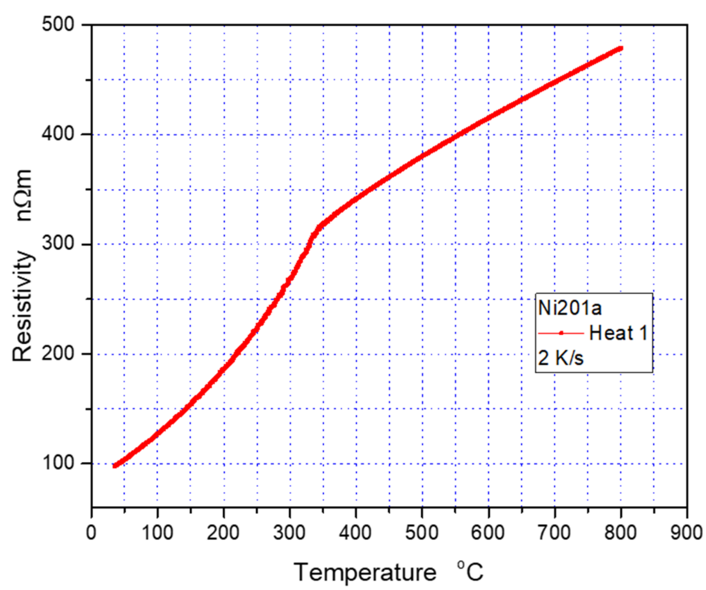

Figure 4 Temperature dependence of resistivity of Ni 201a

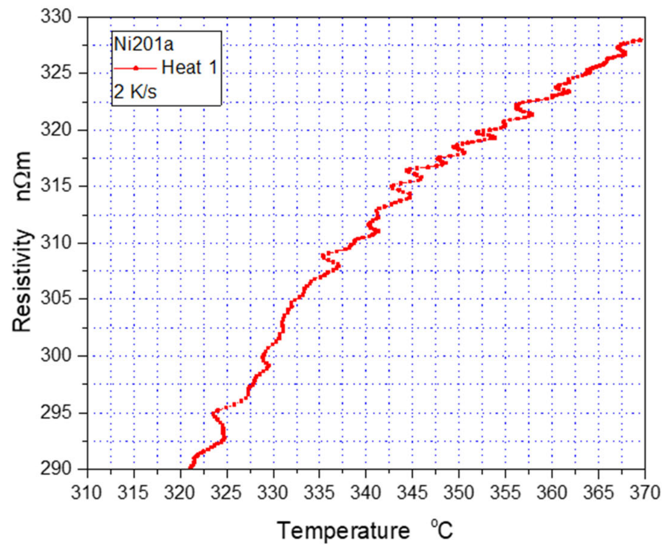

Figure 5 Resistivity around Curie Point of Ni 201a

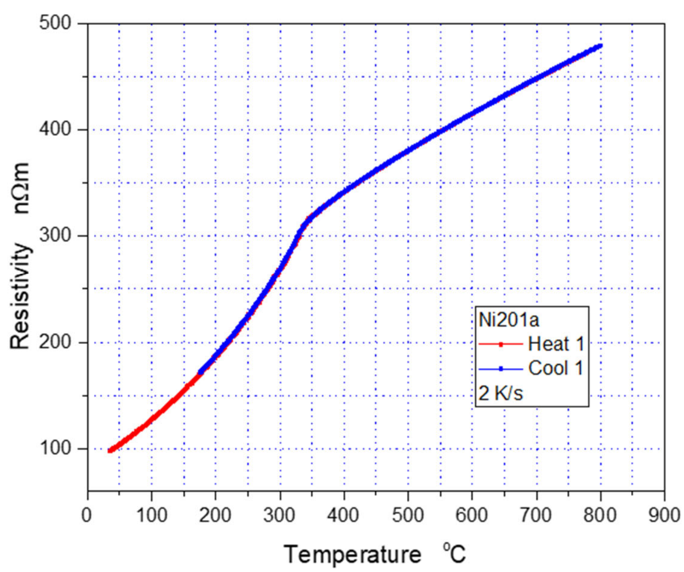

Figure 6 Resistivity on first heating and cooling cycle of Ni 201a

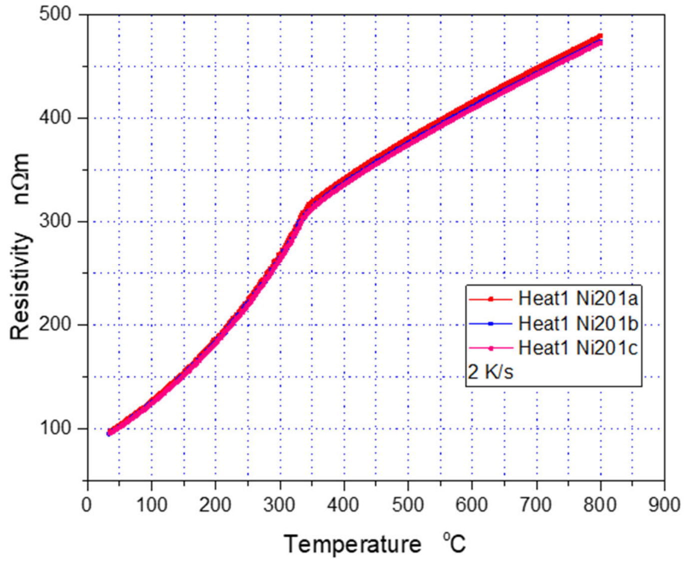

Figure 7 Resistivity on first heating cycle of Ni 201a, b and c

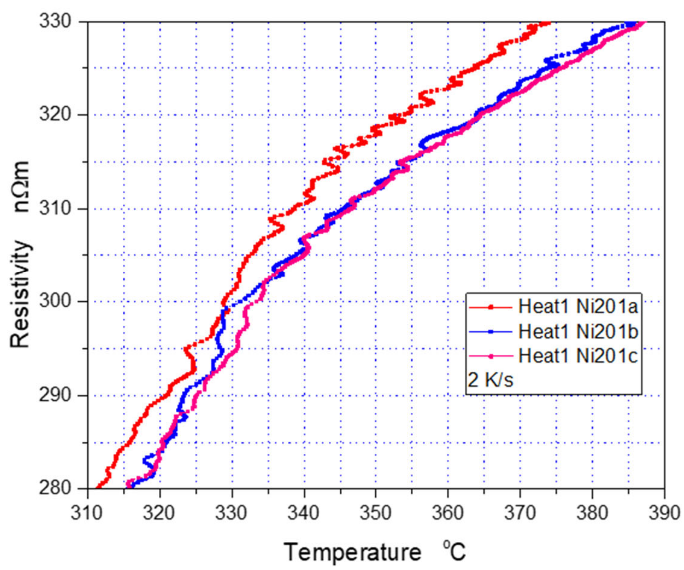

Figure 8 Resistivity around Curie Point in first heating cycle of Ni 201a, b and c

2.1 DIGITAL REPRESENTATION OF NICKEL 201 DATA

Two options are provided for the digital representation of the Ni 201 resistivity/temperature data using the test on Ni201a. One is to provide an excel sheet of the measurements with appropriate uncertainties, as discussed in the summary. The other is to fit a second order polynomial expressions to three parts of the data; i.e. RT to 300 °C (approaching the Curie point), 300 to 400 °C (around the Curie point) and 400 to 800 °C (in the non-magnetic region). The latter are shown in Fig 9 as black, blue and green traces respectively and are given as equations below which are plotted in red in Fig 9. The choice of segment temperatures is arbitrary based on the position of the inflection at the Curie point. The excel data can be obtained as a digital file if required.

The fit coefficients from the three parts described above can be summarized as follows, where Y is the resistivity value in nohm·m and X is the temperature in °C:

Ni 201a	$Y = 88.0 + 0.273\,X + 0.0011\,X^2$	Up to 300 °C
	$Y = 2.93\,X - 305.0 - 0.0033\,X^2$	300 to 400 °C
	$Y = 168.5 + 0.481\,X - 1.16E\text{-}4\,X^2$	400 to 800 °C

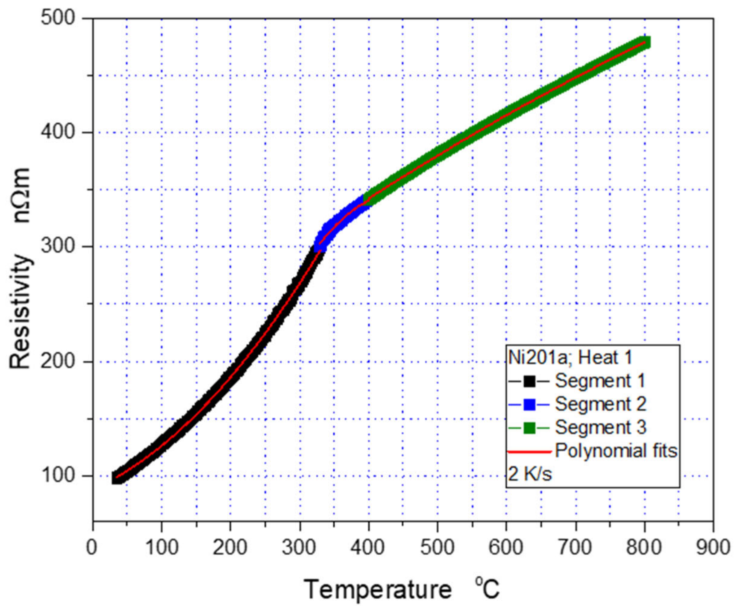

Figure 9 Segmented resistivity polynomial fits for Ni201a

3 NICKEL 125

The resistivity data in the first heating cycle on the first testpiece (coded Ni125a) is shown in Fig 10. The inflection in the curve corresponds to the Curie point. The region around the Ni125a Curie point of about 215 °C is shown expanded in Fig 11. The cooling curve is shown plotted with the heating curve in Fig 12 indicating good correspondence for the applied rates of 2 °C/s. The second heating and cooling cycles were equally consistent but are not plotted. The full resistivity plots on the first heating cycle for all three testpieces (N125a, b ,and c) are shown in Fig 13 while the expanded region around the Curie point is shown in Fig 14.

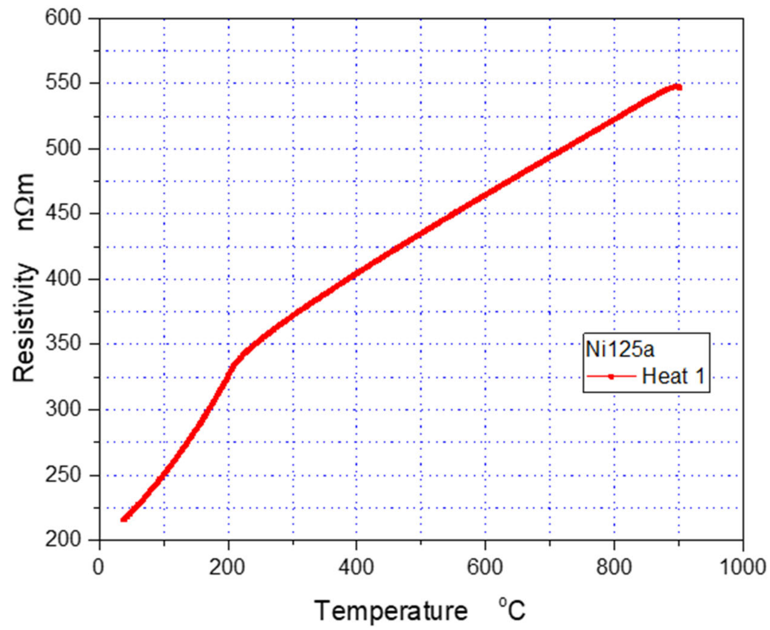

Figure 10 Temperature dependence of resistivity of Ni 125a

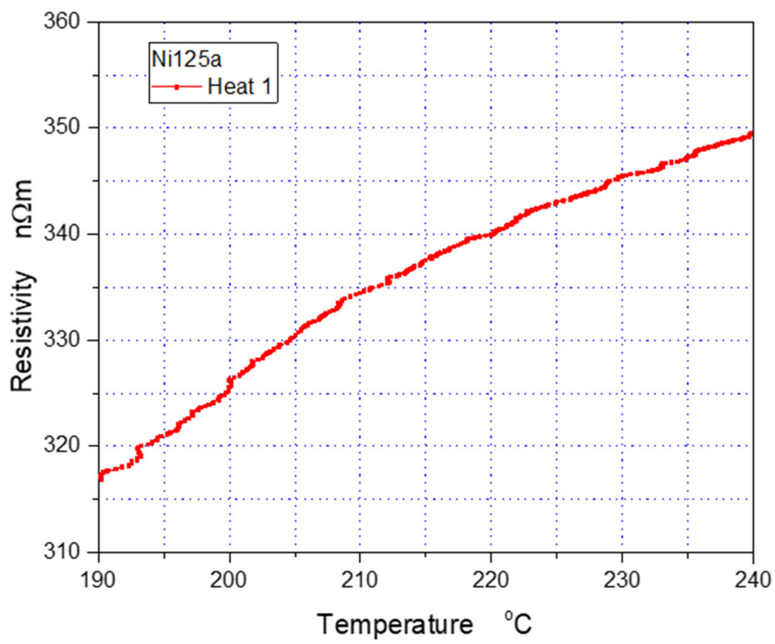

Figure 11 Resistivity around Curie Point of Ni 125a

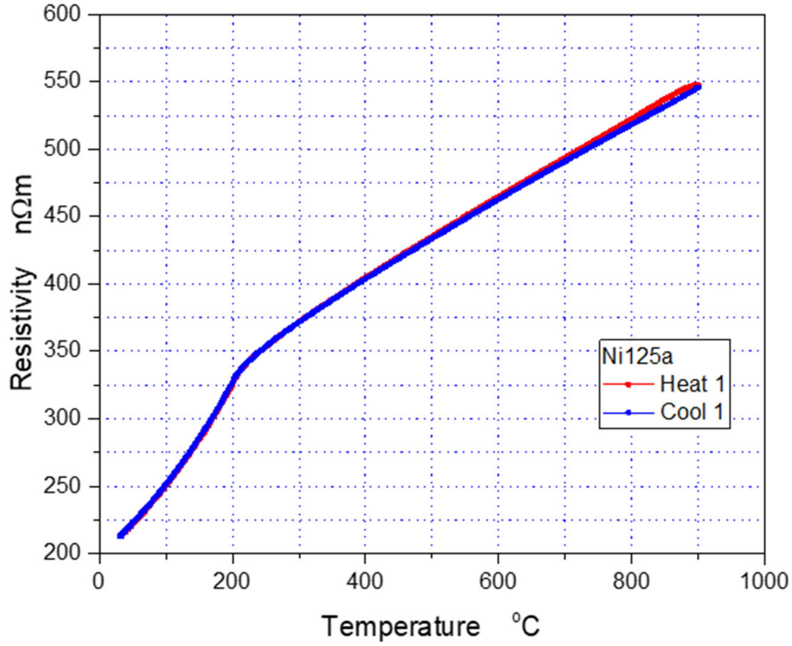

Figure 12 Resistivity on first heating and cooling cycle of Ni 125a

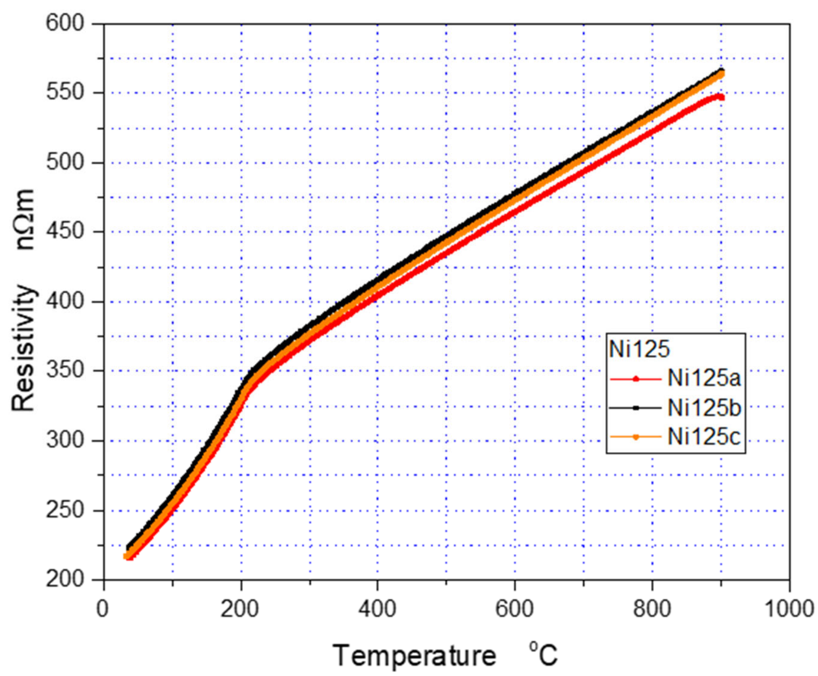

Figure 13 Resistivity on first heating cycle of Ni 125a, b and c

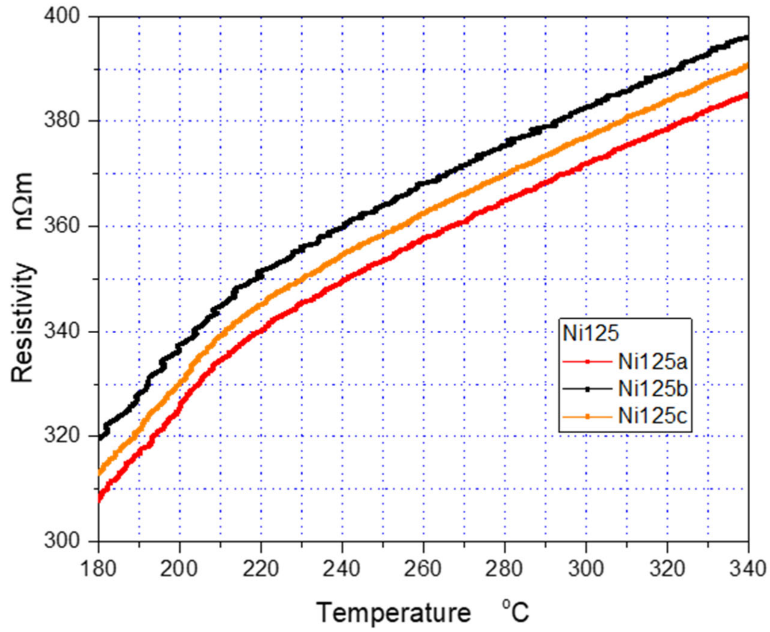

Figure 14 Resistivity around Curie Point in first heating cycle of Ni 125a, b and c

3.1 DIGITAL REPRESENTATION OF NICKEL 125 DATA

Two options are provided for the digital representation of the Ni 125 resistivity/temperature data using the test on Ni125a. One is to provide an excel sheet of the measurements with appropriate uncertainties, as discussed in the summary. The other is to fit second order polynomial expressions to three parts of the data; i.e. RT to 190 °C (approaching the Curie point), 190 to 230 °C (around the Curie point) and 230 to 800 °C (in the non-magnetic region). The latter are shown in Fig 15 as black, blue and green traces respectively and are given as equations below which are plotted in red in Fig 15. The choice of segment temperatures is arbitrary based on the position of the inflection at the Curie point. The excel data can be obtained as a digital file if required.

The fit coefficients from the three parts described above can be summarized as follows, where Y is the resistivity value in nohm·m and X is the temperature in °C:

Ni 125a	$Y = 198.9 + 0\ 399\ X + 0.0916\ X^2$	Up to 190 °C
	$Y = 4.52\ X – 216.4 – 0.009\ X^2$	190 to 230 °C
	$Y = 265.9 + 0.371\ X – 6.47E\text{-}5\ X^2$	230 to 800 °C

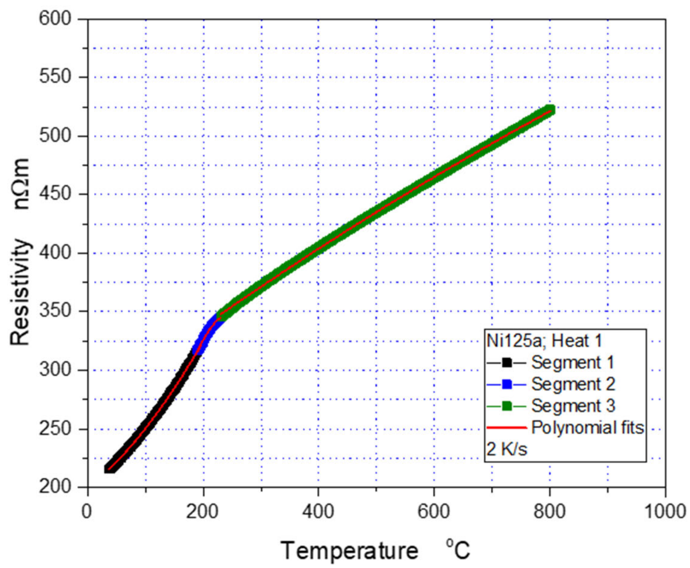

Figure 15 Segmented resistivity polynomial fits for Ni125a

3.2 COMPARISON WITH NPL 2006 REPORT ON NICKEL 125 PROPERTIES

In 2006 the temperature section at NPL measured, for an external company, the temperature dependence of resistivity on Ni 125 testpieces, of totally different provenance to the current materials, using a one-off test system of different design to the ETMT. The results of that exercise are plotted in Fig 16 against the data from the first heating run on testpiece Ni125a in Fig 15. The comparison is good bearing in mind the many differences in procedure and material source. This comparison helps validate the current exercise.

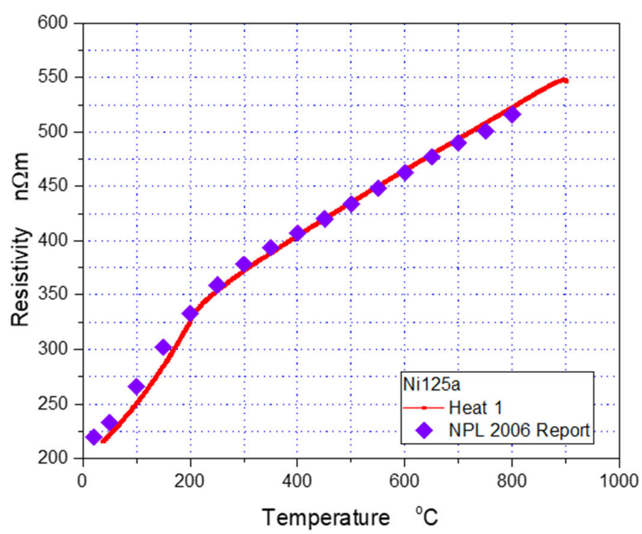

Figure 16 Comparison of resistivities in the two test processes where both alloy source and measurement procedures differed significantly.

4 NICKEL 541

Ni 541 does not have a Curie point and the resistivity/temperature graph is a continuous curve. The resistivity data for two heating cycles up to 800 °C on three separate testpieces (coded Ni541a, Ni541b and Ni541c) were measured and plots for two (Ni541a and Ni541b) are shown in Figs 17 and 18. The data for Ni541c was more of less identical and isn't plotted. The heating and cooling curves for all three testpieces indicate good correspondence for the applied rates of 2 °C/s.

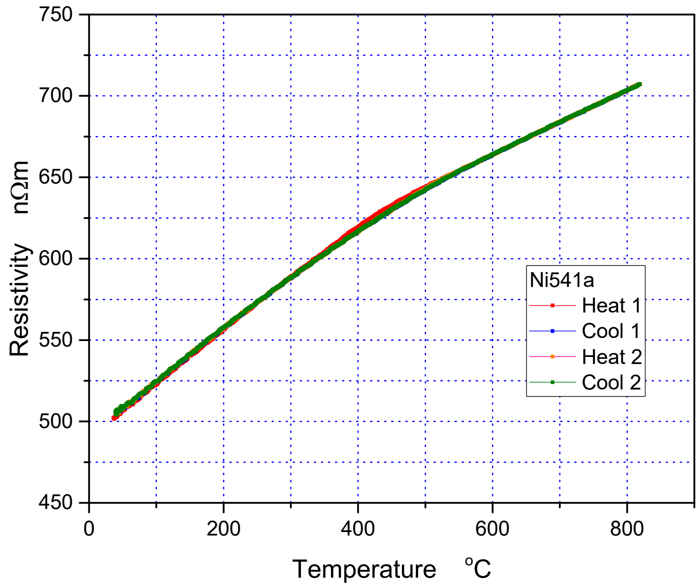

Figure 17 Temperature dependence of the resistivity of Ni541a.

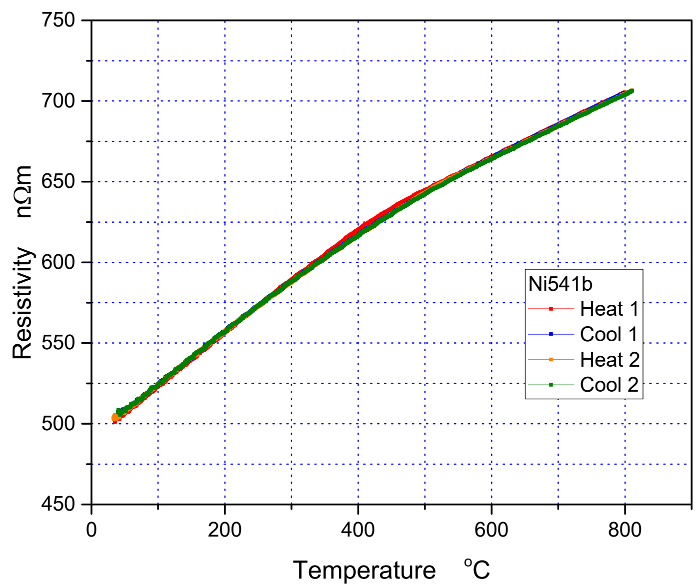

Figure 18 Temperature dependence of the resistivity of Ni541b.

4.1 DIGITAL REPRESENTATION OF NICKEL 541 DATA

Two options are provided for the digital representation of the Ni 541 resistivity/temperature data. One is to provide an excel sheet of the measurements with appropriate uncertainties, as discussed in the summary. The other is to fit a second order polynomial expression to the data i.e. RT to 800 °C. The latter are shown in Figs 19, 20 and 21 and are given as equations below which are plotted in black in Fig 19, 20 and 21. The excel data can be obtained as a digital file if required.

The fit coefficients can be summarized as follows, where Y is the resistivity value in nohm·m and X is the temperature in °C:

Ni 541a	$Y = 489 + 0\ 369\ X – 1.27E\text{-}4\ X^2$	Up to 800 °C
Ni 541b	$Y = 489 + 0.369\ X – 1.26E\text{-}4\ X^2$	Up to 800 °C
Ni 541c	$Y = 489 + 0.370\ X – 1.28E\text{-}4\ X^2$	Up to 800 °C

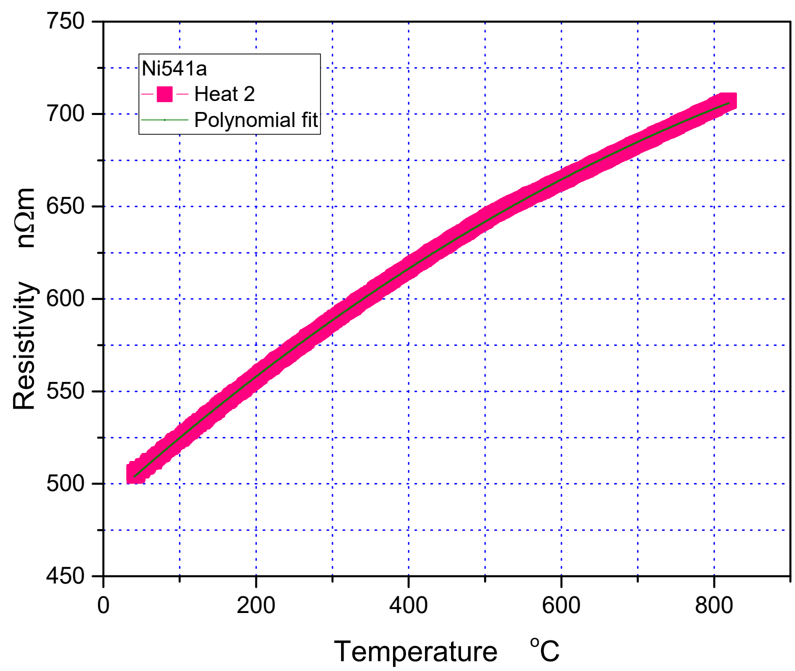

Figure 19 Polynomial fit to temperature dependence of the resistivity of Ni541a.

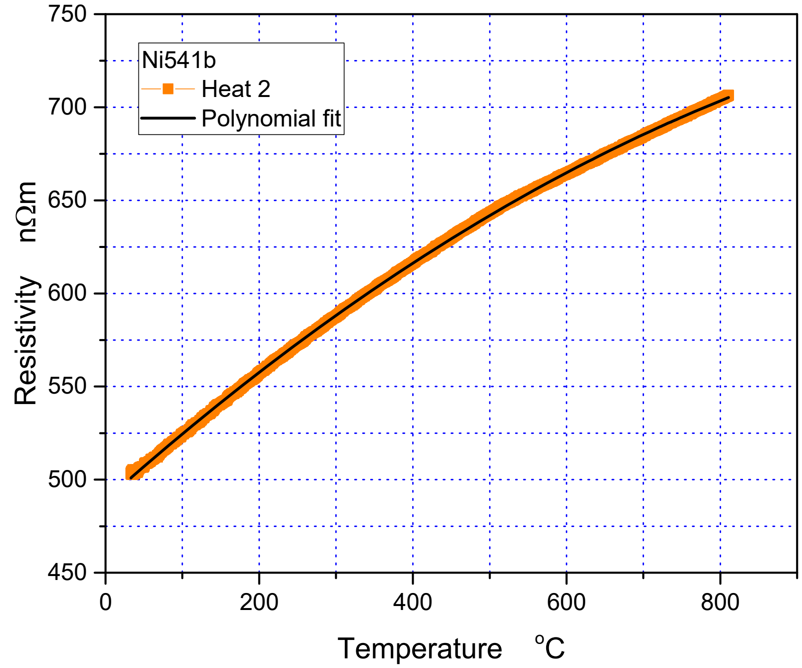

Figure 20 Polynomial fit to temperature dependence of the resistivity of Ni541b.

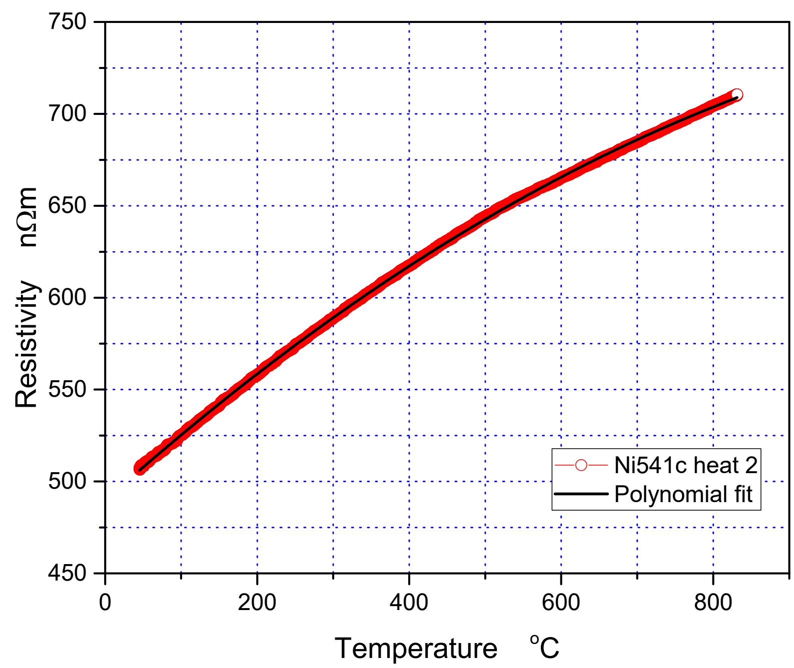

Figure 21 Polynomial fit to temperature dependence of the resistivity of Ni541c.

5 SUMMARY

A study has been conducted on three dilute Ni alloys to develop a digital representation of a physical property, resistivity, that changes in a regular way with changing temperature such that the digital information can be used across different physical testing systems as a calibration tool to give enhanced confidence that experiments are working satisfactorily. All three alloys were found to be suitable and some have distinctive reference points (The Curie Point), The information is directly applicable to a testing system, the ETMT, which allows miniature testpieces of conductive materials to be examined for their mechanical and electrical properties as a function of temperature. Interpretation of the temperature dependence of properties can be enhanced by measuring the electrical resistivity as a function of temperature, where phase or magnetic transformations are well known. This report examined some of the calibration issues associated with digital data regarding current, voltage and temperature with a supporting understanding of uncertainties. The next step is to engage Instron (ETMT manufacture) re digital circulation to current and future users of the Instron ETMT.

The compositions of the Ni alloys were:

Ni 201: Nominally pure Ni (99%); Low carbon version of Ni 200.
Ni 125: Al (1 to 2%); Si (1 to 2%); Y (0.1 to 0.2%); Balance Ni
Ni 541: Al (2%); Cr (2%); Si (2%); Balance Ni

The rationale for investigating these three alloys is that Ni 201 has a well defined Curie point, Ni 125 is a dilute Ni alloy but with a different Curie point and Ni 541, also a dilute alloy, has a smooth resistivity curve up to 800 °C without a Curie Point. These differences were shown to be useful for highlighting variations in behaviour.

6 ACKNOWLEDGEMENTS

The author would like to thank John Burrows of Federal Mogul for supplying some of the Ni alloys tested in this report.

7 REFERENCES

1 Room Temperature Electrical Resistivity Measurements of Metals. NPL Procedure, QPMATERIALS/B/243, March 2010.

2 B. ROEBUCK, M. BROOKS and A. PEARCE. Good Practice Guide for Miniature ETMT Tests, NPL Good Practice Guide No. 137, January 2016