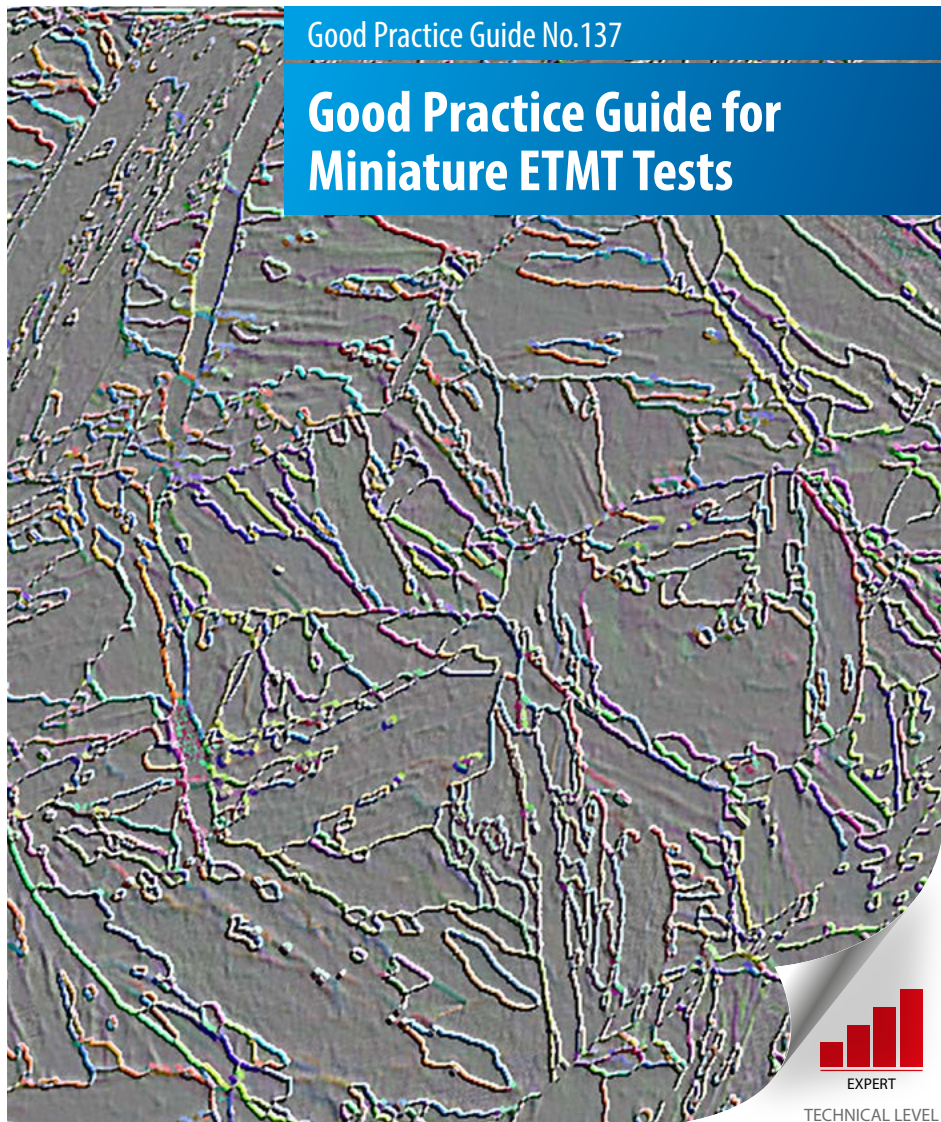


Good Practice Guide No.137

## Good Practice Guide for Miniature ETMT Tests



EXPERT

TECHNICAL LEVEL



Department  
for Business  
Innovation & Skills

FUNDED BY BIS

# Good Practice Guide No. 137

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**Front Cover Image:** *Tempered NiCrMo steel – schematic high magnification image of underlying microstructure derived from orientation map. The image was obtained by Electron Backscatter Diffraction in a Scanning Electron Microscope (Source: NPL).*

# Guide Information

## What is it about?

This guide discusses various aspects of testing miniaturised testpieces of representative engineering alloys, especially at high temperatures with the Electro-Thermal Mechanical Test system (ETMT) developed by NPL and Instron UK. Measurement techniques are reviewed and comments made on uncertainties of measurement and best measurement practice. Typical comparative results with those from macroscopic test systems are presented, as are example case studies illustrating research applications.

## Who is it for?

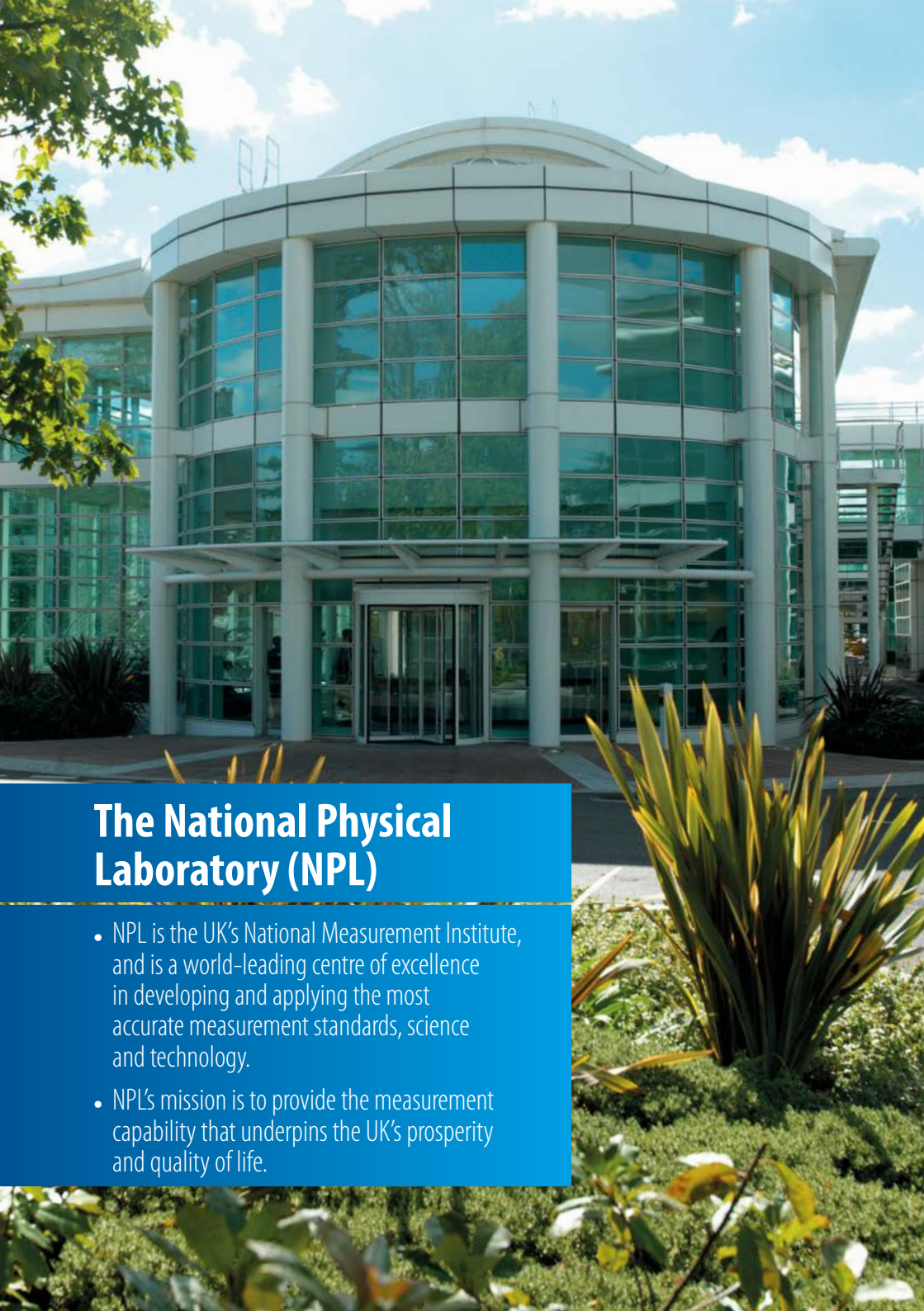
This guide is intended for researchers, scientists and engineers who may be new to the field of miniaturised mechanical testing with the ETMT and are starting, or planning, to use the ETMT in the study of high-temperature alloys.

## What is its purpose?

The purpose of the guide is to provide technical information to users of the ETMT system, and to illustrate examples of applications of this measurement system for the analysis of mechanical properties of alloys, in order to support the use of this novel miniaturised technique to generate high quality mechanical and microstructural data.

## What is the prerequisite knowledge?

The guide assumes that the reader has some familiarity and experience in high temperature mechanical testing; has a good technical knowledge about temperature, strain and load measurement techniques; an understanding of mechanical properties of materials and experience performing mechanical tests using standard Instron application software.



## The National Physical Laboratory (NPL)

- NPL is the UK's National Measurement Institute, and is a world-leading centre of excellence in developing and applying the most accurate measurement standards, science and technology.
- NPL's mission is to provide the measurement capability that underpins the UK's prosperity and quality of life.

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# Foreword

This Good Practice Guide discusses various aspects of testing miniaturised testpieces of representative engineering alloys in the NPL / Instron Electro-Thermal Mechanical Test system (ETMT). Different strain measurement techniques are reviewed including the use of electrical resistance, grip displacement and image correlation. Detailed comments are given in respective chapters regarding load calibration and uncertainties in strain, load and temperature measurements. Experiments on reference materials, at room and elevated temperatures were used to compare miniature with macroscopic deformation behaviour. These experiments confirmed that accurate data can be obtained in many circumstances from miniature samples but in some cases there are discrepancies which indicate the need for further metrology studies. Some of these examples are assessed in case studies contributed by NPL, Instron and ETMT users.

# Acknowledgement

This GPG was underpinned by input from attendees at an ETMT interest group meeting at Instron on 23 April 2014 and discussion following that meeting. We would also like to acknowledge the contribution made by NPL colleague, Mark Gee, to the early development of the system and continuing advice on operation. The guide was produced as part of the Materials Measurement and KT programmes, underpinning research supported by the NMO within BIS.

## ETMT Interest Group members

|                                                                            | Organisation                                  | Interests                                                                                        |
|----------------------------------------------------------------------------|-----------------------------------------------|--------------------------------------------------------------------------------------------------|
| Bryan Roebuck, Matthew Brooks, Jerry Lord, Peter Qusted, Tricia Wilson     | National Physical Laboratory, Teddington      | Elevated temperature (> 1000 °C) testing, strain measurement techniques, temperature measurement |
| Peter Bailey, Andrew Pearce, Peter Fuller                                  | Instron, High Wycombe                         | System calibration, future developments, testing trends                                          |
| H T Pang, Howard Stone, Catherine Rae                                      | Roll-Royce UTC, Cambridge                     | Ni base alloys                                                                                   |
| Himanshu Lalvani, Paul Blackwell                                           | Advanced Forming Research Centre, Strathclyde | Residual stress models                                                                           |
| Roger Reed, Enrique Alabort, Fauzan Adziman                                | University of Oxford                          | Ni and Ti alloy processing and performance                                                       |
| Paul Brown                                                                 | Rolls-Royce, Derby                            | Data for modelling aerospace alloys                                                              |
| Catrin Davies                                                              | Imperial College                              | Thermomechanical Fatigue                                                                         |
| Troy Liu, Philipp Frankel, Chi-Toan Nguyen, Philip Withers, Michael Preuss | University of Manchester                      | Beam line <i>in situ</i> testing; Nuclear materials (Zr alloys)                                  |
| Jeffery Brooks                                                             | University of Birmingham                      | Metallic alloy process modelling                                                                 |
| Ludwig Schneider                                                           | Sandvik Hard Materials, Coventry              | Tool materials                                                                                   |
| Steve Moseley                                                              | Hilti, Liechtenstein                          | Construction tool modelling                                                                      |
| Jonee Zunega                                                               | Element Six, Harwell                          | Hard material TMF                                                                                |



Instron® manufactures, markets, and services materials testing instruments, systems, and accessories used to evaluate the mechanical properties and performance of materials and components such as metals, plastics, composites, textiles, ceramics, rubber, biomedical implants, adhesives, and other materials. Instron's applications technology is used by research scientists, design engineers, and quality control managers in industry, academia, and government. Due to the scope of its international operations and range of products, Instron is considered to be the world's leading full-service materials testing company.

Instron was established in 1946 in Canton, Massachusetts (near Boston). Its founders, Harold Hindman and George Burr, worked together at Massachusetts Institute of Technology (MIT). Hindman was working on a project to determine the properties of parachute materials. After discovering there was no testing machine available accurate enough to meet his requirements, Hindman teamed up with Burr to design a materials testing machine based on the latest electronics and servo-control systems. The resulting prototype was so successful that Hindman and Burr formed Instron Corporation.

Instron Limited was founded in 1960 as the European headquarters of Instron Corporation. Based in High Wycombe, UK, the company was set up to cope with the growing demand from customers in Europe. In 1969 the Instron Calibration Laboratory was one of the first laboratories in Europe to be certified to ISO17025 for force and hardness. The achievement by Instron of the ISO9001 quality standard at the end of 1992, gave official recognition to the effectiveness of the company's insistence on placing the highest possible emphasis on excellence in design, production and customer service.

In October 2005, Instron was acquired by the Illinois Tool Works Corporation (ITW). ITW is a diversified manufacturer of highly-engineered components and industrial systems, and consists of approximately 900 decentralized operations. As part of ITW, Instron is able to meet with and learn from other group operations. Instron is using these relationships to continually improve quality, productivity, delivery, innovation, and ultimately, customer satisfaction.

Today, the High Wycombe facility is the largest manufacturing plant for materials testing machines and systems in Europe, with the largest concentration of engineering design and development of such equipment. Within the European market, Instron is the leader for both electromechanical and dynamic testing machines. The company consistently exports 85 % or more of its production, and Instron has installed machines all over the world.

Since the conception of materials testing machines featuring strain-gauge load cells, Instron continues to invest in research and development programs designed to introduce technologies and services that enhance its customers' testing. The company has successfully introduced numerous innovations and industry firsts through-out its history. Instron utilize a design and operating philosophy where data integrity, safety and protection of investment are paramount, and the development process looks at real working habits and at the users' environment to bring improvements to the testing industry that go beyond improved specifications. Instron has a long history of engagement with standardisation bodies and continues to participate in relevant committees of ASTM, ISO, and BSI, both informing and learning from discussions on test requirements and best practice.

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A background image showing a person from behind, sitting at a desk and working on a laptop. The image is in a monochromatic blue color scheme. The person's head is tilted down, and their hands are on the keyboard. The laptop screen is visible, showing some graphical elements. The overall scene is dimly lit, with the primary light source coming from the laptop screen.

Chapter I

# Introduction

- Measurement framework

# Measurement framework

NPL developed a miniaturised thermomechanical test system, the ETMT<sup>1-13</sup>, which has been subsequently modified, and is now manufactured by Instron under licence. Clearly, there is an important issue of whether or not the data produced by the ETMT is accurate and comparable to that generated from more established techniques (such as tensile or TMF testing on conventional test frames). This query often arises when comparing data generated by the ETMT to that generated by other partners in inter-laboratory comparison exercises<sup>10,12</sup> or when a customer is comparing ETMT data with that previously generated elsewhere. Of especial importance is the necessity to understand when measured strength values are genuinely dependent on a real size effect, or not in the case of miniaturised tests.

The basic ETMT system comprises:

- An environmental chamber with electrical lead-throughs, water cooling for the testpiece grips and a facility for inert gas supply
- An interface electronics unit, allowing computerised control/data acquisition while providing amplified signal outputs
- Computer controlled DC heating power supply with multiple testpiece resistance and thermocouple measurement inputs. The upper temperature limit of tests is governed by the melting point and the resistivity of the test material
- Mechanical loading assembly including a flexible grip system, load cell and displacement transducer (sub-micrometre resolution and mm range), with computer controlled motor for null, mean or fatigue load application or displacement control
- System software to monitor and control each test type. Test data is stored on a computer, which can then be later analysed with conventional programs.

This Good Practice Guide compiles and summarises information from investigations over the past 10 years at NPL and elsewhere that have been conducted to support these objectives. In particular the issue of strain measurement during the mechanical testing of small testpieces, where conventional extensometers cannot be used, has necessitated the development of several experimental approaches; for example, through measurements of resistance changes, through measurements of sample grip displacement and through the use of Digital Image Correlation (DIC) techniques<sup>14-21</sup>. The first part of the guide outlines the experimental approaches underpinning these three methods for gauging the strain imposed on the sample. This is followed by sections addressing calibration tests; comparing ETMT results with RT and elevated temperature tests on different representative engineering materials; strain variation tests and evaluating strain gradients in non-parallel sided (wasted) samples. In a

further section of the guide case studies are used to evaluate some practical issues. The guide also includes specific chapters discussing uncertainties associated with the measurement of temperature, load (stress) and strain.

At NPL there are two ETMTs, one with a maximum designed load of 4 kN and another with a maximum design load of 1 kN currently de-rated with the installation of a 100 N load cell. The comments in this report on load, strain and temperature measurements are common to both. However, a recent review of load calibration of the 4 kN system is noted in chapter 5 on load calibration and uncertainties.

The Instron ETMT systems are based on a typical 2-column load frame with a crosshead mounted, electrically driven ballscrew actuator rated at 3 kN. A 5 kN rated loadcell is mounted on the base of the load frame. The Instron ETMT can be supplied with either a plastic 'low integrity' or stainless steel 'high integrity' enclosure. The size and weight of the Instron ETMT system is much larger than the original NPL variant and makes use of standard Instron parts where possible. As with the NPL system, the specimen is heated by direct resistance heating and utilises a 400A, 8 V DC power supply with thermocouple feedback to effect control. A high specification Instron 8800 control system is used to control both the mechanical and thermal aspects of the ETMT. There are currently 2 system options giving 4 variations of the Instron ETMT system (Table 1).

| Instron ETMT Variants |                                       |
|-----------------------|---------------------------------------|
| Option                | Variation                             |
| Enclosure             | Perspex Low Integrity                 |
|                       | Stainless Steel High Integrity Vacuum |
| Grip Separation       | Nominal Separation 16 mm              |
|                       | Nominal separation 40 mm              |

**Table 1:** Available Instron ETMT Variants.

The systems with the larger nominal grip separation incorporate increased height enclosures. Either grip separation variant allows the upper actuator driven pull rod to move  $\pm 5$  mm from the stated nominal separation distance.

The concept behind the Instron ETMT system is one of standard interfaces, software, controller and frame thus making the ETMT system easy to use, upgrade and applicable to different test methods.

Typical testing methods include:

- Tension tests (ambient and at high temperature)
- Limited compression tests (ambient and at high temperature)
- Thermomechanical Fatigue (TMF)
- Block programme tests (multi-segment test methods) including mode transfers.

Notes on NPL and Instron ETMT procedures are given in Appendix A and a detailed specification for the Instron ETMT can be found in Appendix B (p. 81).

## Chapter II

# ETMT test systems: overview regime

- NPL System
- Instron system

# NPL System

The NPL ETMT consists of an environmental chamber 500 × 250 × 120 mm with electrical leadthroughs, water cooling and inert gas supply facilities. A computer-controlled DC heating power supply (200 A) is used to heat the specimen, with an integral testpiece resistance and thermocouple measurement facility (Figure 1). Testpiece geometries are influenced by testpiece resistance, with higher conductivity materials such as Al and Cu alloys needing to be smaller in cross section than say low conductivity Ni-base alloys in order to generate sufficiently high homologous temperatures. Generally however, suitable testpieces are typically 2 × 1 × 40 mm and heating rates of up to 200 °C s<sup>-1</sup> are possible, dependent on the precise thermal characteristics of the testpiece.

Cooling rates are primarily determined by radiation at higher temperatures and by the thermal diffusivity of the testpiece and loss of heat to the grips. They can typically be 100 °C s<sup>-1</sup> or 10 °C s<sup>-1</sup> for good and poor conductors, respectively. The testpiece grips are water cooled and held at a fixed temperature (nominally 21 °C) to provide a constant reference point. This results in an approximately parabolic temperature distribution for testpieces smaller than about 20 mm in length with a central temperature up to 800 °C<sup>1-3, 11</sup>. Temperature distributions in testpieces heated to much higher temperatures are more uniform in the central 2-4 mm of the testpiece, typically less than ± 3 °C at a central temperature of 1250 °C (“Chapter IV”, ‘Temperature Measurement’, p. 27), because heat losses are dominated by radiation at these higher temperatures.

Mechanical tests such as a tensile test can be performed in the ETMT at room or elevated temperature. During the test the following data are collected relevant to post-test analysis:

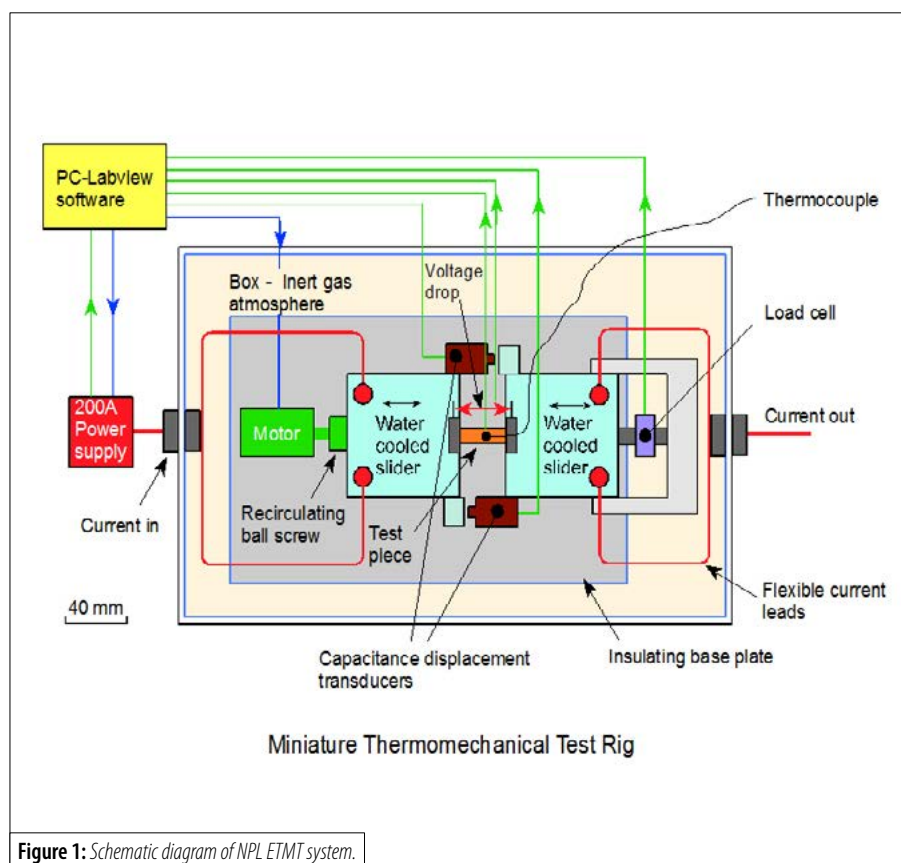
| Time | Load | Temperature | Displacement | Resistance |
|------|------|-------------|--------------|------------|
| s    | N    | °C          | µm           | milliohms  |

Load is measured in the ETMT using a suitable calibrated load cell. Temperature is measured with a type R thermocouple (Pt / Pt-13 % Rh) spot welded to the centre of the testpiece. Displacement is measured at the grips using two capacitance displacement sensors situated laterally either side of the testpiece. Two spot welded, thin, Pt-13 % Rh wires provide contacts, usually approximately 1.5 mm either side of the thermocouple. These are used to measure the voltage drop along the sample and determine resistance when a suitable dc current is passed from grip to grip.

# Instron System

The Instron ETMT system performs essentially the same function as the NPL ETMT, in that it is an 'Electrical Thermal Mechanical Testing system'. Figure 2 shows Instron ETMT block diagram.

The Instron 8800 controller for the ETMT has two axes or control loops. ETMT position, load and strain are controlled as part of the first axis (control loop), with the second providing for specimen heating via closed loop feedback control of current and temperature. Voltage drop across the central section of the specimen can be measured via wires spot welded to the specimen and the resultant voltage between them fed in to the Instron 8800 controller. In the basic system configuration the specimen voltage is a measurement only and cannot be used as a controlling input.



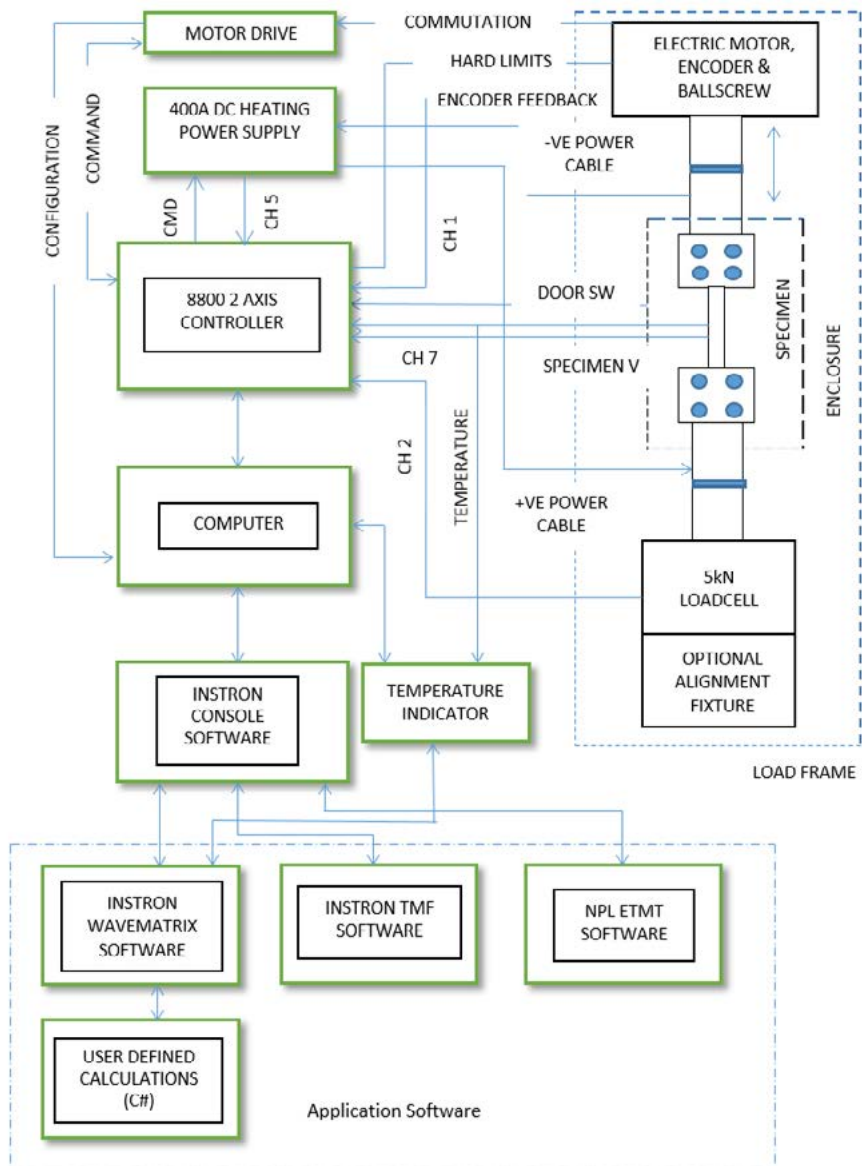


Figure 2: Instron ETMT block diagram.

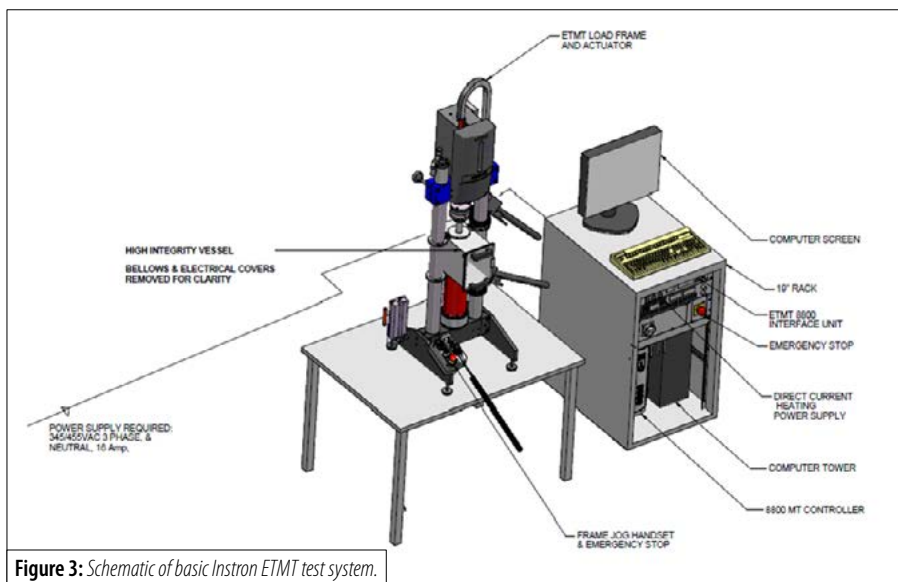


Figure 3: Schematic of basic Instron ETMT test system.

The ETMT system uses Joule's first law, resistance heating, to heat the specimen under test. In order to electrically isolate the central section of the loadstring and testpiece through which electrical current flows from the rest of the system, there are high strength ceramic isolation discs fitted at the top and bottom of the loadstring. The high integrity vessel, when fitted, is manufactured from stainless steel and this is also electrically isolated from the electrically energised region. To comply with safety regulations the electrically energised sections of the loadstring outside of the specimen enclosure are fitted with split covers to prevent access. Figure 3 shows the basic Instron ETMT system.

The Instron ETMT system is supplied either with a high integrity enclosure which allows vacuum evacuation followed with gas back-filling or with a Perspex enclosure with allows simple gas purging. The high integrity enclosure allows the user to operate the ETMT system under three environmental conditions – air, vacuum or under back-filled gas purge. The Perspex enclosure allows the user to operate the ETMT in either air or under simple gas purge. The gas and vacuum system schematic for the high integrity system is shown in Figure 4. A typical sequence of operation to start a test is discussed in Appendix A (p. 101).

The Instron ETMT system accommodates both round and flat specimens. Specimens are manually clamped at each end in grip heads within the environmental chamber via four M5 bolts in a clamping block (Figures 5 and 6).

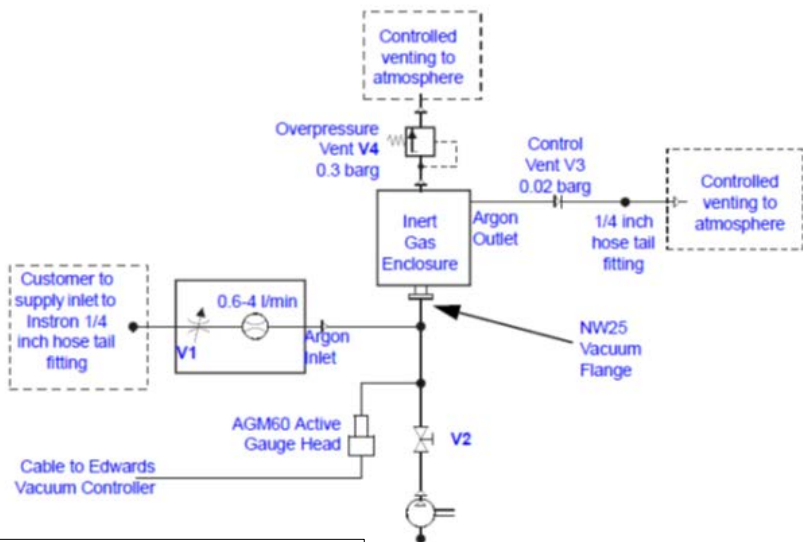


**Figure 4:** Instron ETMT gas and vacuum system schematic.

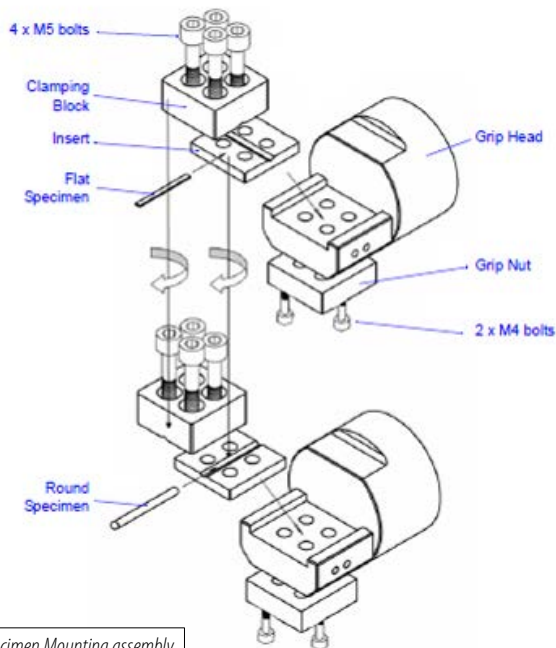
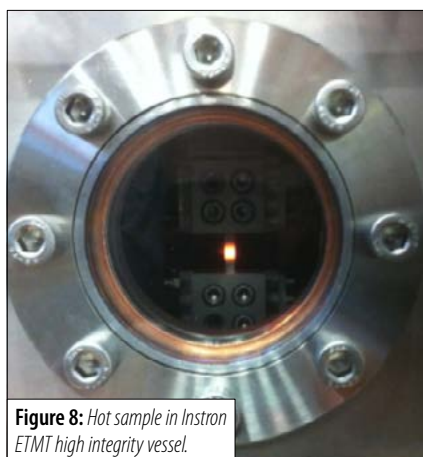
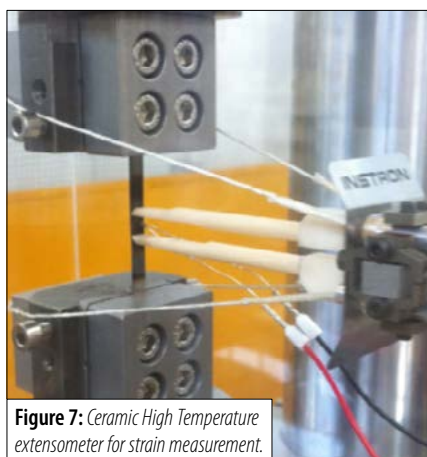
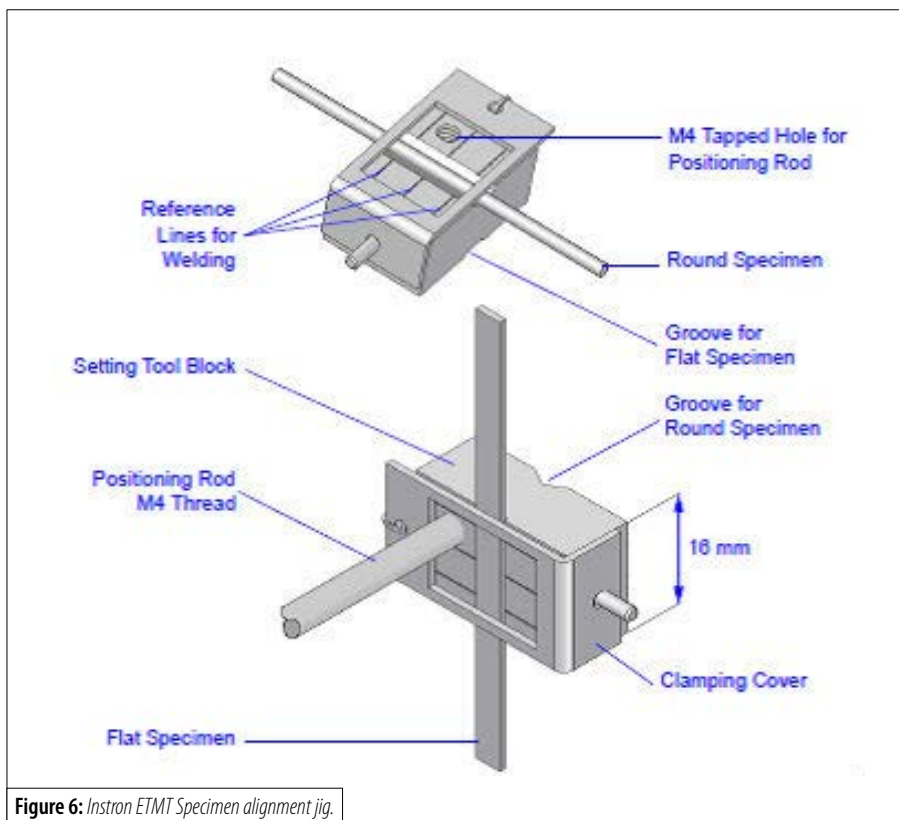


**Figure 5:** Instron ETMT Specimen Mounting assembly.



As with macro scale mechanical testing, horizontal, vertical and angular alignment of the specimen is essential to the generation of accurate, repeatable and reproducible results. Load frame alignment (excluding user's specimens) can be checked and adjusted by using an appropriately strain gauged small specimen linked to Instron's AlignPRO™ software and spherical adjustment fixture.

The key technical differences for the Instron machine with respect to the NPL ETMT system can be summarised as:

- Uses a conventional 'high stiffness' load frame as the mechanical platform
- Vertical or horizontal load frame orientation (useful when used in diffraction studies and mounted on radiation beam lines)
- Uses the standard 8800 based control system for both mechanical and thermal control
- Two main packaged components. Load frame and control system. The control system is housed in a 20U, 19" wide industry standard cabinet and houses the computer, 8800 controller, high current DC power supply, actuator motor drive and ETMT system conditioning electronics
- The Instron 8800 controller has a maximum sample rate of 10 kHz
- Standard Instron 'load and position' transducers are incorporated
- Heating system uses a 400A DC power supply and therefore larger specimens and specimens with lower electrical resistivities can be heated
- 5 mm gauge length ceramic high temperature extensometer can be used for direct strain measurement for TMF testing (Figure 7)
- Options of a high integrity vessel able to be pumped down to  $5 \times 10^{-2}$  mbar vacuum and then backfilled with inert gas if required (Figure 8)
- Vacuum pressure digital readout provided with high integrity vessel which can be connected to a PC via the serial port to data log if necessary
- Option to fit specimen alignment fixture (spherical seat) and when used with instrumented specimen and Instron AlignPRO™ software will provide the best alignment of the system
- Standard Instron application software can be used (Console, Wavematrix, Bluehill and TMF)
- Instron Wavematrix software offers 'block programming' which in turn allows 'mode transfers'.



## Chapter III

# Testpiece manufacture and design

- Testpiece geometry and preparation
- Instrumentation

## Testpiece geometry and preparation

Samples are most commonly Electrical Discharge Machined (EDM) and then lightly diamond surface ground to a nominal size of  $40 \times 2 \times 1$  mm, (Figure 9). It is not good practice to test samples with just an EDM surface as the damage imparted by this method can be quite severe, especially in harder materials. Clearly even 'lightly' ground surfaces, be it by machine or by hand, will introduce some surface deformation and structural change that must be taken into consideration when testing. However, for higher temperatures of test this deformation is likely to be annealed out.

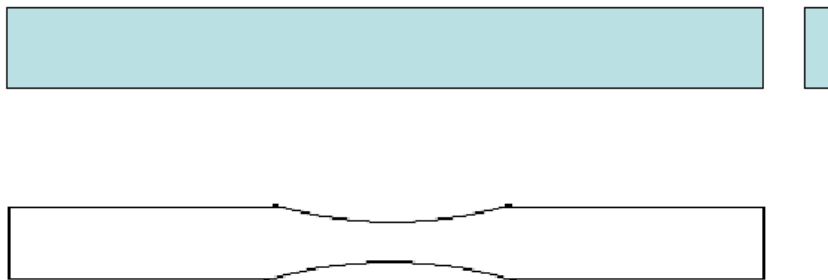


**Figure 9:** Rectangular ( $40 \times 2 \times 1$  mm is typical) and waisted testpiece geometries (width at centre of waist is generally targeted at 1.2 mm with a 60 mm radius to the profile).

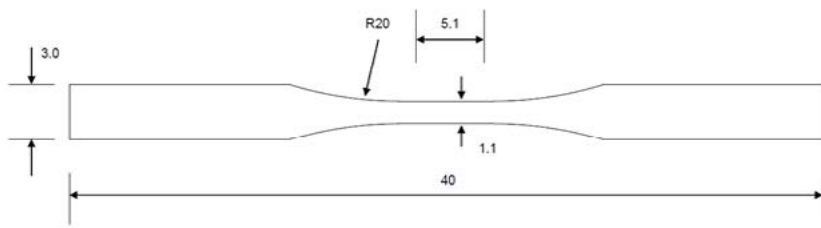


**Figure 10:** University of Cambridge testpiece design for superalloy tests (dimensions in mm).



**Figure 11:** *Typical instrument testpiece with thermocouple and potential leads attached.*

Sometimes the testpieces are waisted to give a nominal width of about 1 to 1.2 mm, because initial tests have revealed that certain samples (Ni base alloys, some steels) can occasionally fracture in cooler regions away from the central hot zone (Figure 9). Waisting the samples concentrates the stress and temperature within the central region of about 2-3 mm. The recommended waist geometry is fairly gentle (a 60 mm radius diamond wheel is preferred) and stress concentration factors due to the waisting are ignored ( $< 1\%$ ) in the calculation of nominal stress versus strain curves. However, this radius affects the temperature profile ("Chapter IV", 'Temperature Measurement', p. 27). For this reason researchers at Cambridge have developed a slightly different geometry (Figure 10), which is similar to the NPL design in Figure 9 but with a parallel sided section in the centre. Other geometries might be suitable, i.e. with wedge grips at either end of a parallel section or round and/or notched testpieces, though these have not, as yet, been systematically tested.

## Instrumentation

Testpieces are generally instrumented, in that thermocouples are attached at the centre and thin metal wires (usually Pt-13 % Rh of 0.1 mm diameter) are attached either side of the thermocouple in order to measure potential drop in the central hot zone. The spacing of the wires is generally about 3 mm (Figure 11). Sometimes it can be difficult to spot weld to certain materials, i.e. copper and its alloys (due to high conductivity) or those materials that have a thin adherent oxide layer (such as Aluminium and Titanium alloys). In these cases it is necessary to have good surface preparation usually by diamond grinding or by using SiC grit paper (grade 320 followed by 1200 is recommended). The surfaces should also be thoroughly cleaned with a degreaser or solvent. Additional good practice for sensor attachment is included in section on thermocouples in the next chapter.

The background of the slide is a blue-tinted photograph of a person's hands working on a circuit board. The person is wearing a watch on their left wrist. The image is slightly out of focus, emphasizing the text in the foreground.

## Chapter IV

# Temperature measurement

- Brief introduction
- Thermocouples
- Verification of temperature measurement
- Reference Materials
- Temperature Control
- Pyrometry
- Test Atmospheres

# Brief introduction

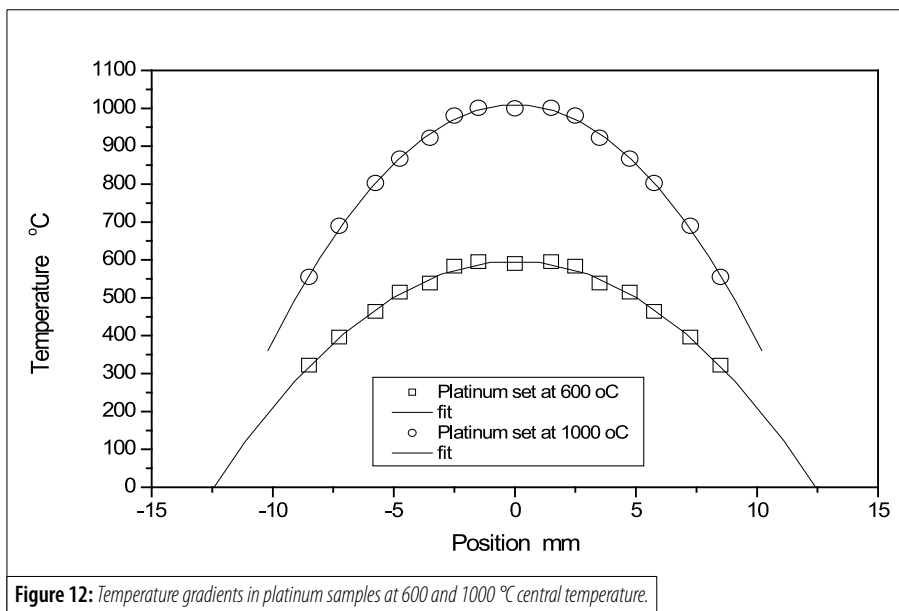
Two methods have been examined for the measurement of testpiece temperature in the NPL ETMT. One was based on pyrometry<sup>11, 12</sup> and the other on the use of type R, Pt/Pt-13 % Rh, thermocouples<sup>1-3, 9</sup>. A systematic comparison showed that the pyrometry measurements were unreliable in many cases due to changes in emissivity of the testpiece surface, especially in tests in air. Consequently, for the most part, temperatures of testpieces are usually routinely measured using the type R thermocouples.

Because the testpiece is held in water cooled grips, a non-uniform temperature distribution develops along the testpiece (Figure 12). Measurements and analysis have shown that the steady state temperature profile along the testpiece is parabolic at temperatures up to about 800 °C<sup>1-3</sup>. The NPL ETMT program software maintains control of temperature even if the testpiece changes in cross section, providing the deformation rate is not greater than about 50 N s<sup>-1</sup>. Heated samples typically deform and fracture in the central region which acts rather like a reduced cross section in a conventional test because for most materials the material is softer in this part of the testpiece. However, it should be noted that single crystal Ni superalloys are slightly stronger at 700-800 °C than at lower temperatures and this can lead to premature fracture away from the hot zone when testing parallel sided samples.

For steady state conditions, the ETMT DC heating current is set to a fixed value that ramps the temperature to that close to the required temperature and which is then controlled by a PID software module with operator variable parameters. The indicated temperature of the central 2 mm of the testpiece is approximately within the range:

| Testpiece temperature accuracies |          |
|----------------------------------|----------|
| Testpiece Temperature (°C)       | Accuracy |
| < 200                            | ± 1 %    |
| 200 - 400                        | ± 2 %    |
| 400 - 1000                       | ± 3 %    |
| 1000 - 1500                      | ± 5 %    |

**Table 2:** Approximate temperature accuracies as a function of the temperature range of the central 2 mm of the testpiece.



Typical plots of temperature gradients are shown in Figure 12 obtained by using testpieces with multiple thermocouples or by moving a single testpiece between the grips to ensure the thermocouple is positioned asymmetrically by different extents for each temperature cycle.

## Thermocouples

Type R thermocouples are generally made individually for each test. However, in some cases it has been shown that thermocouples can be used repeatedly if gently attached and then twisted off for a new test provided there has been minimal metallurgical interaction with the testpiece. This can be checked by metallographic studies or additional temperature cycles where resistivity is monitored as a function of temperature.

At NPL, 0.1 mm diameter certified matched type R thermocouple wire (Pt and Pt-13 % Rh) are fusion welded to form a small bead about 0.25 mm in diameter. The bead is then gently spot welded to the testpiece, taking care not to excessively elevate the local specimen temperature during welding. Spot welding conditions vary from material to material but in general high conductivity samples like Cu, Ag etc. are quite difficult and need higher power input to generate good contact whereas low conductivity materials, such as Ni base alloys, are relatively easy to bond.

A general welding strategy is to initially use a low setting on the spot welder and carefully ramp up the discharge energy (Current & No Cycles) until the point is reached where the testpiece can be lifted gently using the now 'just' attached thermocouple. If there is real concern that this bonding process is likely to substantially change the microstructure within a significant volume of material then further microscopical studies must be conducted to examine the extent of the changes. A further useful check that can be used is to measure the room temperature resistivity of the material in a separate rig to the ETMT and then use this value to compare with the resistivity measurement *in situ* in the ETMT system. If there is a large discrepancy it will probably be due to damage introduced by the spot welding process.

In the NPL ETMT the thermocouple output voltage is processed by a standard LabVIEW® software routine based on the NIST monograph 175 to give a direct reading in °C. Cold Junction compensation is performed in software by defining room temperature when setting up a test.

## Verification of temperature measurement

### Twin Thermocouple technique

The ETMT uses Direct Current to effect sample heating. At any given current, the sample experiences a voltage drop along all or part of its length. Whilst this is beneficial and facilitates the determination of instantaneous resistance that can be used to determine plastic strain, ("Chapter VI", 'Strain Analysis', p. 65), it can also impact the measurement of temperature through the coupling of a 'parasitic' potential drop voltage to the thermocouple emf. The question sometimes arises as to how one can be certain that the measured emf is solely due to the thermocouple and not the current being used to effect heating.

Some fundamental points regarding thermocouples and the ETMT systems are:

The thermocouple hot junction must be a very small fusion bead and not one wire welded on top of another.

- The two legs of the thermocouple must be as small a diameter as possible ( $\varnothing$  0.1 mm to  $\varnothing$  0.15 mm) so as to minimise heat loss due to conduction
- Neither thermocouple leg must be in contact with any conductive material other than the terminations associated with the thermocouple measurement circuit
- The thermocouple bead must be spot welded to the specimen with sufficient energy to ensure it remains attached throughout the test, but not such that it

will cause damage to the composition/structure of the specimen.

The heating power achieved through resistance heating is proportional to the resistance and the square of the current flowing through a sample of electrically conductive material. Thus, the current required to heat a specimen to 150 °C is lower than that required to heat to 400 °C and likewise to 750 °C. If the measurement thermocouple is subject to parasitic voltage coupling, the relative temperature measurement error will increase with the temperature of the specimen. Potentially the thermocouple will over or under report temperature depending on the direction of current flow through the specimen and the polarity of the thermocouple.

One technique that can be used to determine the validity of the temperature being measured by the type R control thermocouple is to attach a thermocouple with a different emf-temperature profile, such as type K, to the other side of the testpiece opposite the type R control thermocouple and to compare measured temperatures for a given control temperature. A typical sequential process is outlined below:

1. Fit type R control thermocouple ( $TC_1$ ) in the centre of the specimen on side A
2. Fit type K thermocouple ( $TC_2$ ) in the centre of the specimen on side B
3. Heat specimen to temperature  $T_1$  using the control thermocouple ( $TC_1$ ) for feedback
4. Read the values indicated by  $TC_1$  and  $TC_2$
5. Heat specimen to a temperature  $T_2$  using the control thermocouple ( $TC_1$ ) for feedback
6. Read the values indicated by  $TC_1$  and  $TC_2$
7. Heat specimen to temperature  $T_3$  using the control thermocouple ( $TC_1$ ) for feedback
8. Read the values indicated by  $TC_1$  and  $TC_2$ .

On the Instron ETMT, the temperature from  $TC_1$  will be reported by 8800 controller. An appropriate indicator will be needed for  $TC_2$ . It is suggested that an assessment of the errors of measurement for both indicators be undertaken in order to fully assess the congruence of the reported values at any given temperature. However, as a first estimate if the temperature reported by both measurement systems is within  $\pm 5$  °C of each other, then they can be said to be in agreement.

If the difference in the measured temperatures for both  $TC_1$  and  $TC_2$  remains relatively small and / or constant, then this would tend to indicate that either both thermocouples have a problem, or that both thermocouples are functioning correctly. At any given temperature, type K thermocouples have a much higher emf output than type R. If the difference in temperature measurement between  $TC_1$  (Type R) and  $TC_2$  (Type K) increases as a function of specimen temperature then this

suggests that an additional voltage is being imposed across the temperature input electronics.

Table 3, illustrates the nominal emf outputs of type R and type K thermocouples taken from ITS-90 at three temperatures. Columns 4 and 5 show the output of the same thermocouples but this time with a simulated parasitic voltage superimposed. Since temperature increases with driving current, the potential drop per unit length also increases and in this example results in an increased error voltage being superimposed on the thermocouple emf. Assuming both thermocouples have similar bead size and are in identical locations with identical surface conditions on opposite sides of the specimen, it follows that where both thermocouples are subject to parasitic voltage superimposition, the magnitude of that voltage would be the same on both thermocouples.

| Thermocouple emf       |                           |                           |                            |                            |
|------------------------|---------------------------|---------------------------|----------------------------|----------------------------|
| Control Temperature °C | TC <sub>1</sub> mV Type R | TC <sub>2</sub> mV Type K | TC <sub>1</sub> + error mV | TC <sub>2</sub> + error mV |
| 150                    | 1.041                     | 6.138                     | 1.249                      | 6.346                      |
| 400                    | 3.408                     | 16.397                    | 4.090                      | 17.079                     |
| 750                    | 7.340                     | 31.213                    | 8.808                      | 32.681                     |

**Table 3:** Thermocouple emf (not allowing for CJC) at 3 temperatures with parasitic superimposition.

Type K thermocouples have a substantially greater emf output as a function of temperature than Type R and thus the apparent increase in temperature (error) due to the superimposition of the parasitic voltage is smaller. Table 4 and Figure 13, below, indicates the approximate temperature error for a given voltage offset at a given temperature for both type K and type R thermocouples.

When performing tests at temperature, it is important to visually inspect the specimen and the temperature readouts, especially when testing at temperatures where thermal radiation is in or near to the visible spectrum. Starting at test temperatures of approximately 600 °C, the thermal radiation of testpieces becomes visible to the eye and the subjective colour, Table 5, can be used as a rough estimate for the temperature of the testpiece.

There have been reports of incidences where Instron ETMT users have thought that the specimen was at 800 °C as was being indicated, but in reality the temperature was below 600 °C since there was no visible sign of heating.

| Temperature errors from a parasitic voltage |                                        |                                        |                               |                              |
|---------------------------------------------|----------------------------------------|----------------------------------------|-------------------------------|------------------------------|
| Control Temperature °C                      | TC <sub>1</sub> (R) Temp Inc Error, °C | TC <sub>2</sub> (K) Temp Inc Error, °C | TC <sub>1</sub> (R) Error, °C | TC <sub>1</sub> (K) Temp, °C |
| 150                                         | 175                                    | 155                                    | 25                            | 5                            |
| 400                                         | 455                                    | 415                                    | 55                            | 15                           |
| 750                                         | 858                                    | 785                                    | 108                           | 35                           |

**Table 4:** Errors in temperature resulting from a parasitic voltage.

| Subjective colour of specimen |                            |
|-------------------------------|----------------------------|
| Nominal Temperature °C        | Subjective specimen colour |
| 480                           | Faint red glow             |
| 580                           | Dark red                   |
| 730                           | Bright red                 |
| 930                           | Bright orange              |
| 1100                          | Pale yellowish orange      |
| 1300                          | Yellowish white            |
| 1400 +                        | White                      |

**Table 5:** Subjective colour of specimen at nominal temperature.

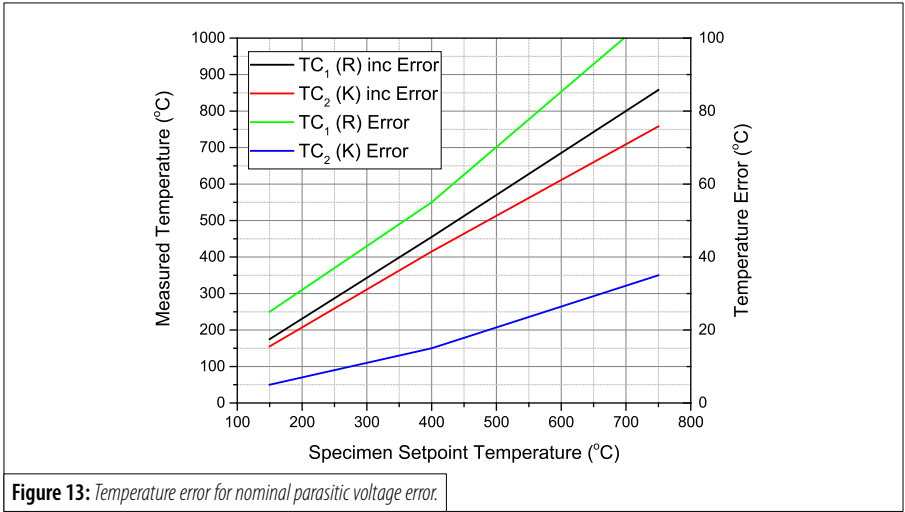


**Figure 13:** Temperature error for nominal parasitic voltage error.

# Reference Materials

A sample of pure Ti or an eutectoid steel can be used in order to check the combined

ETMT software and thermocouple. Both these materials have a well-defined transformation temperature. Repeat measurements have been made using type R thermocouples in the NPL ETMT and the indicated temperature of the transformation is usually within 2 °C of the handbook value (pure Ti – 876 °C; eutectoid steel – 742 °C). Figure 14 shows normalised resistance data up to 400 °C for the eutectoid steel in three separate experiments on different testpieces and thermocouples indicating good repeatability. In future it would be worth investigating the use of Pt or Pt alloys as reference materials for checking the temperature dependence of resistivity (thus performing internal calibration for current, voltage and thermocouple) over a wide range of temperatures since the temperature dependence of resistivity is well known in materials of this type.

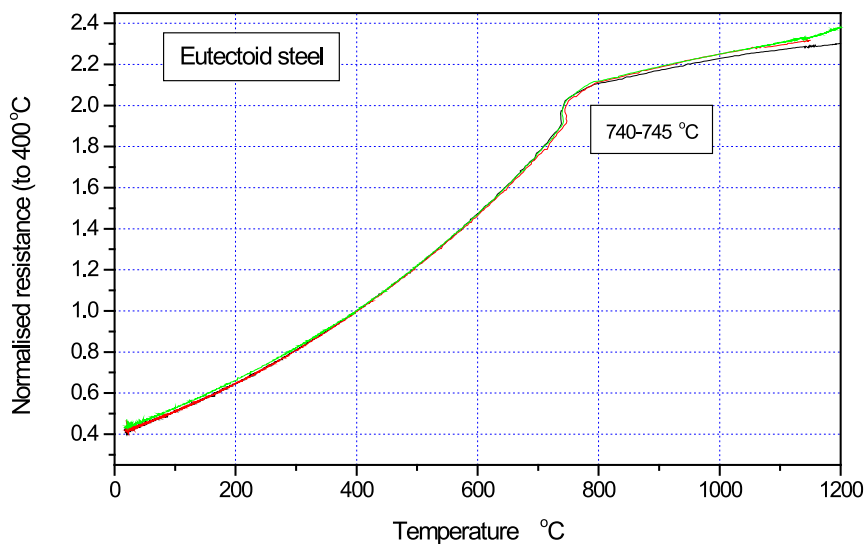


Figure 14: Temperature phase transition calibration curve.

# Temperature Control

Temperature measurement and control via the use of thermocouples requires the sampling, acquisition and conversion of relatively small voltages generated by the Seebeck effect when two dissimilar metals are exposed to a temperature gradient. For example, a type R thermocouple, as typically used in the ETMT, will generate a nominal output of approximately 6.74 mV for a hot junction at 700 °C and reference junction at 0 °C. At this temperature, a measurement error of 20  $\mu\text{V}$  at 700 °C equates to a nominal temperature error of 2 °C.

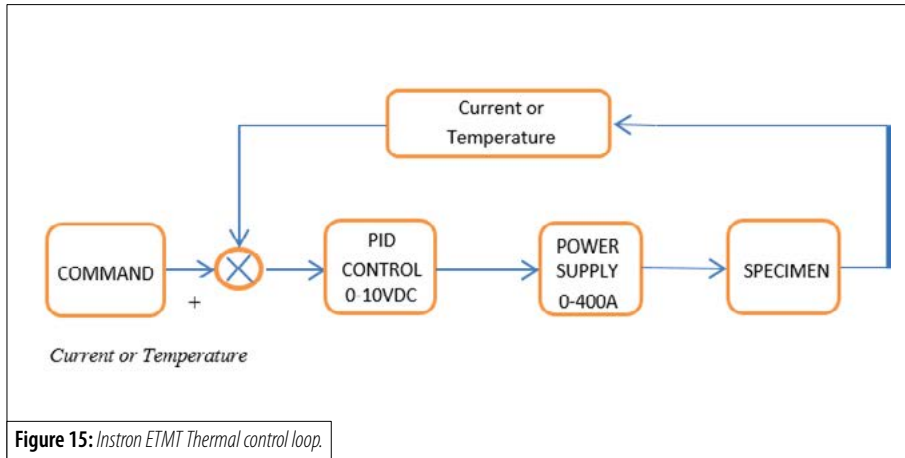
Temperature measurement and control using thermocouples can be very sensitive to electrical noise caused by earth loops, capacitive coupling and RF interference such as might be generated by proximity to electrical mains. A typical temperature controller samples at 10 Hz outputting seemingly stable displayed or transmitted temperature values, which may in part be due to filtering, under-sampling or aliasing of higher frequency noise components such as 50/60 Hz from electrical mains. The Nyquist theorem dictates that the minimum acquisition frequency needed to adequately describe a waveform is twice that of the frequency of the waveform.

The ETMT is required to heat and cool testpieces at extremely high rates of up to 200 °C s<sup>-1</sup>. In order to adequately control temperature ramps to such high rates, the temperature control bandwidth has to be well in excess of the 10 Hz of a conventional temperature controller. The NPL ETMT has temperature control implemented via a software controlled PID loop that runs at a fixed rate of 10 Hz.

Whilst this is sufficient for steady state control, or for slow controlled ramps of say 5 to 10 °C s<sup>-1</sup>, this implementation cannot maintain control at higher rate temperature ramps, either introducing 'steps' or a large lag between the demand and controlled temperature. Thus, it is important to verify the 'real' values of heating rates by inspecting acquired data sets rather than simply depending on nominal set values and ramp rates.

The Instron ETMT system uses a high bandwidth 8800 servo hydraulic controller with two axis control loops, the second of which is configured to control the heating of the specimen via the 400A programmable DC power supply. The Instron 8800 servo hydraulic controller, which has a maximum internal acquisition / control loop of 10 kHz, acts as a pseudo temperature controller and consequently has a much higher control bandwidth than a standard controller, or the NPL ETMT. The increased bandwidth reveals the hitherto hidden problem of signal noise on the temperature feedback signal. Typically this is in the order of  $\pm 3$  °C. A standard temperature indicator is supplied as standard on recent systems which can be used as a 'static' temperature reference.

The 8800 controller commonly has linear inputs. Thermocouple voltages vary in a non-linear fashion with temperature and therefore a special piece of software (Thermolin) is used to linearize the thermocouple control input for use by the 8800 controller. Control can be achieved on either of the thermal feedback channels i.e. current or temperature. A control loop for the temperature control of the specimen is shown in Figure 15.



Irrespective of the system being used, before attempting to control temperature the polarity of the thermocouple must be determined. The polarity of the thermocouple is tested by heating the specimen in current control and monitoring the temperature readout to ensure that the temperature reading increases with increased current.

As with the mechanical aspect of testing, the ability to control the thermal aspects of a specimen under test is influenced by the properties of the testpiece, i.e. its electrical and thermal characteristics. For a given material and specimen geometry once the thermocouple polarity has been verified, the PID control loop parameters need to be determined.

**NB**

If the polarity of the thermocouple is inverted and the system is in temperature control mode the specimen will likely be destroyed.

Experience has shown with the NPL ETMT that the default PID values of Proportional (0.2) and Integral (0.1) are good starting points for temperature control. For materials such as Ti6Al4V, values of Proportional (0.02) and Integral (0.05) have proved to deliver stable control ( $\pm 1^\circ\text{C}$ ) at temperatures up to  $1200^\circ\text{C}$ . Some additional parameters are given

in Table 5. It is worth noting that with the NPL ETMT, it has been observed that operation of the environmental circulation fans within the ETMT box can adversely impact the ability of the system to maintain control. A practice has evolved whereby the fans are used during flushing the system to ensure good circulation of the argon atmosphere but are switched off during short term testing. For longer term creep tests it may be necessary to keep the fans switched on to maintain surface integrity.

Care should be taken when adjusting the PID parameters (Table 6). These parameters directly impact temperature control and a stable system can quickly be made wildly unstable. The PID tuning characteristic of overshoot during setpoint change can be particularly troublesome when targeting temperatures near the solidus / liquidus region.

| Temperature control PID values for NPL ETMT |                      |                   |            |          |
|---------------------------------------------|----------------------|-------------------|------------|----------|
| Material                                    | I <sub>MAX</sub> (A) | Proportional Gain | Derivative | Integral |
| 304 Stainless steel                         | 60                   | 0.01              | 0.05       | 0.0      |
| Eutectoid steel                             | 90                   | 0.02              | 0.10       | 0.0      |
| Hardmetal (cemented carbide)                | 120                  | 0.02              | 0.10       | 0.0      |
| Alloy steel                                 | 100                  | 0.02              | 0.05       | 0.0      |
| Mo alloys                                   | 210                  | 0.02              | 0.10       | 0.0      |
| Nimonic alloys                              | 60                   | 0.05              | 0.10       | 0.0      |
| PI2 steel                                   | 100                  | 0.02              | 0.05       | 0.0      |
| Single crystal superalloy                   | 70                   | 0.01              | 0.05       | 0.0      |
| SA508 steel                                 | 100                  | 0.02              | 0.10       | 0.0      |
| TiAl intermetallic                          | 90                   | 0.02              | 0.05       | 0.0      |
| TP49 steel                                  | 100                  | 0.02              | 0.05       | 0.0      |
| Zircaloy                                    | 80                   | 0.02              | 0.10       | 0.0      |

**Table 6:** *Temperature control PID values for NPL ETMT.*

Since the Instron ETMT uses the same controller for both mechanical and thermal control, the loop tuning tools that are available for tuning the mechanical control characteristics are also available for tuning the thermal control characteristics of the system. Both the current and temperature control loops need to be individually tuned to maximise system performance. Examples of the tuning procedure are given in Appendix A (p. 101).

## Issues relating to resistance heating of specimens

- Experience with the Instron ETMT has shown that it is possible to generate an asymmetric temperature profile over the specimen length; i.e. the temperature is not at its hottest in the centre and/or the temperature profile is not symmetric along the length. Possible causes are: (a) poor specimen gripping causing localised heating at one end relative to the other; (b) variation in specimen microstructure along the length of the testpiece; (c) during deformation, the specimen is not 'necking' in the central region of the specimen.
- As temperature increases and the mechanical stiffness of the specimen reduces the proportional gain (P-term) for the load channel on the Instron 8800 must be reduced. Ideally tuning of the load control loop would be carried out at test temperature or at the mean temperature of a temperature cycle. However, tuning at high temperatures is not always possible since prolonged exposure to high temperature may promote changes in microstructure and the specimen properties. An acceptable compromise is to carry out the tuning of the load channel at a lower temperature and to de-tune the proportional gain (P-term) by 10 %. De-tuning the proportional gain in this manner reduces the possibility of the actuator/specimen control becoming unstable whilst in load control.
- If the thermocouple becomes detached from the specimen while in temperature control, it is almost certain the specimen will be destroyed. The system effectively becomes open loop with no (or rather fixed RT) specimen temperature feedback and will ramp up the current in an attempt to maintain temperature. On the Instron ETMT, there are a few things the user can do to help protect the specimen:
  - a) Set current limits within the Console Software. For example, a high limit could be applied to stop the specimen from overheating. However care needs to be taken if cycling temperature at high rates since the transient currents required to achieve the demanded temperature profile could trigger the Console alarm limit. The low Console limit can also be used to stop a test if the specimen fails (fracture).
  - b) The maximum analogue drive signal from the Instron 8800 controller to the high current power supply can be limited. This has the effect of reducing the maximum current that can be applied to the specimen. This may also limit the ability of the system to achieve the required heating rates.

# Pyrometry

Particular care has to be taken in interpreting pyrometer temperature measurements, particularly in tests in air at the start of the scaling process when oxides are very thin. In some materials differences of 100 °C have been noted<sup>11, 12</sup> between temperatures indicated by pyrometry and thermocouples.

In a series of experiments in the NPL ETMT, aimed at comparing pyrometry with thermocouples, rectangular samples of representative engineering materials were heated in an inert atmosphere (high purity argon – 99.998 % minimum) while measuring temperature simultaneously with a type R thermocouple (Pt/Pt-13 % Rh) and a two-colour pyrometer. Once the temperature of interest was reached, the atmosphere was changed from argon to the use of a standard cylinder of laboratory compressed air (the H<sub>2</sub>O content was not known), and the kinetics of the oxidation process were followed through measurement of changes in the sample resistance and changes in pyrometer output, while maintaining a constant thermocouple temperature. The pyrometer used in the study was an IMPAC system; 0.45 mm spot size; 1.5 and 1.7 µm wavelength. Figure 16 shows a schematic diagram of the experimental set up.

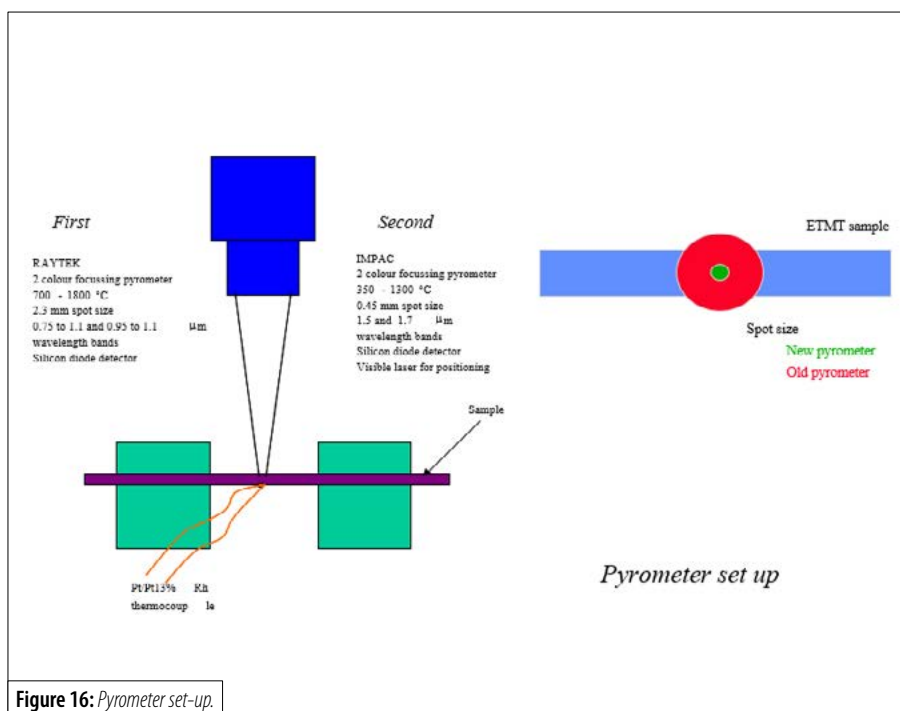


Figure 16: Pyrometer set-up.

The indicated temperature registered by the two colour pyrometer is derived from:

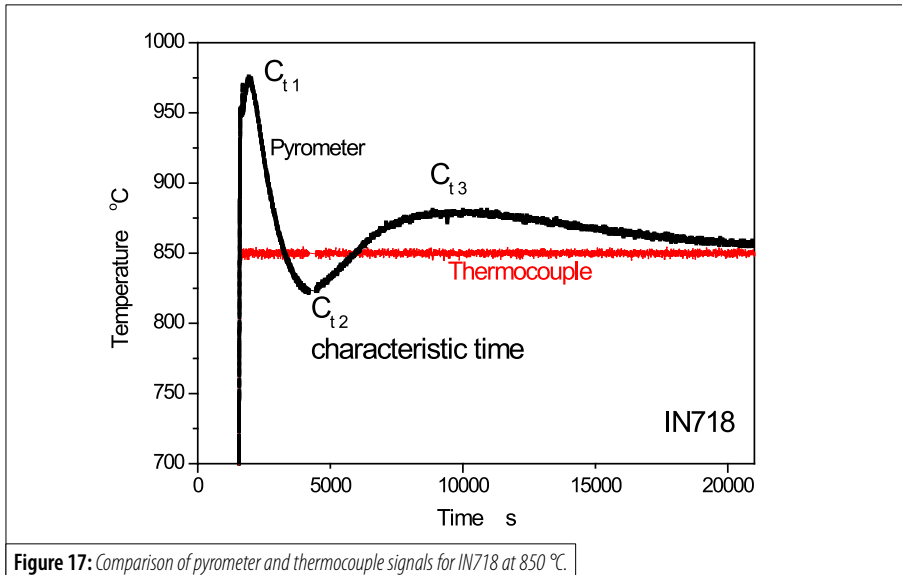
$$T = \frac{B_1}{\left(A_1 + \ln \frac{\varepsilon_1}{\varepsilon_2}\right) - \ln \frac{S_1}{S_2}}$$

where  $T$  is the temperature,  $S_1$  and  $S_2$  are the power outputs from each channel of the two colour pyrometer,  $A_1$  and  $B_1$  are constants and  $\varepsilon_1$  and  $\varepsilon_2$  are the emissivities at each wavelength corresponding to channels 1 and 2. The calculation assumes a known or calibrated dependence of the emissivity ratio on temperature and wavelength.

Two Ni alloys were evaluated as being representative of materials that oxidised slowly at temperatures in the range 700-1000 °C to form thin (< 1000 nm) oxide scales. A Ti alloy was studied for comparison with the Ni alloy, being representative of a material with a good oxidation resistance, but with different oxide characteristics.

## Ni-alloys

In representative Ni-alloys, it is known that the oxidation process is relatively slow so that initial effects in the development of thin films can be more clearly studied. A typical plot is shown in Figure 17 for the IN718 alloy oxidised in air at 850 °C after heating up to temperature in argon.



Large differences can be seen between the initial temperature registered by the thermocouple and pyrometer for the clean unoxidised surfaces in argon. As oxidation proceeded, significant interference effects can be observed. The pyrometer trace has three characteristic features – an initial peak, followed by a minimum and then a second peak, ( $C_{t1}$ ,  $C_{t2}$ ,  $C_{t3}$ ). These features were identified as characteristic times for all the experiments conducted on the Ni alloys. Equivalent traces are seen for similar experiments on Nimonic 90 (Figure 18).

These characteristic features are caused by interference effects due to a small shift in phase between the response of the two pyrometer channels.

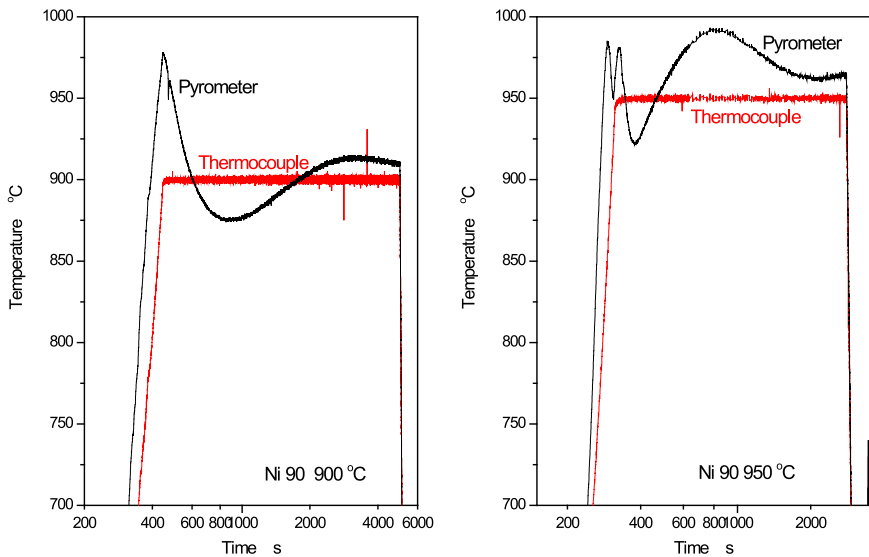


**Figure 18:** Comparison of pyrometer and thermocouple signals for Nimonic 90 at 900 °C and 950 °C.

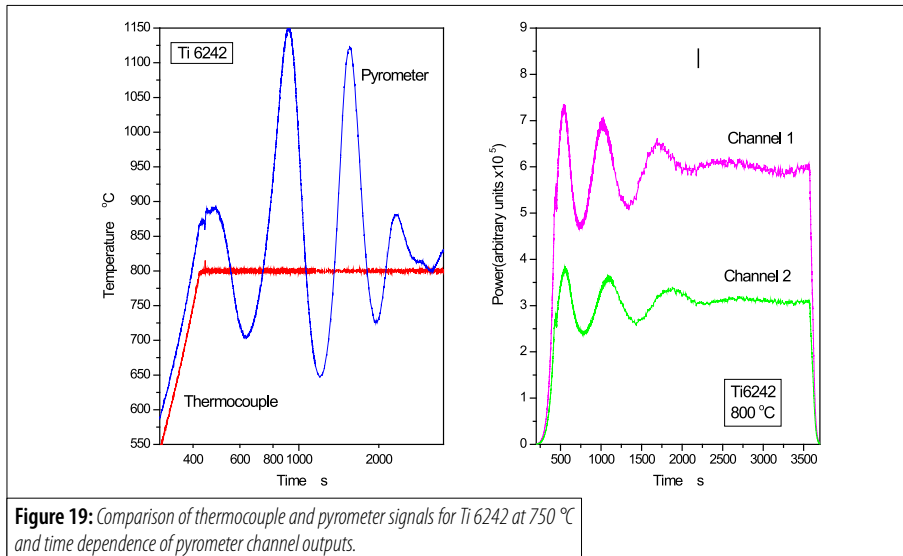
## Ti alloy

The interference effect can be seen even more clearly in an experiment on the Ti 6242 alloy at 750 °C (Figure 19). There are more peaks and they decay more slowly. The difference between pyrometer-indicated temperature and that using the thermocouple can be large, over 300 °C at some points, due to this shift in phase.

Studies in the ETMT system and associated modelling activities<sup>11, 12</sup> allowed the following conclusions to be drawn:

- There were substantial differences between temperatures measured using thermocouples and a two colour pyrometer for a range of representative iron and nickel and titanium alloys
- These differences were time dependent and could be rationalised through a model of emissivity changes dependent on the thickness of the oxide formed
- The two colour pyrometer signal became relatively stable once an oxide of thickness of about 2 µm had developed.

These findings are of significance in the materials testing community because the duration of mechanical tests often does not allow a stable surface condition to develop. Therefore, to allow pyrometry to be used with confidence as a measurement tool for experimental analysis and/or control, it is vital to understand, for each material of interest, how the surface changes with time, both in composition and structure, in the early stages of atmospheric exposure at elevated temperatures.



# Test Atmospheres

Many materials, such as engineering steels, oxidise quite quickly at high temperatures. Other materials interact strongly with oxygen, e.g. titanium alloys. Though some material types are more resistant to the environment, high temperature Ni base alloys for example, it is clearly necessary to have the option of testing in vacuum or controlled atmospheres. An option on the Instron ETMT system is for the high integrity vacuum vessel, as described in the paragraph 'Instron System' (p. 17), enabling tests to be performed in either reduced pressure or backfilled environments. The NPL system is only sealed with an O-ring and in this case an inert gas purge is used to keep testpieces clean. Generally, high purity Ar is sufficient but occasionally the use of a reducing atmosphere such as Ar-5 %H<sub>2</sub> has been used for testing those material systems where the oxidation kinetics are particularly fast. For the Instron vacuum option it is also useful to purge the container several times before the test with an inert gas before final evacuation or alternatively filling the container again fully with an inert gas.

It may be possible in the future with the Instron ETMT to utilise gases which mimic, for example, fireside service conditions. These could include the following:

- NO<sub>x</sub>
- H<sub>2</sub>S
- Non-stoichiometric reactions.

The use of these environments would necessitate additional testing overheads familiar to those performing tests in aggressive gaseous environments:

- Gas leak detections and notification systems
- Controlled venting of exit gases through process gas extract systems
- Routine health and safety checks
- Risk assessments incorporating gaseous environment.

Testing in different gaseous environments allows another dimension to the characterisation of a material. For example, mechanical loading; temperature histories; environmental exposure.



## Chapter V

# Load and stress measurement

- Instron ETMT
- Verification of load and stress measurements in the NPL ETMT
- Most Recent Load Verification Tests in the NPL ETMT
- Load Application
- Influence of Cooling Water Temperature

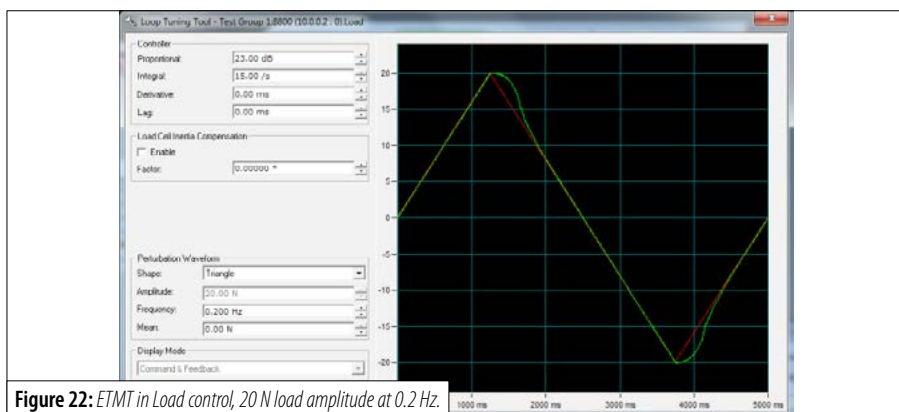
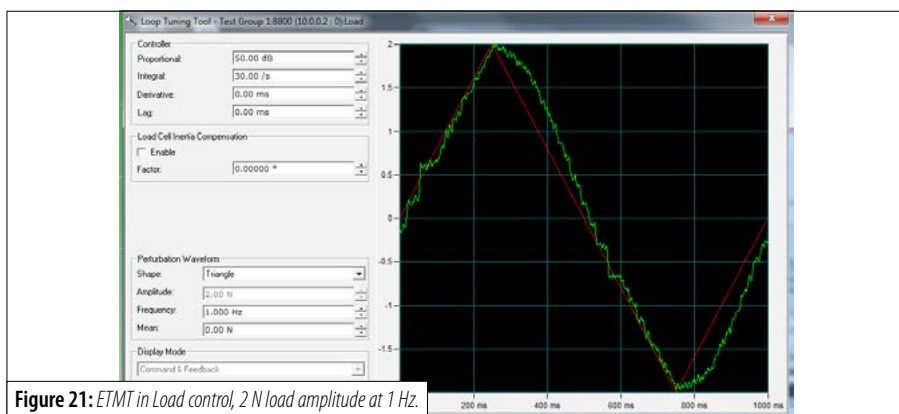
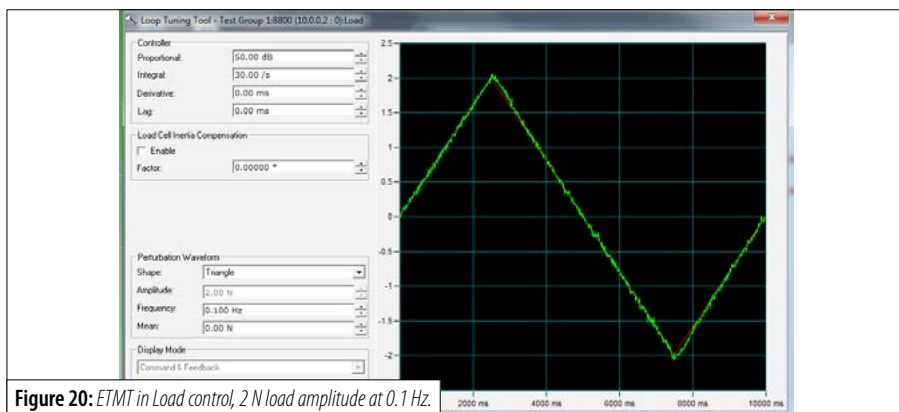
## Instron ETMT

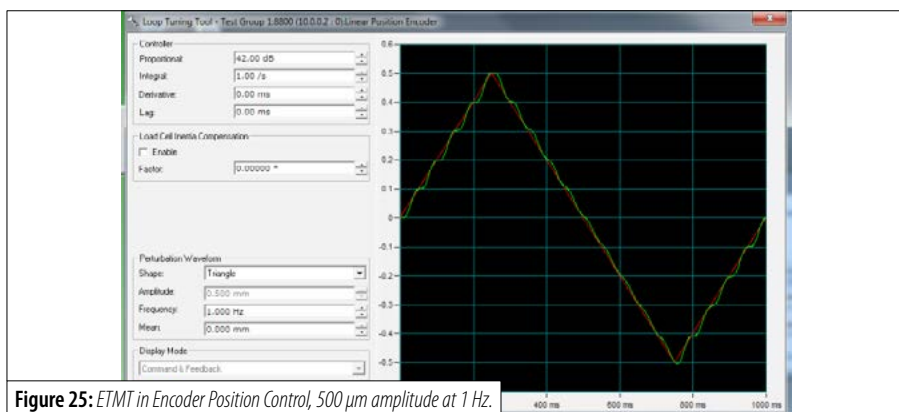
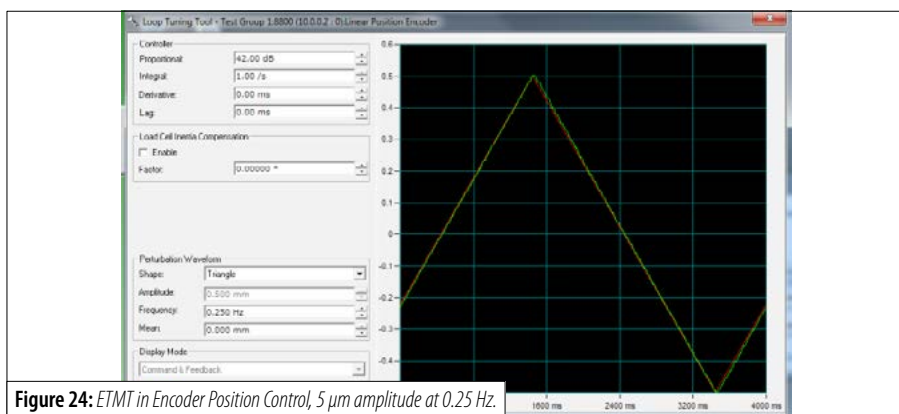
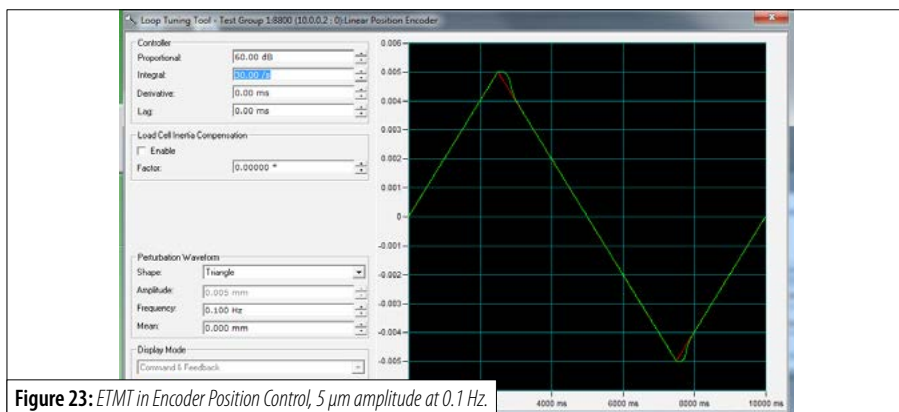
The ETMT system is designed for use with small specimens when compared to typical material testing systems. Mechanical test performance is dependent upon high resolution feedback and control of load, position and strain. In addition any electrical noise must be kept to a minimum in order for the feedback signals to be useable. One potential problem exists with signal noise emanating from the high current (up to 400 A) heating cables. The cables used to connect between the high current power supply and the load frame are 185 mm<sup>2</sup> cross section and are either 3 m or 5 m long. The supply and return cables must be twisted together, effectively forming an unshielded twisted pair cable in order to reduce radiated electromagnetic interference and therefore reduce the possible noise induced in the transducers. In addition, the two high current cables must be separated as far as possible from the other cabling between the 20U control cabinet and the load frame.

Load is measured by a strain gauge based gauged element complete with integral 'Shunt Calibration' to allow automatic calibration. Depending on user preferences, the loadcell can be verified against a reference loadcell. The standard ETMT loadcell is fitted in the lower section of the loadstring outside the low / high integrity vessel. The working volume inside the low / high integrity vessel is quite small and thus a conventional verification loadcell cannot be used. Rather, a miniature loadcell needs to be fitted along with suitable fixtures.

The mechanical control characteristics of the system are inherently a function of the mechanical characteristics of the material under test at test temperature as well as the demanded mechanical control i.e. amplitude and frequency. Therefore, the Proportional, Integral and Derivative parameters define the control characteristics need to be tuned to the material / test conditions. The loop tuning tool provides this capability. However, in essence the Proportional gain needs to be reduced with increasing demand waveform amplitude, frequency and test temperature (Figures 20 to 25).

Depending on the mechanical characteristics of the specimen, mechanical loading (Displacement, Load and Strain) frequencies of up to 3 Hz can be achieved. Loop tuning is essential for the generation of quality data. The Instron Console Software has a good user interface for loop tuning. It is recommended to perform manual loop tuning as opposed to auto-tuning since this allows users to become familiar with the specimen characteristics.





## Verification of load and stress measurements in the NPL ETMT

The usual approach taken to verify the load reporting of the NPL 4 kN ETMT is through a calibration transfer device. By installing the transfer device in the ETMT as if preparing to calibrate the load, it is possible to compare the load demand requested and the value reported back by the NPL ETMT software with an independently calibrated device.

The transfer device used to calibrate the 4 kN ETMT is a 5 kN model SSM S-type loadcell manufactured by Interface Advanced Force Measurement, used in conjunction with an RDP E-725 signal conditioning and display unit. The load cell and indicator are calibrated every two years by NPL Force section, as per BS EN ISO 376:2004. When calibrated in April 2012, the transfer device calibrated as class 00 in tension over the range 5 kN to 0.5 kN and class 1 in compression over the range 5 kN to 0.2 kN. This equates to a maximum expanded measurement uncertainty of 0.06 % and 0.24 % respectively. A repeat calibration of the 4 kN ETMT performed in July 2012 is reported in detail in the section 'Most Recent Load Verification Tests in the NPL ETMT' (p. 56).

Recently, having completed a series of tests on the ETMT, a student guest worker discovered a 400 MPa discrepancy between the data generated at about 700 °C using the ETMT on samples of Udimet 720 Li (a Ni base alloy) when testing in tension to data generated elsewhere using a Gleeble test system<sup>32</sup> when testing in compression.

Whilst there could be other explanations for this discrepancy, such as strain rate effects or even real differences between strength in tension or compression, this prompted a re-evaluation of the NPL ETMT with a view to determining whether or not there was a hitherto undiscovered flaw in the measurement system that could account for this discrepancy.

After verifying the load, a series of tensile tests were performed with a selection of materials to cover a range of tensile loads up to approximately 1500 N. All testpieces were of standard ETMT geometries at 40 mm × 2 mm × 1 mm and deformed at a loading rate of 5 N/s. An Instron 6027, serial number H1597 fitted with a 20 kN loadcell, serial number UK 731 was used to generate the reference test data for comparison on samples of the same geometry. Tests on the Instron 6027 machine were performed at a constant displacement rate, determined from the effective displacement rate during the initial elastic loading portion of the ETMT test data. The materials selected were:

- Aluminium 5052
- Aluminium 7010
- Nimonic 75 Creep Reference Material
- CMSX4 single crystal superalloy
- MAR M247 superalloy.

All the samples were 2 × 1 mm in cross-section except CMSX4 which was 1 × 1 mm. Figures 26 to 35 compare the tensile test results for the materials selected when performed on the 4 kN ETMT and the Instron 6027. As can be seen, there is on the whole very good agreement between the stress data generated from the two test systems.

Typically, the maximum discrepancy between the results generated by the ETMT in comparison with the Instron 6027 is much less than 50 MPa. The highest observed scatter in the data for Aluminium 7010 is thought to be due to material variability rather than measurement error on either the ETMT or Instron systems.

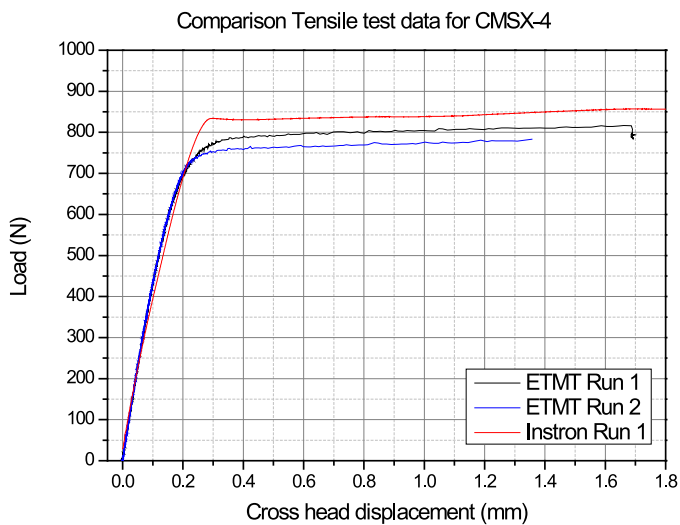


Figure 26: Comparison of load vs crosshead displacement data for CMSX4.

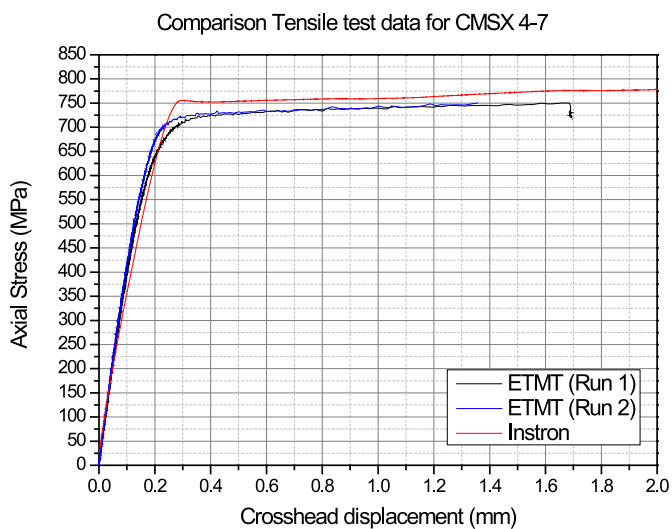


Figure 27: Comparison of Tensile Stress vs crosshead displacement data for CMSX4.

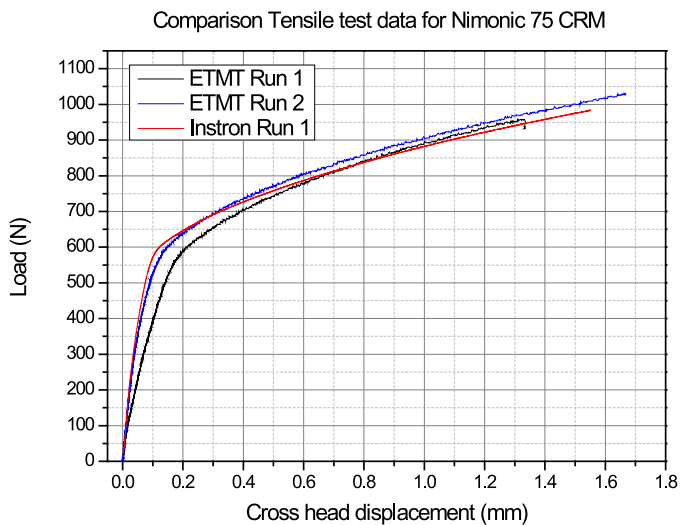


**Figure 28:** Comparison of load vs crosshead displacement data for Nimonic Creep Reference Material.

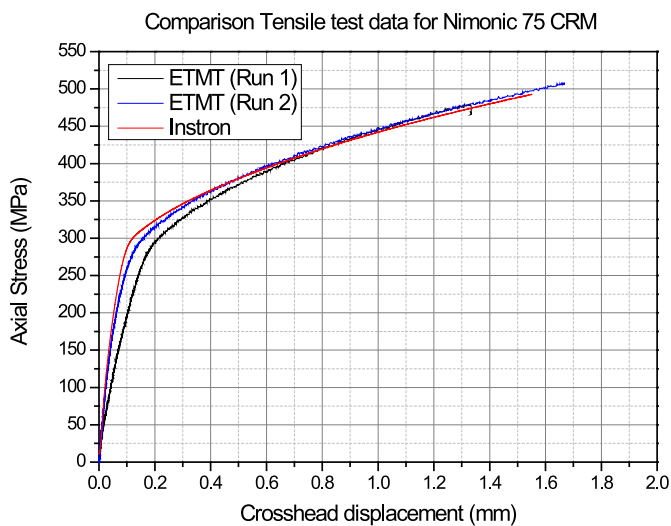


**Figure 29:** Comparison of Tensile Stress vs crosshead displacement data for Nimonic Creep Reference Material.

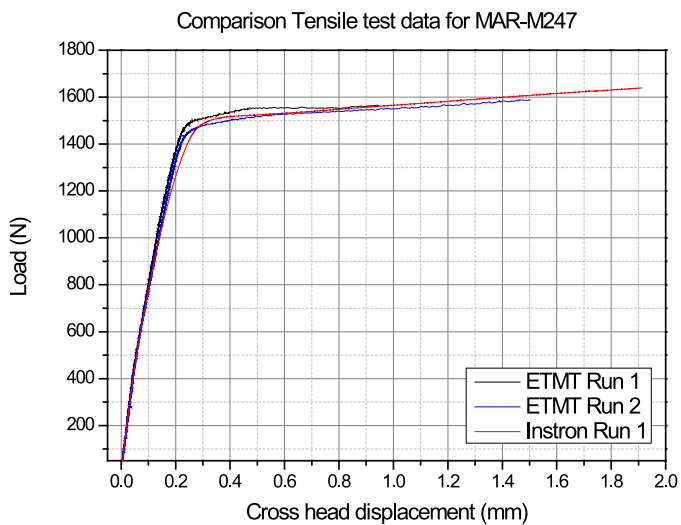


Figure 30: Comparison of load vs crosshead displacement data for MAR-M247 superalloy

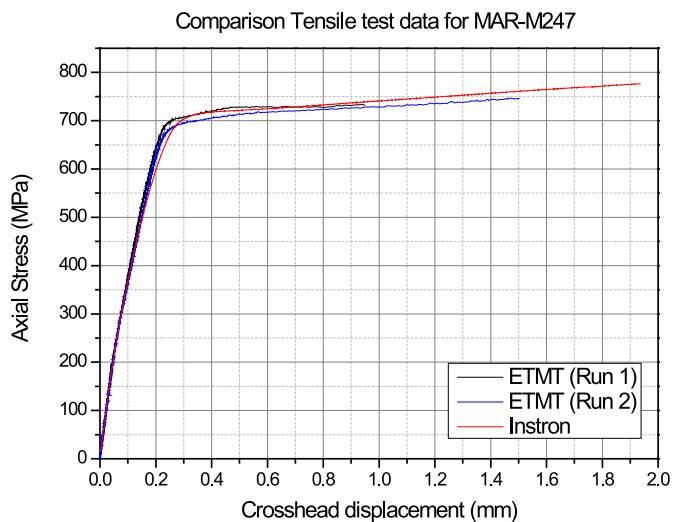


Figure 31: Comparison of Tensile Stress vs crosshead displacement data for MAR-M247 superalloy.

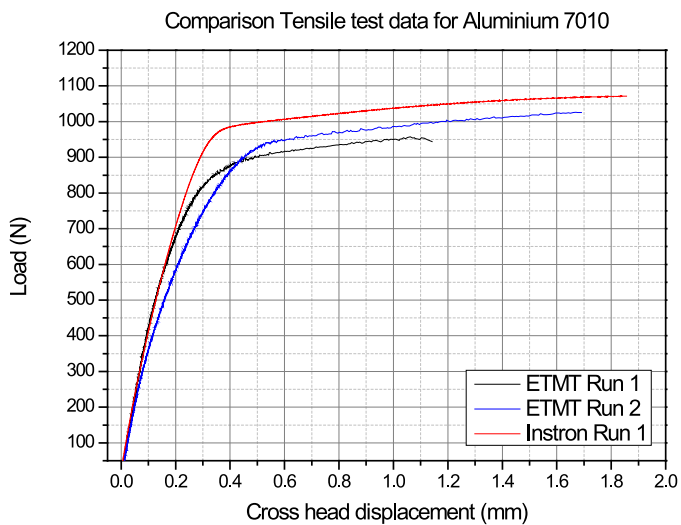


**Figure 32:** Comparison of load vs crosshead displacement data for Aluminium 7010.

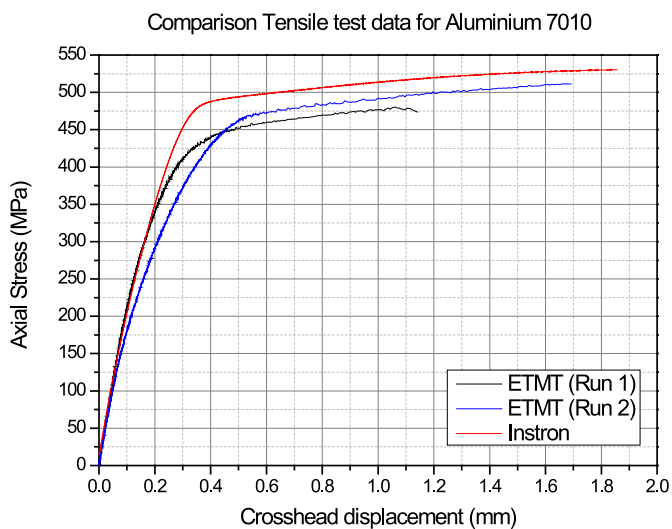


**Figure 33:** Comparison of Tensile Stress vs crosshead displacement data for Aluminium 7010.

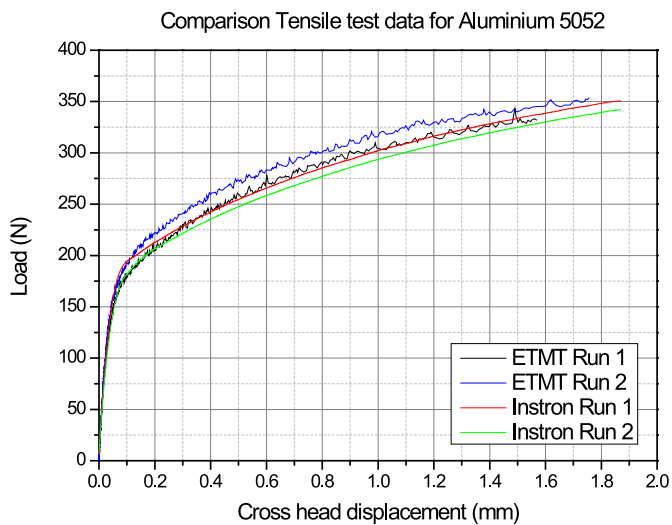


**Figure 34:** Comparison of load vs crosshead displacement data for Aluminium 5052.

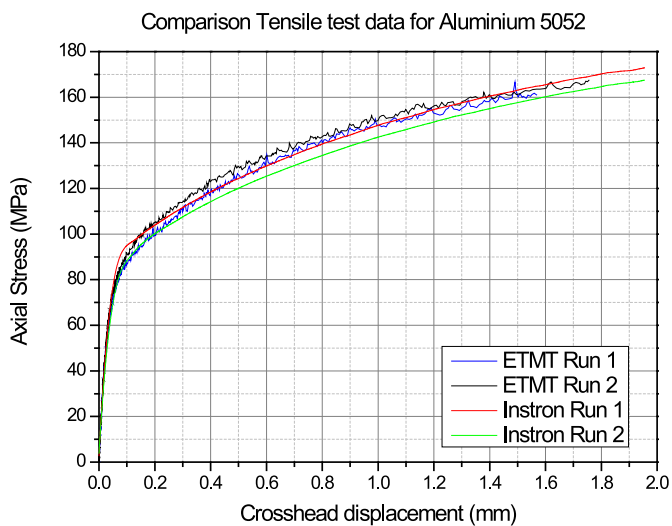


**Figure 35:** Comparison of Tensile Stress vs crosshead displacement data for Aluminium 5052.

## Most Recent Load Verification Tests in the NPL ETMT

Figures 36 and 37 illustrate the data generated from an earlier direct load verification exercise in 2011. In Summer 2012, the 4 kN ETMT load was recalibrated and updated calibration coefficients were applied to the ETMT control software. Figures 38 and 39 illustrate the comparison between ETMT reported load and the calibrated load of the transfer device post calibration following this latest calibration process.

The initial assessment of the 4 kN ETMT from the 2011 calibration revealed a significant difference between the software requested setpoint and the load as seen by the calibration transfer device; up to almost 100 N at 3500 N demand, but more significantly 25 N at or about 0 N. Figure 37 clearly illustrates the error at low loads approaching 20 % of requested set point.

Figure 38 illustrates the absolute error in N as a function of ETMT setpoint load following the Summer 2012 recalibration. It can be seen that the absolute error in controlled load is now about  $\pm 10$  N across the full range of applied loads. When viewed as a percentage of setpoint, the recalibration has reduced the error from ~20 % at low loads and 2-4 % at high loads, to 6 % at low loads and less than 0.5 % at loads greater than 1000 N compression / 1500 N tension.

Whilst the errors detected here were not sufficient to account for the 400 MPa discrepancy seen by the guest worker, it has demonstrated the need to not only regularly calibrate the ETMT but also to periodically assess the ETMT load calibration for drift and ongoing accuracy.

## Load Application

Loads (stresses) are applied in both Instron and NPL systems with an option of either displacement control or load control with rates chosen by the operator, typically in the range 0.1 to 10  $\mu\text{m/s}$  or 0.1 to 20 N/s. The choice of deformation rate is to be decided by the relevance of the desired data to material performance since changes in deformation can significantly alter the values obtained in strength measurements in elevated temperature tests.

## Influence of Cooling Water Temperature

The influence of the temperature of cooling water circulating through the ETMT grip system is typically thought of stabilising the nominally parabolic temperature profile along the gauge length of a testpiece, with the temperature of the cooling water

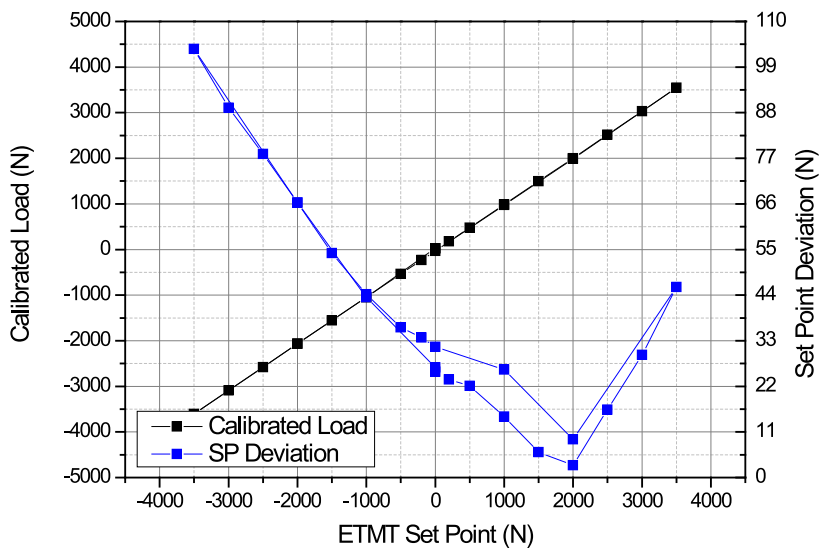


Figure 36: Set point deviation in ETMT load vs requested set point.

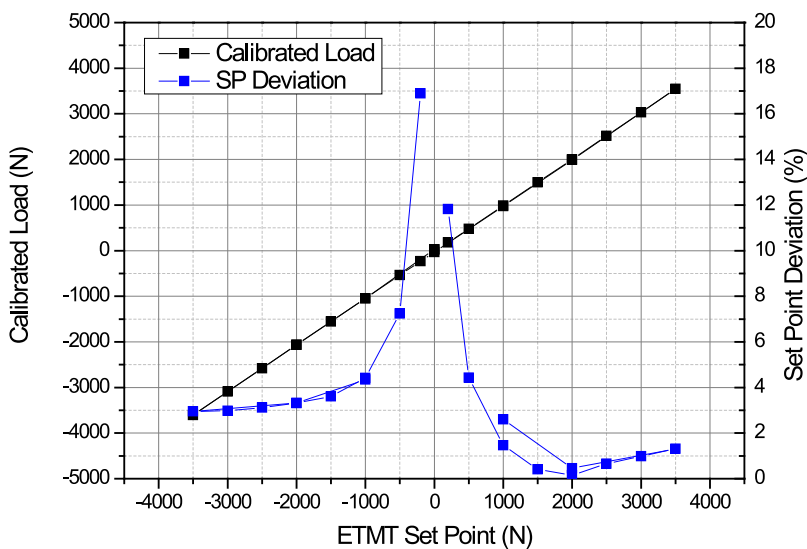


Figure 37: Error in ETMT load (% set point deviation) vs requested set point as percentage of set point.

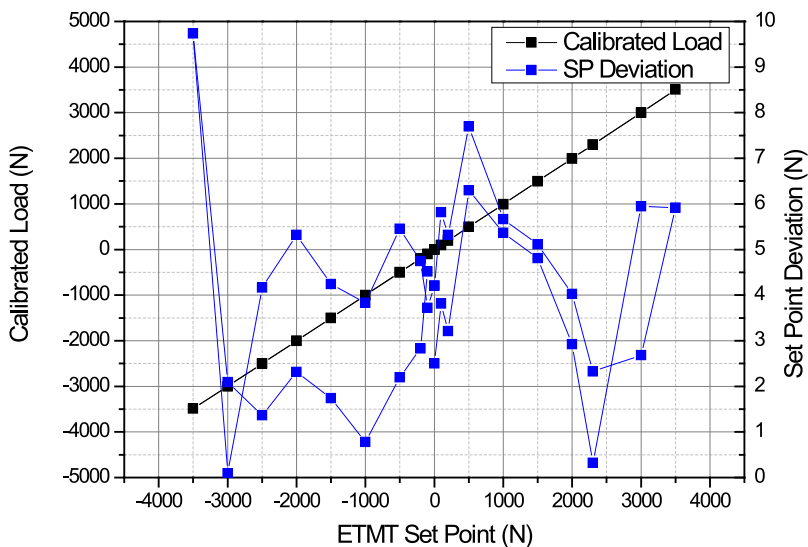


**Figure 38:** Set point deviation in ETMT load vs requested set point post calibration.

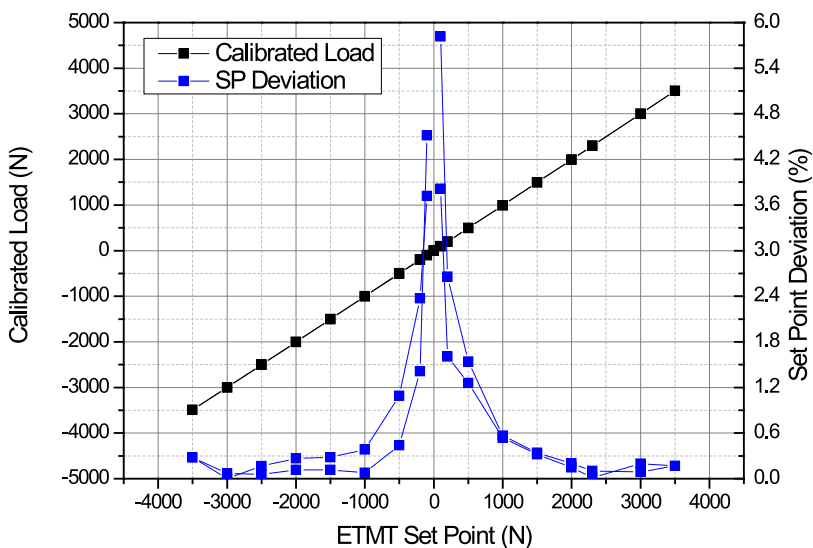


**Figure 39:** Error in ETMT load (% set point deviation) vs requested set point as percentage of set point post calibration.

fixing the temperature of the cold grips at the ends of the testpiece. The possible interaction with generating mechanical test data has until recently been overlooked.

During long term ETMT tests such as creep or stress relaxation, it is assumed that the contribution of the test frame in terms of compliance is constant and thus any variations in load or displacement are a function of the material under test. During recent 200 hour stress relaxation tests on austenitic steels at 700 °C, fluctuations in load were observed that could not be attributed to deviations in testpiece centre temperature or setpoint changes since the NPL ETMT drive motor had been switched off to hold a constant grip separation.

A series of experiments were conducted to reveal the sensitivity of the 1 kN NPL ETMT to the temperature of cooling water running through the grips. Starting with the ETMT in normal operating conditions with cooling water flowing through the grips a small tensile load of 200 N was applied to an instrumented testpiece at room temperature. With the system in one of three states – i.e. Motor off, Displacement Control or Load control – the cooling water was switched off and the ETMT allowed to drift towards the ambient room temperature which was reported by the Building Management System as being  $22\text{ °C} \pm 0.1\text{ °C}$ .

Figures 40 to 45 show the recorded load and grip position data along with testpiece temperature as a function of elapsed time. Testing under the initial state of 200 N with the motor off, Figures 40 and 41, shows load decaying as testpiece temperature (no longer being actively cooled by cold water flowing through the grips) increases. It would be expected that for a fixed system, as the testpiece temperature increases and it expands, the load would increase. The load decays is indicative of the expansion of the NPL ETMT system being greater than that of the testpiece, which is borne out by the apparent decrease in grip separation. It is worth noting that, once cooling had been re-established and a steady state attained, the load was 5 N lower than initially set.

That the same trend for the load and displacement data is repeated for the test under displacement control, Figures 42 and 43, was surprising. The expectation was that the system would respond to maintain the set displacement and thus minimise the impact on system load. It could be that the voltage output of the displacement probes is temperature dependent and this is influencing the ability of the NPL ETMT to maintain position. Further work is needed to resolve this small discrepancy.

Figures 44 and 45, which show the response of the system in load control to changes in cooling water flow and thus grip temperature, demonstrate good load control over the duration of the test with a slight ( $< 0.5\text{ }\mu\text{m}$ ) increase in grip displacement. This can be attributed to the difference in expansion coefficients between the testpiece and the ETMT mechanical assembly. The discontinuity in the data when the cooling

water flow is re-established, is a function of the analogue control electronics ability to discriminate deviation from set point and move to correct the error.

More research is thus needed to explore some of these small discrepancies and to expand the data to include testpieces at temperature (which, without cooling, introduces the complication of heating the load cell) and the introduction of thermocouples to measure the grip temperature. It is clear however that consideration needs to be given to the stability of the temperature of the cooling water being used on the ETMT in order to generate data that are not influenced by these temperature changes. This seems to be especially the case for long term tests being performed under displacement controlled conditions or for those tests where properties are being determined from grip position.

It is recommended that cooling water be allowed to circulate through the ETMT grips for at least 30 minutes prior to commencing testing to allow the system to achieve equilibrium.

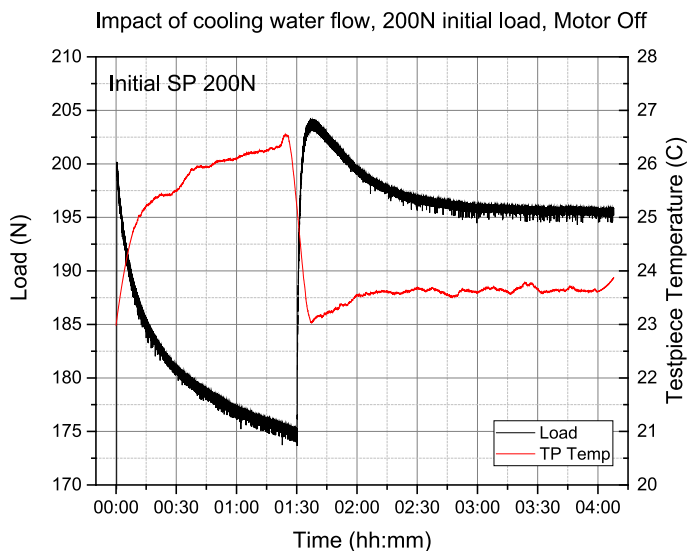


Figure 40: Impact of grip temperature on load for testpiece at 200 N with motor switched off.

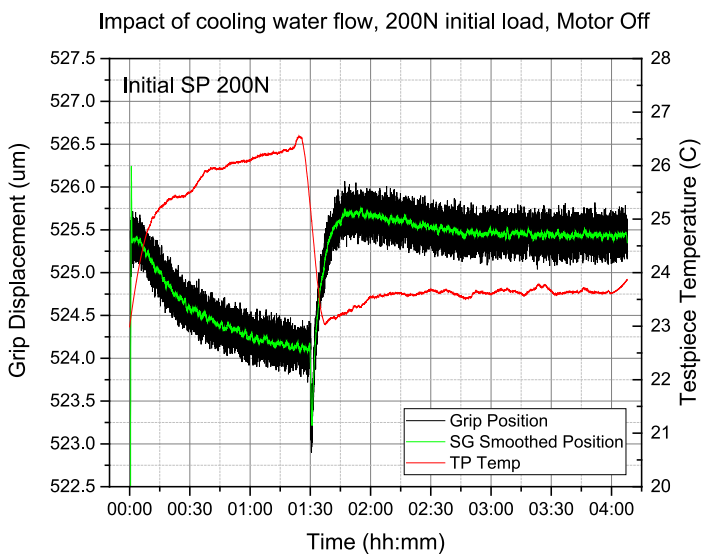


Figure 41: Impact of grip temperature on grip position for testpiece at 200 N with motor switched off.

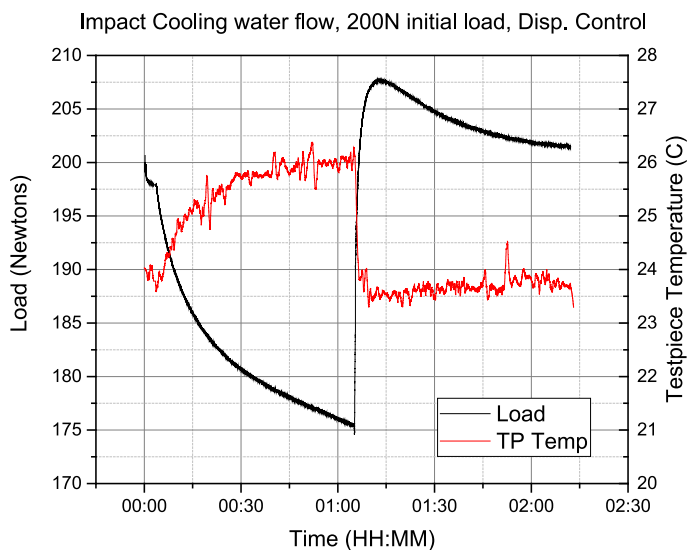


**Figure 42:** *Impact of grip temperature on load for testpiece at 200 N in displacement control.*

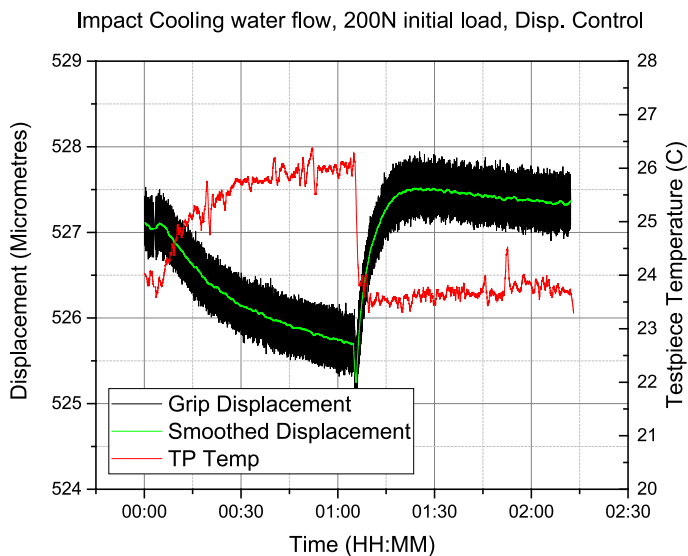


**Figure 43:** *Impact of grip temperature on grip position for testpiece at 200 N in displacement control.*

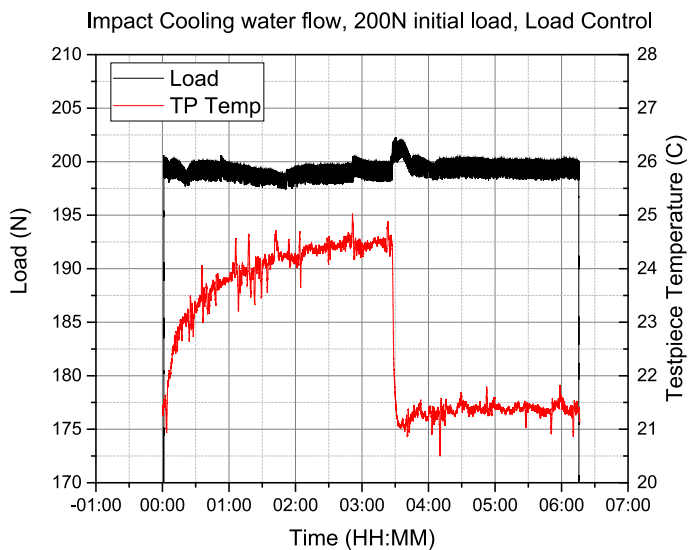


Figure 44: Impact of grip temperature on load for testpiece at 200 N in load control.

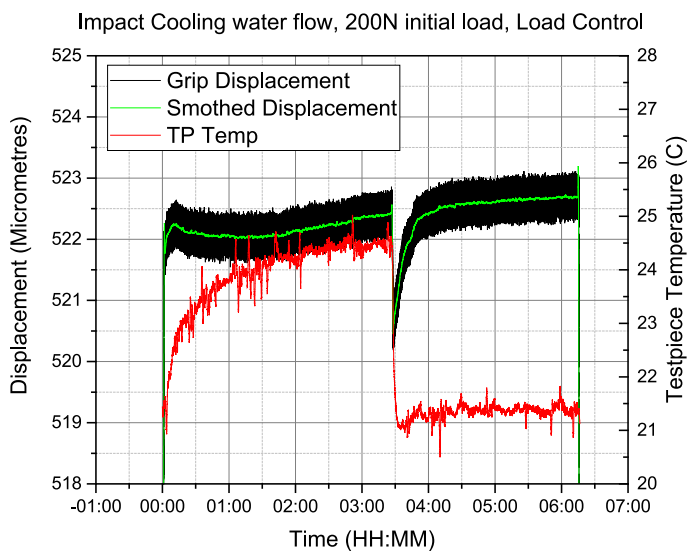


Figure 45: Impact of grip temperature on grip position for testpiece at 200 N in load control.



## Chapter VI

# Strain Analysis

- Brief introduction
- Displacement Measurements (Method A)
- Resistance Measurements (Method B)
- Strain Variation Tests
- Image Correlation (Method C)
- Instron ETMT extensometry

## Brief introduction

Load or displacement and time data are used to calculate loading, stress or displacement rates. These are important parameters to measure in tests at elevated temperatures where measured deformation rates are sensitive to how fast the test is conducted. Loads are converted to engineering stresses by dividing by the testpiece cross sectional area. If true stress is required then allowance must be made for the change in cross-sectional area during the test.

Historically, when using the NPL ETMT, testpieces have been too small to attach conventional extensometers (NB: 2) for strain measurements so mechanical properties are evaluated in one of several ways by plotting:

- load or stress against displacement – Method A
- load or stress against plastic strain (calculated from change in resistance<sup>1-7</sup>, or by assuming a nominal gauge length in the hot zone) – Method B
- load or stress against strain measured using image correlation methods – Method C.

Methods A and B are well established but method C is in the development phase.

With the advent of the Instron ETMT, a range of contact extensometry has become available.

## Displacement Measurements (Method A)

For method A the displacement measured at the grips is a combination of displacement due to the testpiece extension and displacement due to the compliance of the test rig. For the NPL 4 kN ETMT the latter has been measured to be  $0.02 \mu\text{m N}^{-1}$ . This can be checked by inserting a very stiff testpiece (either by high Young's Modulus or large cross-section that does not significantly deflect under load). This can be calculated for whatever material is inserted between the grips. Thus the corrected load / displacement curve can be determined (Figure 46).

**NB**

Instron are currently testing a miniature ceramic clip gauge with a nominal 5 mm gauge length that could be suitable for testing some ETMT geometries. Further developments are expected with this technology.

The corrected stress (or load)/displacement curve can be used to estimate proof stress by drawing a line parallel to the nominal elastic line at an offset from the origin. For room temperature tests the offset can be calculated by dividing the

displacement value by a nominal figure for the gauge length (gl). Typically this might be 18 mm for a grip separation of 20 mm. Some experimentation is necessary to establish this value.

For elevated temperature tests (say greater than 700 °C) it can be assumed that there is an effective gauge length (gl) associated with the defined central temperature. This effective gauge length (gl) needs to be established by experiment or FE modelling. An example of a curve corrected in this way using an effective gauge length of 5 mm is shown in Figure 47 from the data in Figure 46.

## Resistance Measurements (Method B)

For the resistance method for plastic strain,  $\varepsilon_p$ , is calculated from:

$$\varepsilon_p = \ln \sqrt{\frac{R_t}{R_s}}$$

where  $R_s$  is the resistance of the central region (typically 2-3 mm) at the start of the test and  $R_t$  is the resistance during the test. This equation has been derived assuming constant volume during deformation and neglecting elastic strains.

Tests analysed by this method fall into two types, dependent on the material under test.

Type I – The curve of stress/load against plastic strain is more or less vertical initially (Figure 48). Proof stress is then obtained by drawing a vertical line at the required offset strain. A comparison of the load (stress) / plastic strain from displacement (using a nominal gauge length of 5 mm) and resistance (Type I) calculations is shown in Figure 49 from a typical test at elevated temperature.

Type II – It has been observed in some tests at elevated temperature on Ni base materials that stress/strain plots obtained by this method of analysis have a pseudo elastic region (Figure 50), probably because the material's intrinsic resistivity changes with strain due to the high content of precipitates with an ordered structure. Further work is needed to explore this possibility. However, the plot can still be used to define a proof stress by drawing a line parallel to the 'pseudo' elastic region at a suitable offset strain. The slope of this line should not be used to calculate an elevated temperature Young's Modulus value (NB: 3).

**NB**

It is worth pointing out again that the resistance method measures only plastic strain and this negates the possibility of measuring the temperature variation of Young's modulus. If knowledge of this parameter is required then image correlation is a more promising method.

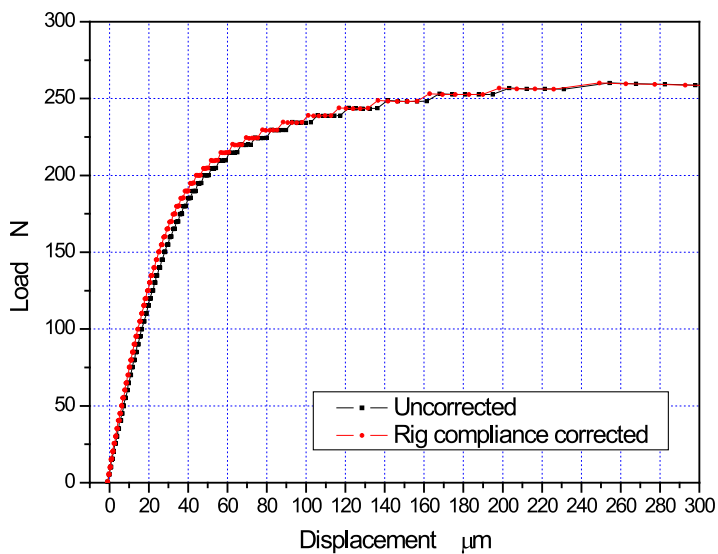


**Figure 46:** Typical load/displacement curve uncorrected and corrected for rig compliance.

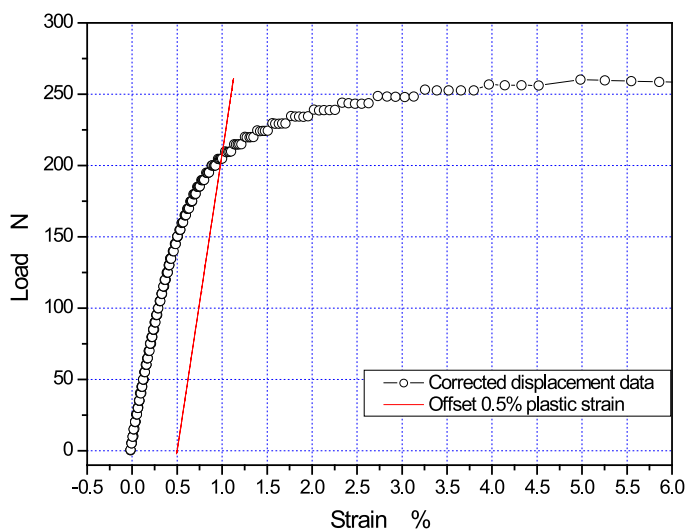


**Figure 47:** Estimated load (stress)/strain curve in test at 1000 °C indicating 0.5 % proof stress determination.

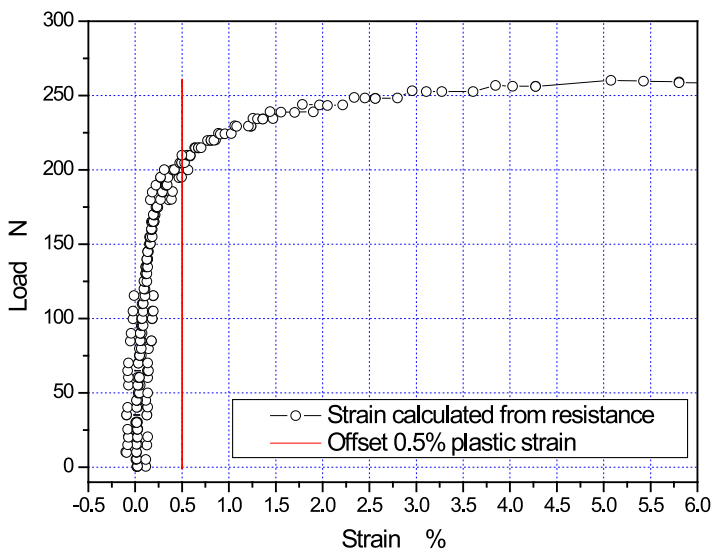


Figure 48: Type I load (stress)/strain curve from resistance plot.

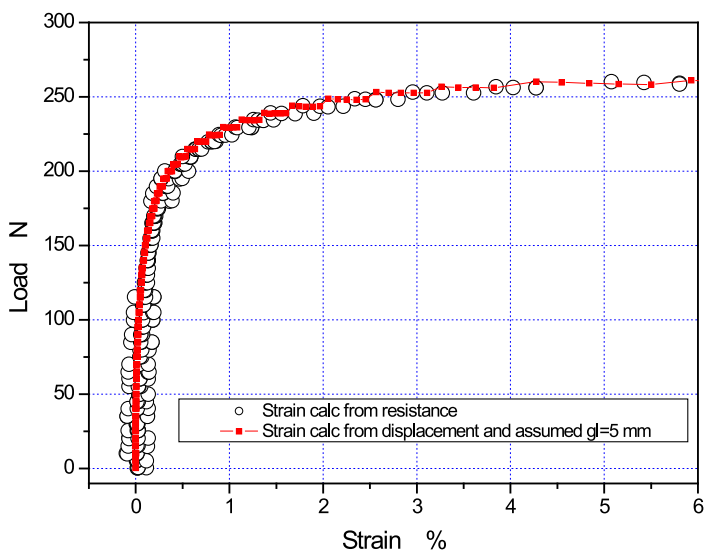


Figure 49: Comparison of load (stress)/displacement plots from displacement and resistance calculations.

Resistance values are calculated from measurements of voltage and current. Typical heating currents are around 50-150 A and are controlled by the dc power supply to 4 significant figures. Typical voltages are around 100 to 200 mV and are also measured to about 0.1 % uncertainty. Consequently, the resistance ratio and thus nominal strain values using this technique have a similar uncertainty.

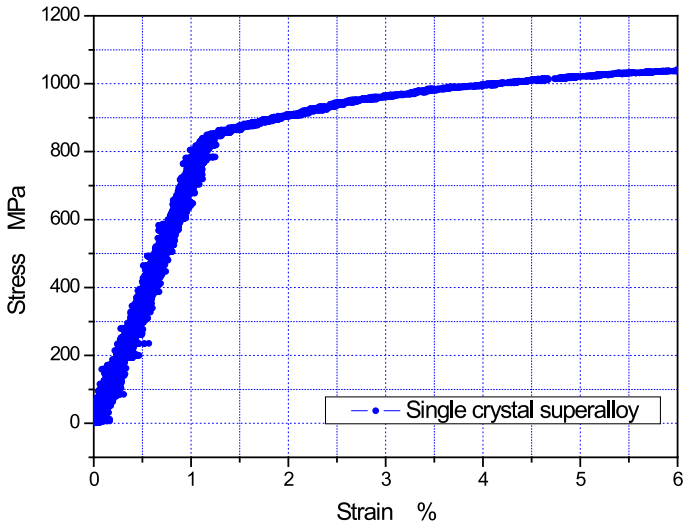


**Figure 50:** Type II Stress/strain curve from resistance plot shown 'pseudo' elasticity in test at 600 °C on superalloy single crystal material.

However, this is not realised in practice in the NPL ETMT because, most testpieces, when hot, are within a box in which an inert gas, usually Argon, is stirred with a small fan. The resulting convection currents require the heating control system to continually vary the current to maintain the required temperature. When the fan is off, the variability is much less and more accurate resistance (strain) measurements are possible. Typical graphs showing this effect are shown in Figure 51. For the Instron ETMT where a vacuum chamber is used this uncertainty is not present and the resulting strain measurements are more precise.

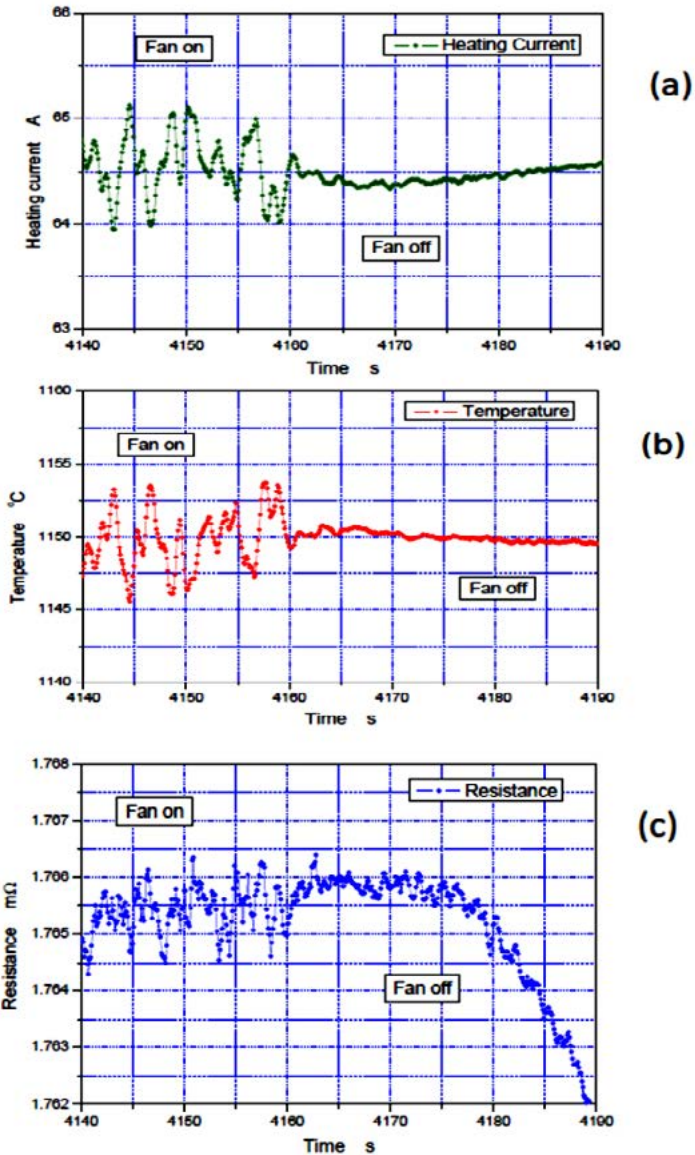


**Figure 51:** (a) Showing the effects on heating current, temperature and resistance of having fan off or on: Heating current (with and without stirred argon) and resulting temperatures (b) (with and without stirred argon) for a metallic testpiece at a nominal temperature of 1150 °C just at the start of a compression test (where resistance (c) starts to decrease).

## Strain Variation Tests

In order to confirm the plastic strain calculations from the resistance method, a waisted sample of 304 stainless steel was deformed in tension at room temperature. Miniature tension testpieces were prepared by electrodischarge machining (EDM) and light diamond grinding followed by an anneal at 1100 °C for 30 min. The testpieces were asymmetrically waisted (Figure 52) in order to provide strain gradients within a single sample following deformation. This was performed at a loading rate of 10 N/s. The tensile test was conducted in two parts. The testpiece was deformed in tension and then unloaded at a plastic strain of about 15 %. It was then retested to give a total strain of about 35 %. Before the test an array ( $3 \times 100$ ) of microhardness indents was placed on the surface of the sample using a load of 1 N with a spacing in the direction of strain of 0.2 mm and 0.2 mm in the transverse direction. The spacing of the indents was measured after the deformation test in order to calculate local strain and its variation along the tapered testpiece. Plastic strain in the central 2 mm region of the testpiece was also measured using the resistance method<sup>1-7</sup>.

The tensile test results are shown in Figure 53 plotted as stress against strain. Plastic strain was calculated from the resistance data. Elastic strain was calculated assuming a Young's modulus value of 195 GPa and added to plastic strain to give a total strain value. The results are shown in two parts corresponding to the first and second cycles of the test. The stress/strain curves agreed well with macroscopic test data. The variation of strain along the testpiece as measured by the spacing of the microhardness indents are shown in Figures 54 and 55 respectively for the first and second parts of the deformation cycle.

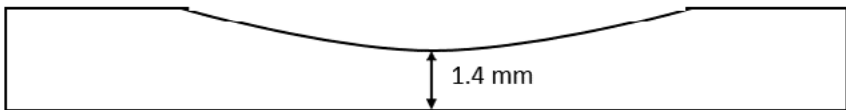


**Figure 52:** Schematic diagram of asymmetrically waisted tensile testpieces.

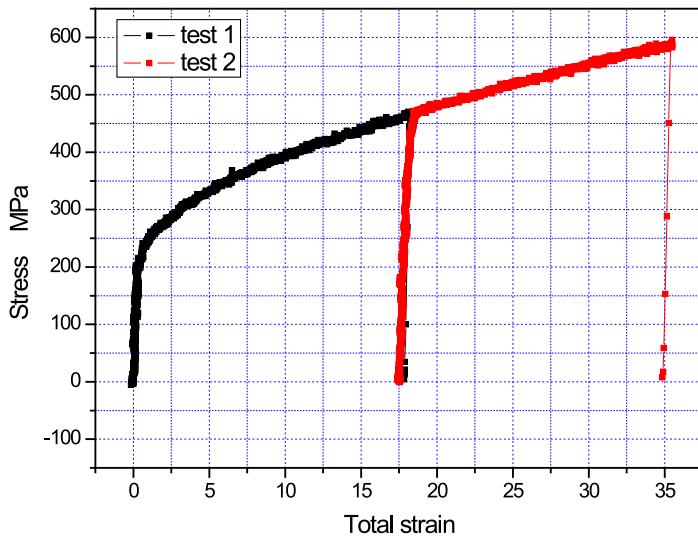


**Figure 53:** Tensile stress/strain curves from a waisted 304 stainless steel testpiece.

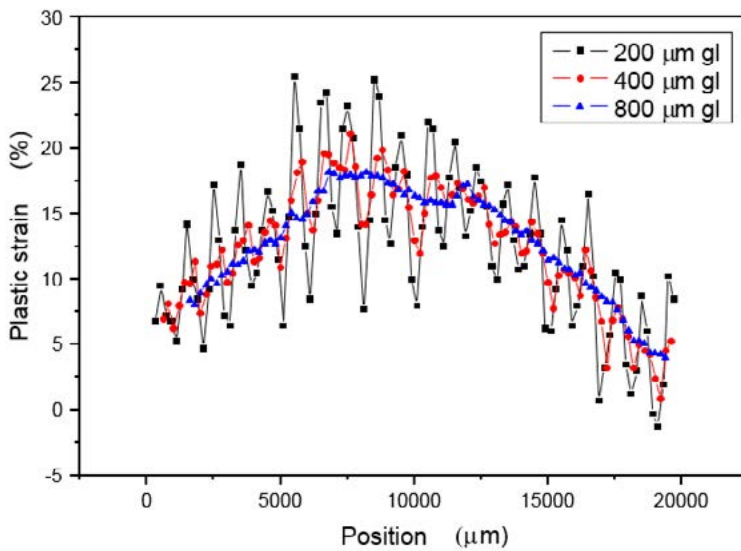


**Figure 54:** Strain variation along testpiece after strain to about 18 %.

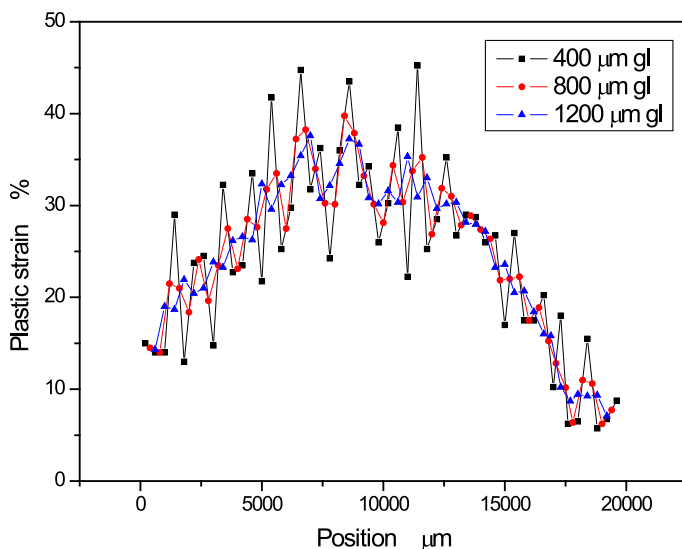


Figure 55: Strain variation along testpiece after strain to about 35 %.

The strain values were calculated using sequential gauge lengths (gl) of 200, 400, 800 and 400, 800, 1200  $\mu\text{m}$  respectively. It can be seen that locally there was considerable point to point variation (NB: the grain size was more than 100  $\mu\text{m}$ , so gauge lengths of 200 or 400  $\mu\text{m}$  might be expected to reveal grain to grain variation in deformation). This variation was smoothed out for larger values of the gauge length. The central strain values of about 17.5 % and 35 % agreed very well with those measured using the resistance method.

## Image Correlation (Method C)

Digital image correlation techniques (DIC) work by comparing images of a component or testpiece at different stages of deformation and tracking blocks of pixels to measure surface displacement<sup>14-21</sup>. In this way full field 2D and 3D deformation vector fields and strain maps can be acquired. The position of the centre of the pixel blocks is determined to sub-pixel accuracy over the whole image using sophisticated correlation functions, from which the vector and strain components can be calculated.

Depending on the sub-pixel algorithms used, the claimed resolution for 2D DIC

approaches 0.01 pixel for displacement measurement. In practice, however, factors associated with the experimental set up, such as the quality of lighting, camera noise, lens distortion, the scale and type of pattern or coating on the testpiece etc, will degrade the images and affect the accuracy of the DIC calculations, so 0.1 pixel is probably more realistic. This means that, for a miniature testpiece with a gauge length of 10 mm and a camera resolution of 1 Megapixel ( $1000 \times 1000$  pixels), if the gauge length fills the field of view, DIC will be able to resolve displacements to between 0.1-1  $\mu\text{m}$  resolution. For full field studies the strain resolution is typically 100  $\mu\epsilon$ , so is good for plastic deformation studies but less reliable at small strain values, and challenging for elastic property measurement. The absolute strain resolution depends on the camera resolution, field of view, scale of the image relative to the pattern on the surface, the uniformity of strain in the testpiece, the number of pixels in each pixel block, and the gauge length over which the strain is calculated. Choices must be made about the size of the pixel blocks (also described as ‘facets’, ‘windows’, and ‘sub-sets’) as this effectively defines the gauge length for strain measurement and has an important influence on the spatial resolution and accuracy of the strain and displacement measurement. Generally, for uniform displacement fields, larger windows give better strain accuracy, whilst smaller windows give better spatial resolution. Table 7<sup>18</sup> summarises the calculated errors and uncertainties that might be expected with different window sizes. An advantage of analysing the images with DIC is that, once the images have been captured, they can be reprocessed using different window sizes and DIC parameters to highlight different features on the testpiece.

| Effect of DIC window on displacement and strain measurements |                                         |                                                          |
|--------------------------------------------------------------|-----------------------------------------|----------------------------------------------------------|
| Size of DIC window (pixels)                                  | Accuracy of calculated vectors (pixels) | Accuracy of calculated strain values (with 50 % overlap) |
| 128 × 128                                                    | 0.01 to 0.03                            | 0.094 %                                                  |
| 64 × 64                                                      | 0.02 to 0.05                            | 0.30 %                                                   |
| 32 × 32                                                      | 0.05 to 0.2                             | 1.25 %                                                   |
| 16 × 16                                                      | 0.1 to 0.3                              | 5.0 %                                                    |

**Table 7:** Effect of the DIC analysis ‘window’ size on the calculated accuracy of the displacement and strain measurements<sup>18</sup>.

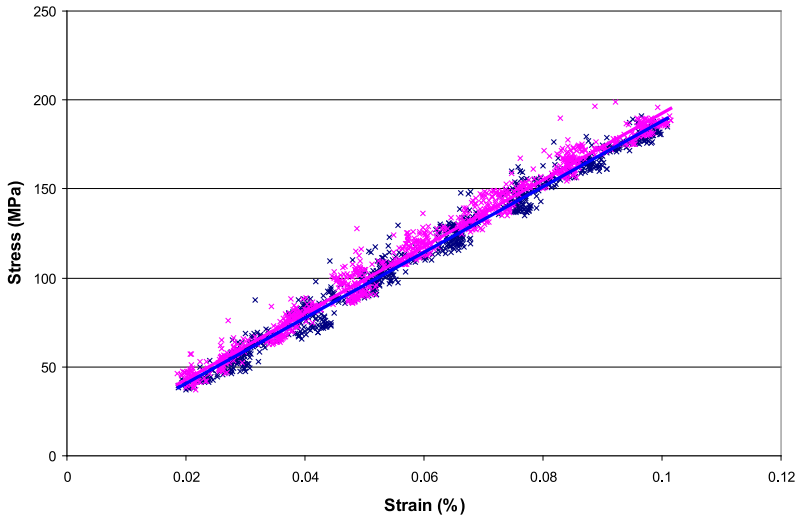


**Figure 56:** Real time DIC measurements on a miniaturised tensile specimen in the NPL ETMT, showing the linear fit to the data for elastic modulus calculation.

DIC requires a random pattern on the sample, and different approaches can be used to generate this, including conventional polishing and etching of the sample to reveal grain structure and texture, the application of a pattern to the testpiece using a fine airbrush or applying light, random scratches to the surface. The scale of the pattern relative to the pixel size is important, and a consideration for measurements made under the microscope or SEM where the application of a very fine random pattern is difficult. Uniform illumination is also essential and a dedicated local light source should be used to ensure consistency of illumination throughout the test. LED ring-lights offer a good solution particularly in combination with a macro or telecentric lens.

It should be noted that using a conventional small scale test machine with a fixed crosshead can lead to difficulties with DIC, as the original part of the image can move out of the field of view and the correlation breaks down. This can be overcome by using a larger field of view, multiple cameras that overlap, or designing a test machine where both grips move and the central gauge length of the testpiece remains at the centre of the image.

DIC is generally used as a full-field application, but some variants of DIC software use the tracking of discrete markers or features for measuring displacement and strain.

Because the processing power required is much reduced, there is scope for real time measurement of displacement and strain over several pairs of markers on the testpiece. Measurements have been made on miniaturised tensile specimens in the NPL EMTT used by Peterson *et al.*<sup>16</sup> to examine the potential of the DIC technique for measuring elastic modulus. Figure 56 shows results at NPL from a series of tests on a stainless steel 304 alloy. Although there is some scatter and noise in the data, due in part to the load control, the fit through the data gives values of modulus of 185 GPa and 187 GPa for the two specimens, and reasonable strain resolution ( $10\text{ }\mu\epsilon$ ) below that typically expected for full field DIC.

DIC has been used successfully for tracking markers on the testpiece with the NPL ETMT at temperatures up to  $800\text{ }^{\circ}\text{C}$  without special cameras or IR filters. At higher temperatures, there is an increased likelihood that thermal currents and airflow in front of the lens will lead to distortion of the image and breakdown of the correlation; techniques are being developed based on high-speed sampling to overcome this. Oxidation should be avoided by testing in vacuum or inert atmosphere to prevent changes in the surface appearance and a breakdown in the correlation. Normally with DIC the images are compared relative to the initial (unloaded/starting) state, but if there are large deformations or changes to the surface condition, it is possible to do the correlation between successive images and generate cumulative strain fields. Potential issues with oxidation of testpieces or surface diffusion effects changing the appearance of the testpiece surface with time can be overcome to some extent by using a robust and frequent monitoring regime to ensure that the correlation does not break down between images. This may not work in all cases however.

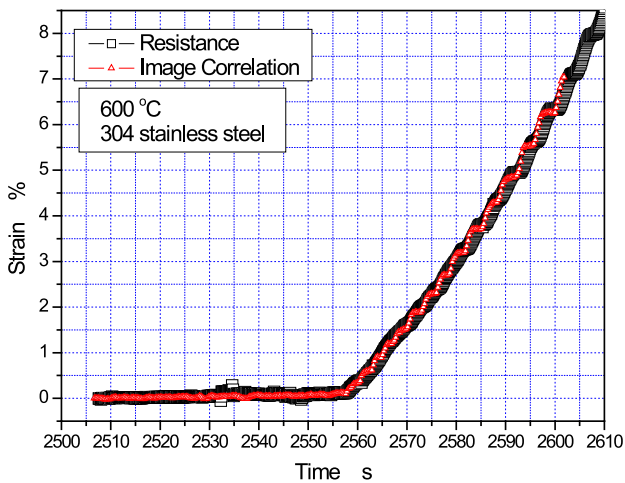


Figure 57: Comparison of strain measured by image correlation and resistance changes<sup>23</sup>.

Another example confirming the utility of either resistance or image correlation techniques for the measurement of plastic strain was obtained in a tensile test conducted on 304 stainless steel at 600 °C where strain was measured using Digital Image Correlation (DIC) concurrently with the measurement of change in resistance. Figure 57 shows excellent agreement between the two methods, confirming that DIC has good prospects as a non-contact method at elevated temperatures, and that resistance changes can be taken to be an accurate technique for assessing strains. In further support of using resistance changes for measuring strain, it should be noted that typical increases in resistivity due to high dislocation densities are very small (0.1 nΩm for 10 % strain in Nickel<sup>22</sup>) compared with the resistivities usually measured in elevated temperature tests (500 nΩm at 600 °C for steels).

## Instron ETMT extensometry

To date, three types of contact strain measurement devices have been supplied with the Instron ETMT:

- **Clip-on extensometer**

This extensometer has a 12 mm gauge length, with a range of  $\pm 2.5$  mm. It is not connected directly to the specimen, since this would result in current flowing through the extensometer. Rather, this extensometer is used to measure the grip separation of the load string via ceramic tubes.

- **LVDT**

As with the Clip-on extensometer this device does not connect directly on to the specimen, since this would result in current flowing through the LVDT. The LVDT is used to measure the grip separation of the load string via Teflon bushes.

- **Extensometer**

This extensometer, Figure 58, has a 5 mm gauge length, and displacement range of +2.5 / -1.5 mm. This extensometer uses RA ceramic extension rods and can this connect directly to the specimen. Testpiece deformation is measured between the two ceramic rods. This extensometer is the only choice if TMF or LCF testing is to be carried out without applying post processing.

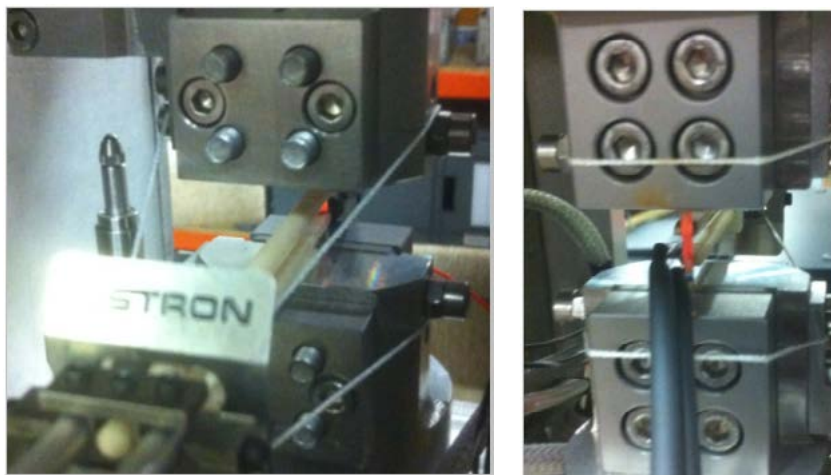


**Figure 58:** Instron 5 mm gauge length extensometer fitted to Instron ETMT.

Extensometers (1) and (2) require post processing of the recorded data if strain is to be extracted from the specimen extension. It must be noted that, due to the method DC heating between water cooled grips, there is a large parabolic temperature gradient, as shown in Figure 12 ("Chapter IV", 'Temperature Measurement', p. 27). Standard LCF and TMF testing requires the temperature gradient between for the strain measurement region to be within  $\pm 5^{\circ}\text{C}$ .

All three extensometers can be automatically (electrically) calibrated. If traceable calibration is required, the strain devices can be verified by using calibrated external reference transducer(s). Care must be taken when balancing (zeroing) the Strain sensors. The linear displacement range of the transducers is short and testing could be undertaken in a relatively non-linear portion of the transducer.



## Chapter VII

# Reference Tests

- Comparison tests on a reference material
- Comparison tests at high temperatures

## Comparison tests on a reference material

Because the use of resistance changes for strain measurements is unconventional, a series of comparison tests on a tensile reference material (Nimonic 75) were performed. The results of ETMT uniaxial tests at 600 °C using the resistance method for strain measurement are shown in Figure 59 compared with the results of tests on conventional macroscopic testpieces that used extensometers to measure strain. The agreement was good and confirmed that plastic strain calculations through monitoring changes in resistance is an acceptable measurement method. The steps in the ETMT curve are thought to be real, reflecting progressive serrated yielding events<sup>24, 25</sup>.

## Comparison tests at high temperatures

To further address the concern of reproducibility, experiments were also conducted to obtain mechanical data on a Ni-base alloy, IN718, which could then be compared with a recent review of the high temperature strength of this material<sup>26</sup>. The miniature ETMT test data was obtained by heating the testpiece to a suitable temperature (in the range 950-1200 °C) then applying a constant stress (in the range 5-100 MPa) and measuring the resulting strain rate (which was reasonably constant after a small primary region). Strain was calculated from change in resistance of the sample. The results obtained using the miniature system are shown in Figure 60 where stress is plotted against the Zener-Hollomon parameter (obtained using an activation energy of 300 kJ mole<sup>-1</sup>). The solid bold line is the best fit to the Lewandowski and Overfelt<sup>26</sup> reference data. The data fit line is extended with a fainter line to lower values of applied stress. It can be seen that the data from the miniature system agree very well with this best fit reference data over a wide range of stress and temperature. It must be remembered that all the ETMT results were obtained on a *single* small sample tested over a period of 2-3 h. The data at the highest temperatures were obtained first of all. Clearly there is little history dependence in the results; i.e. the material is not significantly work hardening/softening between individual tests.

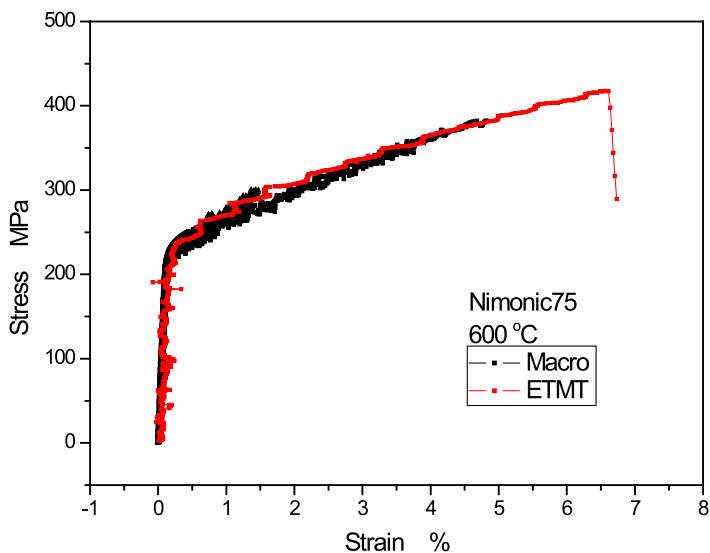


Figure 59: Comparison of miniature ETMT uniaxial tests with macroscopic tests at 600 °C on a Nimonic 75 reference material.

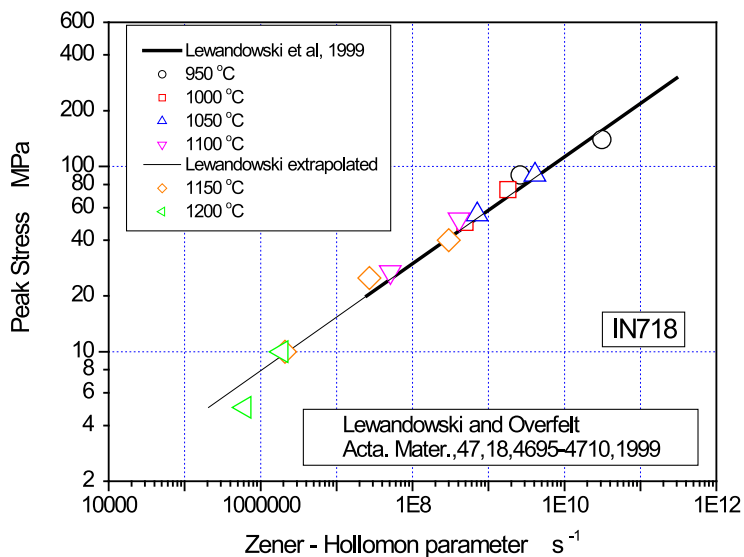


Figure 60: Comparison of ETMT Zener-Hollomon parameters obtained on a Ni-base alloy with reference data on the same alloy<sup>2,26</sup>.



## Chapter VIII

# Case Studies

- ETMT Drivers
- Case Studies

## ETMT drivers

The ETMT multiproperty or TMF test system variants have many advantages over conventional machines:

- Smaller samples allow costs to be minimised in testpiece preparation
- Cooling rates can be easily controlled in some cases to quite high values – up to  $100\text{ }^{\circ}\text{C s}^{-1}$  is possible. Heating rates can also be fast and accurate
- Property distributions in heterogeneous materials can be investigated efficiently
- The system lends itself to multitest capabilities in single samples or *in situ* thermomechanical cycles
- Physical properties, such as electrical and thermal conductivity can be evaluated as well as mechanical properties
- There is a possibility of working in controlled atmospheres or vacuum
- Samples can be tested after extraction from used components to assess remnant life
- New alloys can be quickly assessed to shorten development cycles when available material may be in short supply
- There is good visual access to the testpiece for image correlation and other non-contact strain techniques.

In this section a number of case studies provided by attendees to the Interest Group meeting at Instron on Wednesday 23 April 2014 are used to highlight some recent uses of the system with a view to indicating particular metrology issues. The meeting was initiated by contributions from Dr P. Brown (Rolls-Royce) and Dr S. Moseley (Hilti AG) setting out important aspects of processing materials where miniaturised testing procedures can make a useful contribution.

Many engineering materials and manufacturing procedures come under scrutiny of process modelling groups at industrial organisations. It is challenging to model materials, but modelling can shortcut a huge array of physical trials which are expensive, time consuming and can waste valuable raw materials. Properties of materials on the micro-scale affect the macro-scale of engineering.

Modelling can help to pinpoint when actual physical trials are needed which aids manufacturing by improving processes and material selection for various products. Product design can be advanced as well ensuring the product is manufactured at an acceptable cost and with the required performance level. Process modelling group activities also entail work on materials at the intermediate stages of manufacturing which requires an even larger volume of testing as they often assess the most

extreme ranges of conditions. Working at extremely high temperatures often close to the melting point and with cooling rates dependent on the microstructure, researchers continually look for ways to link macro-scale properties to underlying microstructures and chemistry. Physical testing is clearly expensive and there is a need to design faster to meet requirements. New materials are important but there is also a requirement to get more out of the materials already in service.

The ETMT can be used on smaller samples which reduces cost and provides opportunities for more testing. In order to certify a material for use engineers require a large volume of test data which is collected in a database. Engineers use this information for design specification.

## **Instron ETMT Customer Requirements**

Users of general material test machines generally fall in to two categories:

- basic, and just want to 'press a button' and the data is there in a format easy to use
- experienced scientists or engineers who want and need to extract various specific, unique properties which require more than the 'standard application / test'.

ETMT users need to have a very good understanding how the ETMT system works and to understand its limitations and compromises in order to maximise its capabilities. As a consequence the ETMT favours the second category of user.

ETMT customers need tests for mechanical and physical material properties. Over the last few years Instron have been asked to provide calculations to allow measurement of the following properties:

- electrical resistivity as a function of temperature
- electrical resistance
- thermal conductivity
- electrical power
- thermal expansion coefficient.

When users drive the ETMT using Wavematrix as the application programme, the above properties can be directly or indirectly calculated using C# Software routines. The C# routines are user programmable and are calculated during the testing.

The use of the ETMT system is gradually being seen as a method to carry out true 'miniature' TMF testing. Instron has been looking at the use of a small (5 mm gauge length) conventional ceramic rod extensometer to develop strain data. Using the

ETMT as a TMF test system would allow small specimens to be tested in the same manner as conventional TMF specimens. There is much work to be carried out to correlate the conventional TMF macro sized specimens with the ETMT miniature specimens, but research and interest in this field has commenced.

Historically, the issues with the ETMT that have inhibited its uptake for TMF testing are:

- lack of a uniform temperature region in the centre of the specimen
- lack of a direct strain measurement jig
- ease of test set-up and interpretation of results.

## Case studies

### NPL and Hilti – Bryan Roebuck (NPL) and Steve Moseley (Hilti)

The rationale for this research was to investigate the flexibility of the ETMT system by conducting various tests at very high temperatures on representative hardmetal tool materials with special emphasis on the difference between tensile and compressive stress states. For example, a carbide tip on a steel masonry drill bit has different kinetic energy and cooling characteristics at the join because the elastic and mechanical properties between the two materials are diverse. Representative WC/Co hardmetals were shown to be considerably stronger in compression than tension (Figure 61); though microstructural studies to highlight the underpinning mechanisms remain to be identified. The compression/tension asymmetry was evident both for constant creep loads and monotonically increasing stresses. Flow stress curves could be fitted to power law functions allowing the temperature dependence of the stresses to be codified in an efficient manner for modelling. Clearly there are metrology issues associated with undertaking multiple tests on one sample. Water cooled grips were used to hold samples in place as water is important for heating and cooling samples and maintaining stable temperature profiles. By testing in compression/ then tension; and then tension followed by compression, it was discovered that the behaviour in tension is not the same as behaviour in compression. Strain values, however, may have possibly varied because of the history-based response of the metal and modelling is recommended as a way to investigate this further. In essence, the lesson from this case study was that single testpieces can be used for multiple tests provided the accumulated strains are kept to a few percent, but some trials are needed to establish this approach for each material of interest. It is possible that in between tests an annealing process could be used to allow greater strains to be applied.

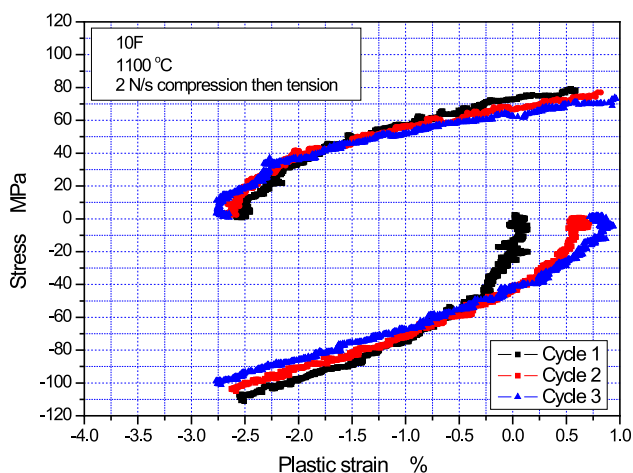


**Figure 61:** Asymmetric flow stresses in tension and compression at 1100 °C in tests on a hardmetal.

## University of Cambridge – H.T. Pang, Howard Stone and Cathie Rae

The University of Cambridge are using the ETMT for temperature, strain and displacement monitoring in tests on Ni base alloys with some tests reaching 1200 °C.

For mechanical tests such as tensile/ compression, LCF, TMF and creep typical sample dimensions are  $3.0 \times 1.0 \times 40$  mm with the required testpiece geometry as outlined in Figure 2 in section 3.1. Tensile/ compressive tests and tensile tests on the ETMT generally require displacement-controlled tests between 700 and 1100 °C.

For LCF on the ETMT the tests have been performed at 750 °C under stresses of 900 to 1150 MPa (Figure 62). Displacement control and out-of-phase (OP) loading have been used for TMF studies using the ETMT. It was noted that with superalloys testpieces could fracture off-centre (at lower temperatures) when the central temperature was around 750 °C (associated with the highest strength). True stress/strain curves were found difficult to assess once testpieces started necking just before fracture and locally the temperature could increase faster than could be accommodated by the PID control system.

Finally it was suggested that experiments with different, lower thermal conductivity, grip materials might be a useful avenue to explore to reduce temperature gradients in the test samples.

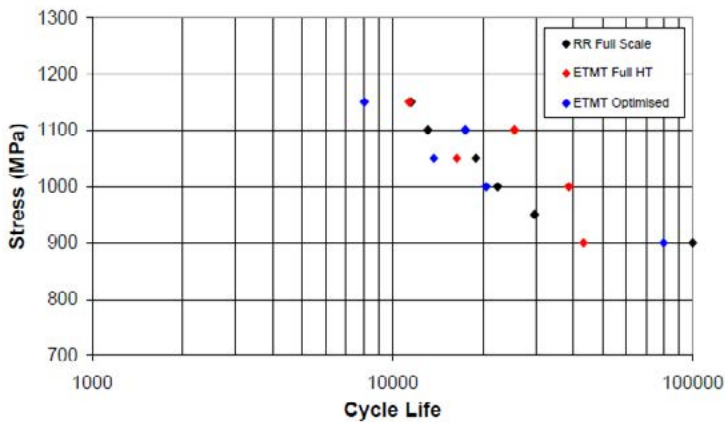


Figure 62: LCF studies comparing ETMT with full scale geometries.

## University of Manchester – Philipp Frankel, Philip Whitters and Michael Preuss

The University of Manchester is investigating Zirconium alloys<sup>27</sup> for fuel cladding in nuclear reactors and have used the ETMT to evaluate oxidation and irradiation effects (Figure 63). The anisotropic growth of fuel assemblies under irradiation is a concern for the nuclear industry as any bowing of any fuel rods could potentially affect the insertion of control rods needed to regulate the reactor core. Varying the material heat treatment can affect grain texture with the possibility of reducing irradiation growth strengths – the ETMT has been used to help answering how and why phase transformations affect microstructure in unpredictable ways. From a metrological viewpoint it was shown that recrystallization gradients with testpieces could be used to advantage (with modelling) to increase the efficiency of data measurements as a function of temperature.

Furthermore, the rapid heating kinetics of the ETMT could be used to advantage for understanding transients in oxidation studies and also the effects of applied loads on the kinetics of the oxidation.

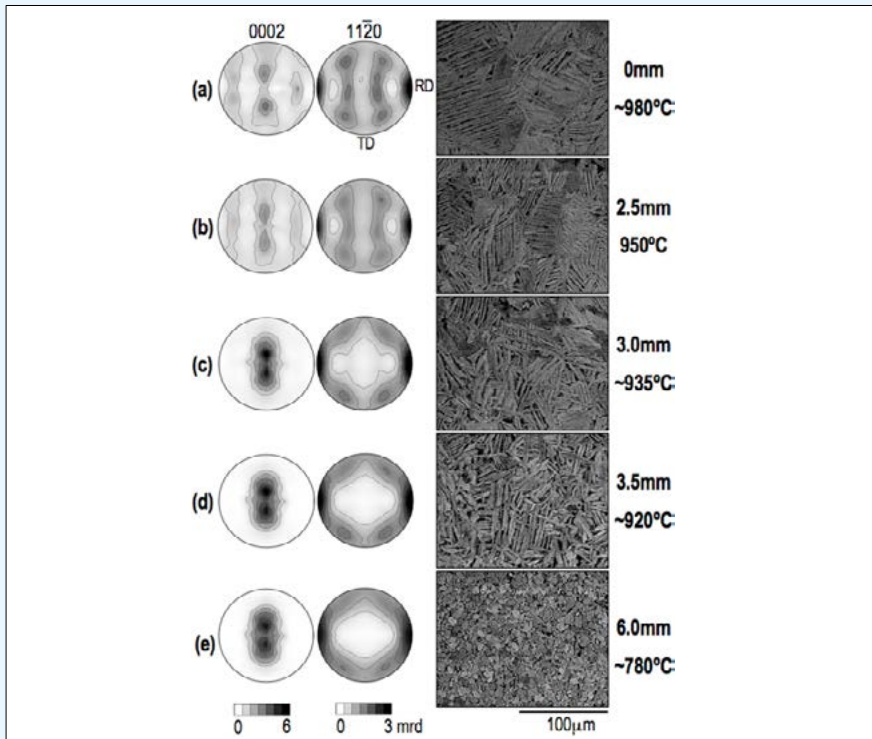


**Figure 63:** The texture evolution of cold-rolled Zircaloy-2 sheets during  $\alpha \rightarrow \beta \rightarrow \alpha$  phase transformation was characterised in situ by using high energy synchrotron X-ray diffraction. Recalculated pole figures and backscatter electrons micrographs from different positions after heat treatment. (a) corresponds to the centre of the sample<sup>1</sup>.

## Oxford University – Roger Reed, Enrique Alabort and Fauzan Adziman

Professor Reed's group has extensive experience of using the ETMT over several years. These include testing materials to be used in extreme environments; developing new alloys; stress-jump tests; recrystallisation kinetics of single crystal alloys and superplasticity tests. Conventional tests are expensive and time consuming and may not be appropriate for the depth of study, funding and materials available.

In the past, Oxford have studied the recrystallisation kinetics of single crystal alloys comparing the ETMT results with conventional methods in two studies a) the re-crystallisation of single crystal turbine blades and b) the study of strain-induced recrystallisation in CMSX-4.

Studies in tensile testing on the ETMT outlined by Cox *et al*<sup>28</sup>:

- Specimens of  $1 \times 1 \times 40$  and  $1 \times 2 \times 40$  mm were machined from single crystal bars
- Up to 4 % strain was imposed, at ambient, 950, 1025 and 1125 °C (corresponding to temperatures at which flow stress is 600, 400 and 200 Mpa, respectively) and strain rate  $4 \times 10^{-4} \text{ s}^{-1}$
- Annealing (Figure 64) followed to promote recrystallization.

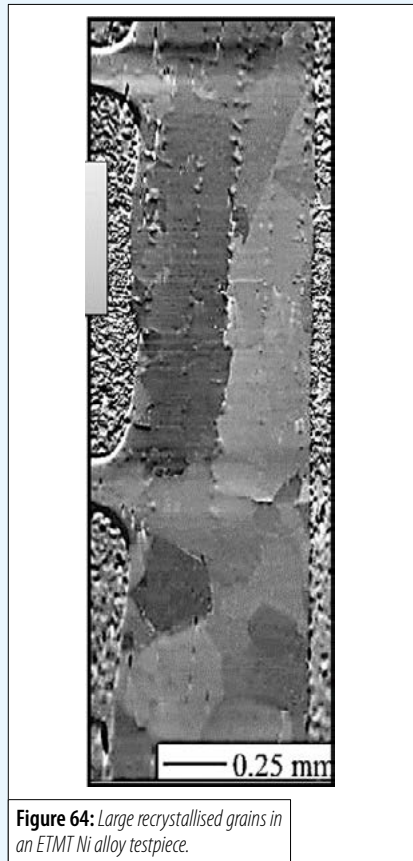


**Figure 64:** Large recrystallised grains in an ETMT Ni alloy testpiece.

The results showed that plastic strain is more damaging when introduced at higher temperature which highlights importance of understanding the form of dislocation damage. No recrystallisation for annealing occurred at 1275 °C or less.

Panwisawas *et al*<sup>29</sup> continued the study of strain-induced recrystallisation in CMSX-4. The influence of mould geometry on induced plasticity was studied using conventional tensile test methods. Only one bar per casting could be extracted which limited the number of bars that could be studied.

Both studies showed the advantages/disadvantages of different testing methodologies to study strain-induced re-crystallisation. Conventional tensile tests conducted in 2013 by Panwisawas *et al*<sup>29</sup> showed that the matrix of results was limited by material availability; in Cox's ETMT test in 2003 a well-defined recrystallisation boundary was visible in a complete result matrix. The ETMT offers a more efficient use of material for applications where availability is a limiting factor.

At the present time Oxford is investigating the use of the ETMT in a series of tensile tests to study superplasticity in Ti-6Al-4V. The 'alloy by design' cycle links tensile properties to chemical composition so multiple compositions need to be tested and validated. Conventional mechanical testing requires pre-test prediction of mechanical properties, fabrication of the new alloy and machining test samples before it can be tested – this can take approximately three months and require third party suppliers at increased cost. Using the ETMT equipment the same pre-test prediction of mechanical properties, fabrication of the new alloy and machining test samples still have to be done, but the samples are only 4 cm long and can be machined on an EDM. The need for external suppliers is reduced thus decreasing expenditure and the whole process can take place in-house. It is fast as well – from design to validation in potentially one week.

For mechanical testing of Ti-6Al-4V at superplastic conditions, Oxford's preliminary experimentation suggested good correlation between mechanical responses measured using ETMT and more conventional approaches (Figure 65).

However, preliminary superplasticity tests showed significant oxidation on the sample surface which needs to be fully avoided for surface studies. The ETMT might offer significant advantages in the alloy-by -design cycle, particularly in terms of speed and cost of the iterative process, but for Ti alloys surface oxidation must be prevented.

Future studies at Oxford are likely to involve thermomechanical fatigue (TMF) stress states. TMF deformation mechanisms are highly composition-dependent.

Different fracture mechanisms are observed for different conditions and super alloys so Oxford researchers will be looking at ETMT miniaturisation effect in TMF tests. Scientists will need to address the representative volume problem and size effect of the ETMT samples in TMF. Reproducibility of micro- and macro-mechanics using ETMT TMF is particularly important for polycrystalline nickel-based superalloys where the number of grains across the specimen thickness might be relatively small.

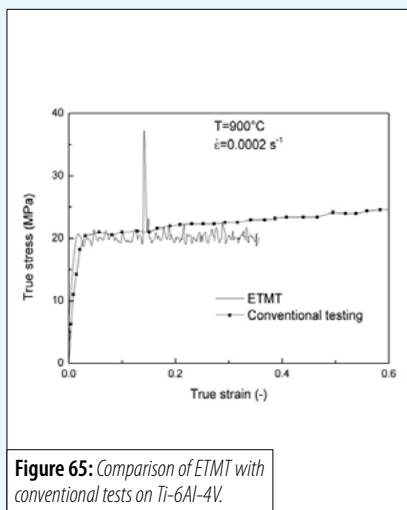


**Figure 65:** Comparison of ETMT with conventional tests on Ti-6Al-4V.

## NPL –

### Bryan Roebuck and Matthew Brooks

Two further case studies from published research at NPL are included to highlight additional metrology issues.

#### Resistivity measurements for phase transformations

It is important in every material under study to have some information on the temperature dependence of the resistivity of that particular material. This is especially the case if the resistance method has been used to calculate strain values in a mechanical test since the method depends on the assumption that the resistivity remains constant at that particular temperature. For some materials, like pure Pt for example, the resistivity increases monotonically with increasing temperature and there is no phase change but in many engineering materials, like Ni superalloys, for example, the resistivity changes quite significantly with temperature (Figure 66) as the volume fraction of the precipitate phase (Ni<sub>3</sub>Al) decreases at high temperatures. Also in many Aluminium alloys at intermediate temperatures the precipitates are small and the size can vary with time at a particular temperature giving rise to time dependent changes in intrinsic resistivity. Clearly in this case the temperature profile prior to and during any mechanical test is of great importance. Suitable experiments in the ETMT to evaluate these factors is therefore desirable in order to fully understand the results of any mechanical tests at elevated temperatures.

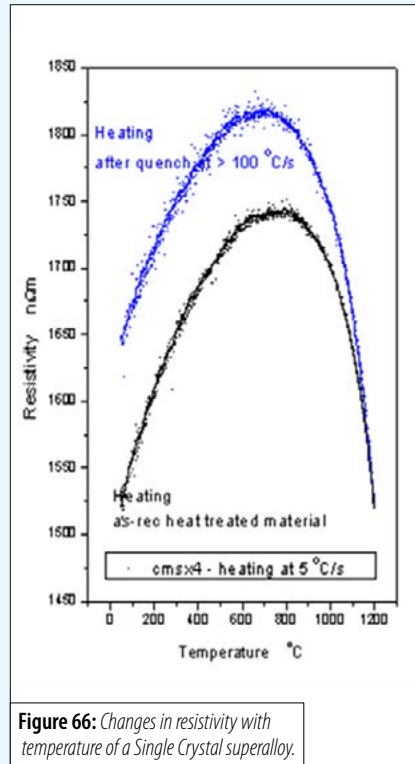


**Figure 66:** Changes in resistivity with temperature of a Single Crystal superalloy.

#### Thermomechanical tests on Nimonic 90

In a European project to develop a TMF standard<sup>30</sup> NPL contributed a study<sup>31</sup> of miniaturised testing of a Ni base alloy (Nimonic 90) which was characterised in several ways; by comparative measurements of temperature using either pyrometry or thermocouple technology; by measurements of resistivity to better understand

internal phase changes (Figure 67) and provide background for interpreting resistance measurements; through uniaxial mechanical tests to provide information on stress versus strain behaviour at temperatures relevant to the thermomechanical fatigue tests (TMF) and through miniature TMF tests for contrast with the macroscopic European interlaboratory comparison. This study helped address several metrological aspects of using the ETMT on small specimens, in particular the identification of damage (crack) development (Figure 68) and load drop rate as samples approached final failure.

An important additional observation was the measurements of resistance changes seemed to give an earlier warning of impending failure than following the progress of the drop in maximum load.

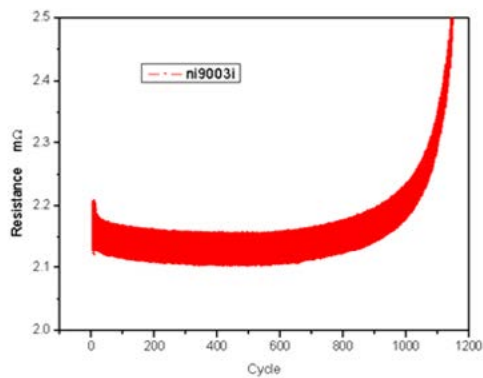


**Figure 67:** Increase in resistance in final stages of TMF test.

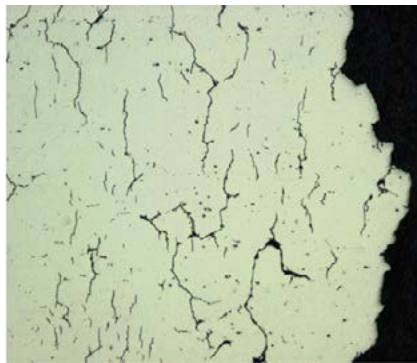


**Figure 68:** Crack morphology in miniature fractured TMF sample of Nimonic 90.

## Advanced Forming Research Centre and University of Strathclyde – Himanshu Lavani and Paul Blackwell

Case studies carried out on the ETMT system at AFRC include:

- **Thermal cycles on Ti64 and Ti6246 alloys**

Objective of this study was to look at phase transformations in Ti64 and Ti6246 alloys by simulating heating and cooling cycles above and below beta transus. A range of heating and cooling rates were incorporated and the fraction of re-crystallisation was investigated for various heat treatment cycles.

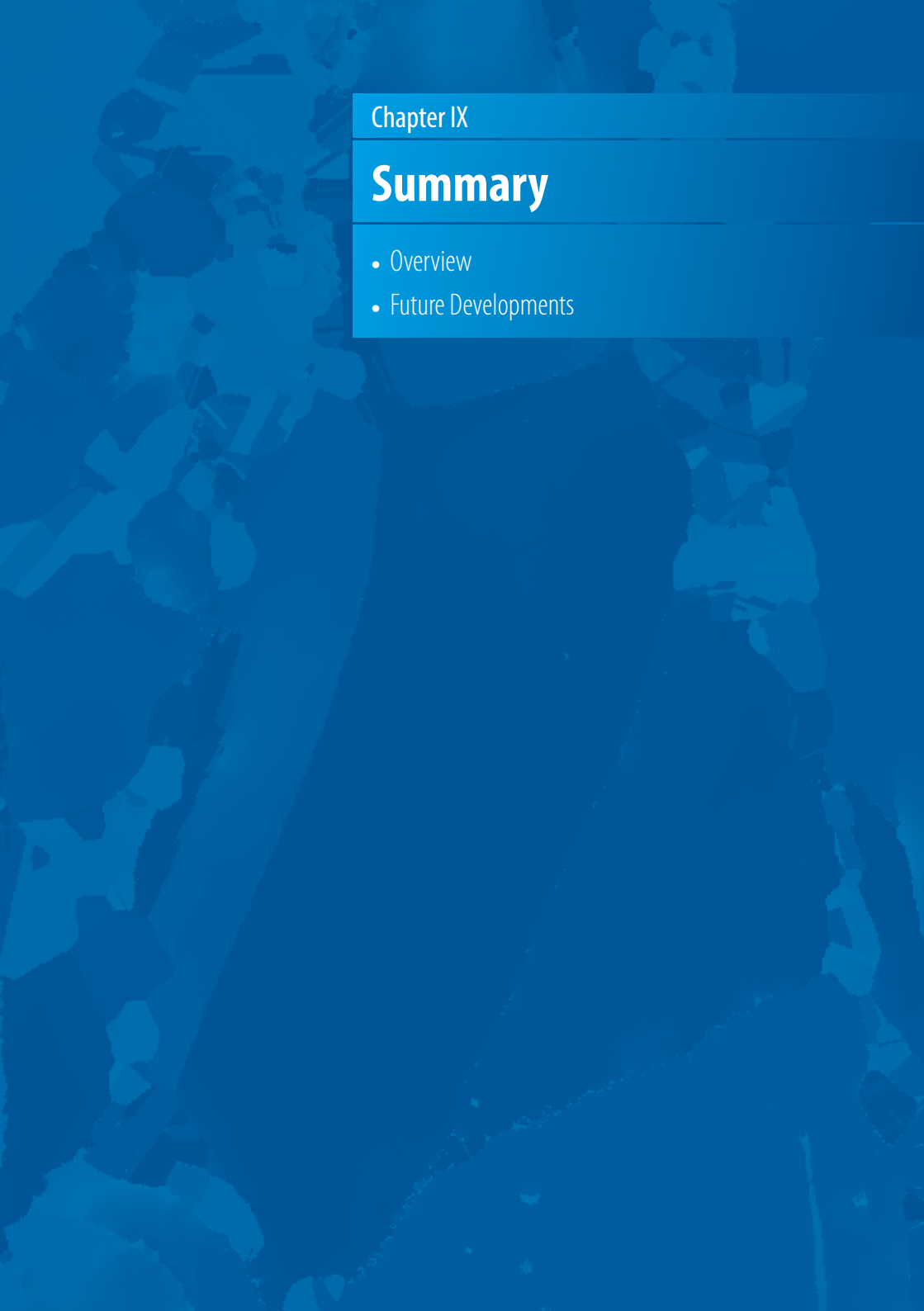
- **Low cycle fatigue characteristics in an H13 tool steel material**

- **Thermal cycles on Zirconium alloys to investigate phase transformation by resistivity measurements**

Various heating and cooling rates were incorporated (10 °C/s to 100 °C/s) for a temperature range of 900 – 1000 °C. Resistivity measurements were acquired as a function of time.

AFRC recommend that it is good practice to carry out tuning of the PID temperature control parameters for different materials at temperatures of interest. This is due to the temperature dependence of the stiffness of the material and so load control is more sensitive to PID values at higher temperatures. Most studies incorporating only thermal cycling require the specimen to be kept at 0 N pre-load. Hence it is crucial to set appropriate PID parameters before heating the specimen at high temperature. Loop tuning functionality of the ETMT system should be used to find the appropriate PID values for the alloy of interest.

Measurement of yield strength, Young's modulus, UTS, elastic and plastic strain in range of materials such as stainless steels, Inconel 625 and HAZ with different cladding are all topics of interest to benefit from the ETMT practicalities in future studies. AFRC also plans to study oxidation behaviour in these materials at different high temperatures. The DIC (digital image correlation) system will be used to study strain measurement in a range of different alloys on the ETMT machine.



## Chapter IX

# Summary

- Overview
- Future Developments

## Overview

The work presented in this report provides quantitative data to give confidence in the ETMT as a small scale measurement system for generating mechanical test data on representative materials. In particular, the evaluation of tensile test data from the ETMT compared with that generated on an Instron test frame demonstrates that the ETMT test is directly comparable with macroscopic tests.

Future work to continue this evaluation could include further detailed assessment of size effects (ETMT testpieces vs standard tensile testpieces), particularly for samples less than  $1 \times 2$  mm cross section and for measurements of ductility and creep properties when influenced by interactions of the sample with the environment (e.g. oxidation). Microstructural influences (i.e. grain size compared with testpiece dimensions) should be more systematically investigated. Furthermore, an assessment of the measurement uncertainty for the load and stress values generated by the ETMT when calibrated over different load ranges would be worthwhile, together with extending the assessment to include more tests at very high temperatures, especially using the DIC method.

## Future Developments

The Instron ETMT is under continual development, taking advantage of developments in measurement technology and to meet customer requirements while the NPL ETMT is being currently used to assess some issues regarding DIC for high temperature strain measurements. The following list summarises possible innovations for the Instron system.

- **Increased resolution of contacting extensometer(s)**

To accommodate smaller test specimens, the measurement transducers will need increased resolution.

- **Easier fitting of contacting extensometer(s)**

If the test specimens are small, it follows the extensometer(s) will be smaller when compared to standard 'macro' size specimens. The extensometers for the ETMT system must be strong enough to be fitted by users, but light enough and small enough to not to damage the specimen. The space available inside ETMT enclosures is small and therefore complex mounting mechanisms (sprung loaded fixtures) are not really possible, so conventional 'cord' mounting seems the most suitable.

- **Real-time strain control using DIC**

The current DIC system provides an analogue voltage output equivalent to the monitored strain. Whilst acceptable during quasi-static testing as a monitor

channel it is not currently feasible to use as a control feedback signal. There are three significant inhibitors to the use of real-time strain control: (a) digital to analogue conversion latency (time lag) in the DIC system; (b) analogue to digital conversion latency (time lag) in the mechanical testing system; (c) DIC Internal image processing time.

The long term aim is to have a purely digital interface between the DIC system and the load frame system. Increasingly efficient use of processing resources through higher speed algorithms concurrent with increases in processor speeds will ultimately increase the DIC feedback bandwidth to the point where DIC can be used as a control channel.

- **New design of loadstring incorporating simplified grip head changing**

Currently the grip heads can be removed and new ones fitted. Sealing is required to ensure the loadstring and grip heads are water tight. PTFE is currently used future designs should incorporate a different sealing arrangement. Grip heads allowing threaded specimens would be beneficial.

- **ETMT with Torsion axis for use with 'low integrity enclosure'**

Over recent times there has been a rise in the requirement for tension/torsion testing with macro size specimens. It seems fair to that suggest ETMT will eventually be required to have torsional capability. The easiest and first version would be for system with low integrity enclosures since these have no sealing around the loadstring and therefore no complicated sealing is necessary.

- **Move ETMT load frame platform from ballscrew actuator to linear electric motor (Variant of Instron's E3000 system)**

Using the linear electric motor based E3000 load frame would increase the operating frequency of the ETMT mechanical loading axis and provide additional standard features. The product roadmap for the E3000 includes its development as a tension/torsion testing frame. Adaptive specimen tuning (based on specimen stiffness) would be beneficial and this is standard on E3000 systems.

- **Increase cooling capability using cryogenic Nitrogen gas**

Controlled specimen heating is relatively simple to achieve given a testpiece with sufficient electrical resistivity and appropriate cross sectional area.

Controlled cooling, however, can be more difficult to achieve over a full range of temperatures. Specimen cooling is governed by thermal conductivity to the water cooled grips and radiation to the immediate environment in the fitted enclosure. The maximum cooling rate decreases with temperature and may be less than that required to meet a temperature profile. The introduction of forced purge gas or cryogenic Nitrogen cooling directed over the specimen could be used to effect controlled cooling when the demand temperature profile. This would not be available for tests being performed in vacuum.

- **Heating of non-metallic / electrically conductive materials**

It is possible to heat non-metallic specimens by using an analogy with induction heating where non-metallic specimens are heated by using a sculptor. In the case of the ETMT a split metallic cylinder could be used to radiate heat energy to a non-metallic specimen. Due to the required delta temperature between the split cylinder and the specimen the maximum achievable specimen temperature would be limited. This approach has been trialled successfully in the NPL ETMT in tests on soldered joints in PCB boards. Temperature measurement is an issue in this type of experimental setup. A thermocouple would probably not be able to be welded on to the specimen as it is usually likely to be an electrical insulator. Pyrometry could be a solution. The temperature of the sculptor (via a thermocouple welded on to the sculptor) could be used to determine the specimen temperature; this could be possible so long as the emissivity of the sculptor and the specimen together with the geometry sculptor and specimen geometry are known.

## Appendix A

# Sample ETMT Procedure

- Generic Notes on Performing Tests on NPL ETMT
- NPL ETMT Test Start Procedure
- Sample NPL ETMT Test Procedures
- Typical Instron ETMT Test Start Procedure

# Generic Notes on Performing Tests on NPL ETMT

The NPL ETMT can be operated in one of two modes; either to conduct so called multi-property tests and also to conduct Thermal Mechanical Fatigue (TMF) tests (with a range of load – temperature waveform options, including sine, triangular, trapezoid, TMF and arbitrary).

Table A1 shows a typical list of options available for performing a test, when in multi-property mode, more combinations are possible.

A variety of tests can be performed using various combinations of these options, including creep, fatigue, tensile and uniaxial.

| Options for performing ETMT tests in multi-property mode |                               |                                                      |
|----------------------------------------------------------|-------------------------------|------------------------------------------------------|
| Temperature                                              | Load                          | Temperature control                                  |
| Constant Temperature                                     | Constant Load                 | Direct manipulation of high and low current values   |
| Cycling Temperature                                      | Cycling Load                  | Software manipulation of high and low current values |
| Ramping Temperature                                      | Ramping Load                  | PID manipulation of high and low current values      |
| Cycling + Ramping Temperature                            | Cycling + Ramping Temperature | PID manipulation of high and low current values      |

Table A1: Options available for performing a test, when in multi-property mode.

## Saving data

Data can be saved at any time during an experiment by clicking on the ‘Save to Disk’ on / off button. Data will be saved to disk, under the file and path name specified previously, and according to the option selected from the drop down box. These options are:

| Options for saving data to disk |                                                                                                                     |
|---------------------------------|---------------------------------------------------------------------------------------------------------------------|
| Option                          | Description                                                                                                         |
| Save every cycle                | Saves all data                                                                                                      |
| Save every <i>n</i> cycles      | Every <i>n</i> cycles, saves all data logged for that cycle                                                         |
| Save every displacement         | Saves all data for cycle when displacement at enf of high cycle changes by more than the set value in $\mu\text{m}$ |

Table A2: Options available for savings data to the disk.

The user can elect to start and stop saving data as often as they wish during an experiment, the new data always being appended to the end (bottom) of the data file. The save options can also be changed at any point during an experiment, giving the user the flexibility, say, to record all the data at the start of an experiment and then at every 20 cycles. For each cycle, data is acquired and saved (if selected) at 10 samples per second.

## Ending an experiment

To end the experiment and stop the program – click the VI power-off button. At the end of the current cycle, the user will be prompted to ‘save the last cycle’, selecting OK, or Cancel will terminate the program. The program then enters a safe shutdown routine which if required resets the load to 0 N and waits until the testpiece has cooled to room temperature. Clicking ‘cancel’ on the pop-up dialog terminates this routine and stops the program.

To remove the testpiece from the ETMT, the procedure is as follows:

- Switch off the DC power supply
- Shut off the argon gas flow to the environmental chamber & remove lid
- Disconnect thermocouple & PDrop leads from sample instrumentation
- Remove testpiece.

## NPL ETMT Test Start Procedure

This Procedure assumes that the cooling water has been running to the grips for at least 30 minutes and that the loading assembly is thermally stable.

1. Run NPL ETMT software
2. When Prompted supply filename operator comments and confirm test limits for load, temperature and displacement
3. Set the position of the grips to define the test gauge length and confirm the displacement transducers are in an appropriate position for the test. A typical starting position is 16 mm grip separation with arbitrary displacement reading of 500  $\mu\text{m}$  (to allow compressive and tensile deformations). To set the gauge length, set a tensile or compressive load of

**NB**

Changes to any of the ‘Cycle Controls’ will only take effect at the end of the current cycle. Changes to the numeric temperature controls are effective immediately, but all on/off switches update at the end of the current cycle.

about 20 N and switch the motor on until the desired position is reached. A 16 mm gauge block inserted between the grips can be a useful aid to this task

4. Check the instrumentation on the testpiece. In particular, ensure that the thermocouple bead is cleanly attached with no secondary wire cross-overs or contact with testpiece
5. Insert testpiece into grips and position such that the thermocouple (if attached) is in the centre of the gauge length. A compass with twin needle points can be used to verify the centricity of the thermocouple
6. **Make sure that power supply is switched off during testpiece insertion to prevent arcing when the circuit is completed**
7. If loading testpiece to loads greater than 1 kN, Loctite glue applied to one surface of the testpiece in the grips can help to prevent slipping in the grips. Ensure the other surface has a good electrical contact with the grips
8. Remove load from testpiece by setting load to 0 N and switching the Motor **On** until zero load is reached. Attach testpiece instrumentation to internal wiring in ETMT. Once attached, switch on power supply. When the power supply completes the initialisation process, it will emit a short pulse of current. If this might damage the testpiece (i.e. if very thin), short the two grips using a metal bar, for example, an Allen key (NB). Set the high and low current cycle controls to gently heat the testpiece and verify thermocouple polarity. If inverted, click on the invert T/C button to correct for thermocouple polarity and restart program
9. Fit lid to ETMT and start purge gas flow. For most materials, Pureshield Argon (99.99 % Argon) will be sufficient to inhibit oxidation. For materials that are particularly oxidation sensitive, Argon-5 % Hydrogen can be used. Leave Argon to flow for 30 minutes to purge the chamber. The circulation fans should be operating to ensure good circulation of the purge gas

**NB**

When completing attaching the thermocouple to the internal wiring, the ETMT software may detect an over temperature condition and trip an alarm ending its execution. Restart program if necessary.

**NB**

Experience has shown that the control software can become unstable and crash if left running following switching on the DC power supply. Restarting the software once the power supply is on fixes this issue.

10. Stop the program by clicking on the VI Power button, which will turn blue. The programme will end at the completion of the current cycle. Restart program and when prompted verify the test information which will be kept from the previous run. If required, select edit or create material specific PID parameters for the test (NB: See p. 104, bottom)
11. The system is now ready for testing to commence.

## Sample NPL ETMT Test Procedures

### Resistivity as a function of Temperature

1. Using calibrated micrometer, measure dimensions of testpiece and determine cross sectional area
2. Determine Room Temperature resistivity of testpiece by alternative method, e.g. 4 point probe technique
3. Instrument testpiece with thermocouple and PDrop wires. Measure separation of PDrop wires, e.g. by use of travelling microscope or microscope with travelling stage
4. Install testpiece into ETMT and start software as per 'NPL ETMT Test Start Procedure' (p. 103)
5. Set load control to 0N and switch 'Motor On' to start controlling to 0 N
6. Switch Data Save to On, selecting save every cycle to ensure all data saved to disk
7. Set Current controls to 1 A and allow system to stabilise, recording RT resistivity data
8. Ramp temperature at proscribed rate to target temperature, then return at same rate to RT
  - a) Set Cycle Time High & Low to 0.5 seconds
  - b) Set Cycle increment to increment every cycle
  - c) Set High and low Temperature increment to required setting (e.g. 5 °C)
  - d) Switch on Temperature control and activate PID control. If necessary, adjust PID control values
  - e) Switch on increment button to start ramping temperature
  - f) At target temperature switch increment button off. Negate the temperature increment setting (e.g. to -5 °C) and switch increment button on
  - g) When testpiece temperature approached RT, and the system can no

- longer control temperature by reducing current, switch Temperature control off, reverting to steady state current of 1 A
- h) Wait for testpiece to return to RT, then repeat steps a-g as necessary
- 9. End test by setting current to 0 A, switching motor off and VI Power off
- 10. Calculate instantaneous resistivity from resistance, testpiece dimensions and PDrop wire separation. Adjust the measured PDrop separation to the effective separation required in order to normalise the ETMT data to the independently measured RT resistivity.

## High Temperature Tensile Test

1. Using a calibrated micrometer measure dimensions of testpiece and determine cross sectional area
2. Instrument testpiece and measure separation of PDrop wires e.g. by use of travelling microscope or microscope with travelling stage to determine effective gauge length
3. Install testpiece into ETMT and start software as per 'NPL ETMT Test Start Procedure' (p. 103)
4. Set load to 0 N and switch 'Motor On' to start controlling to 0 N
5. Switch Data Save to On, selecting save every cycle to ensure all data saved to disk
6. Set Current controls to 1 A and allow system to stabilise, recording RT resistivity data
7. Ramp temperature at proscribed rate to target temperature
  - a) Set Cycle Time High & Low to 0.5 seconds
  - b) Set Cycle increment to increment every cycle
  - c) Set High and low Temperature increment to required setting (e.g. 5 °C)
  - d) Switch on Temperature control and activate PID control. If necessary, adjust PID control values
  - e) Switch on increment button to start ramping temperature
  - f) At target temperature switch increment button off. Displayed temperature should control to within +/- 1-2 °C
8. Note initial zero strain resistance. If intending to test to target strain value, determine resistance that equates to target strain
9. Ramp load or displacement at proscribed rate to target stress / strain, or specimen failure
  - a) Set Cycle Time High & Low to 0.5 seconds

- b) Set Cycle increment to increment every cycle
  - c) Set High & Low load (position) increment to required setting e.g. 1  $\mu\text{m}$
  - d) Ensure temperature increment controls set to 0
  - e) Set desired testing mode – i.e. Load or Displacement control
  - f) Check cycling controls are at start point (I. 0 N or current controlled position) and enable cycling
  - g) Switch on increment button to start ramping load / position
  - h) If not testing to failure, switch off increment button once at target resistance (strain) or Stress (NB, bottom left)
10. End test by unloading testpiece and ramping temperature to RT
- a) Set Load to 0 N and switch cycling off and set control mode to load control to remove load from testpiece (NB, bottom right)
  - b) Ramp temperature to RT by following step 7, but with a negative temperature increment. Ensure that the load cycling controls are set to 0
  - c) As testpiece temperature approaches RT and the system can no longer control temperature by reducing the heating current, switch temperature control off. The system will revert to the 1A current set in step 6
11. Once system is at RT and 0 N, shut system down
- a) Switch off Data Save
  - b) Switch off VI Power
  - c) Switch off purge gas.

### NB

Depending on what the intention of the test is, there may be one or more options at the end of the test, e.g. quenching for microstructural analysis, annealing to perform another test on the same testpiece or safe shutdown of the experiment.

### NB

Care needs to be taken when switching cycling off and changing control modes to ensure that the testpiece is not inadvertently loaded from mis-matching control values.

# Thermal Fatigue Testing

- 1. Using a calibrated micrometer measure dimensions of testpiece and determine cross sectional area
- 2. Instrument testpiece and measure separation of PDrop wires e.g. by use of travelling microscope or microscope with travelling stage to determine effective gauge length
- 3. Install testpiece into ETMT and start software as per 'NPL ETMT Test Start Procedure' (p. 103), selecting TMF as test type when prompted
- 4. When prompted, define thermal and mechanical cycle waveforms, e.g. sinusoid, triangular, trapezoid or custom
- 5. Set mechanical control mode to Load or displacement
- 6. Enable Data Saving
- 7. To Run a test in Load control:
  - a) Ensure system in Load Control
  - b) Set Load to 0 N and switch 'Motor On' to control to 0 N
  - c) Enable testpiece heating by clicking 'Heating On'. Testpiece will immediately start to heat up and control to defined temperature profile
  - d) Once temperature profile has been established, apply the loading cycle by clicking 'Loading On'. Applied load will start to follow defined loading profile
- 8. To Run a test in displacement control:
  - a) Ensure system is in Displacement Control. At end of current cycle, the buttons 'Get Comp Data' and 'Apply Compensation' will become enabled
  - b) Stet motor control button to on. The system will start to control to 0 N
  - c) Turn on heating to the testpiece by clicking the 'Heating On' button. Testpiece will start to heat up and control to defined temperature profile
  - d) To compensate for thermal expansion effects, compensation data needs to be applied to the displacement control profile. Once thermal cycle is established, select compensation method from list below:

| Method of Comparison | Effect                                                                                           |
|----------------------|--------------------------------------------------------------------------------------------------|
| None                 | No Compensation applied                                                                          |
| Average              | Average displacement over No selected cycled applied                                             |
| Full Array           | Complete set of thermal expansion displacement data averaged over selected No of cycles applied. |

- e) Click 'Get Compensation Data' to commence collecting the thermal expansion compensation data. The button will stay highlighted until all data has been collected. The compensation – temperature graph will be updated at the end of each cycle
  - f) Click on 'Apply Compensation' to apply the temperature – displacement compensation data to the displacement defined control profile. The button stays highlighted until action completed at the end of the current cycle
  - g) Enable displacement control by clicking on the 'Loading On' button. System will immediately start to control to compensated displacement profile
9. To reduce size of data file, change data save option to 'Save every *n* cycles'. Set *n* to an appropriate number
  10. The system will continue the test until such time as the testpiece fails.

## Typical Instron ETMT Test Start Procedure

1. Prepare specimen and instrument as necessary with thermocouple(s) and potential drop wires (2 off)
2. Ensure load axis of Instron 8800 control is enabled
3. 8800 is in Position control with specimen protect disabled
4. Set grip distance (typically 16 or 40 mm) using grip separation block and calibrate position (encoder) channel
5. Install specimen within grips and gradually tighten the 4 off M5 screws per grip block
6. Place the system in to load control and enter a setpoint of 0 N then 'loop tune (PID)'
7. Place the system to position control with specimen protect disabled (NB)
8. Fit the front door to the vessel and ensure the rear plate is fitted and screwed on tightly
9. Close gas purge valve
10. Open vacuum isolation valve
11. Start vacuum pump
12. Allow system to pump down to pressure in the region of  $5 \times 10^{-2}$  mbar
13. Close vacuum isolation valve
14. Open inert gas valve

15. Gas will fill the vessel and then vent through the primary relief valve (time dependant on gas flow rate)
16. Once the system is purged the specimen can be heated (remember to mode change to load control if no load is to be applied during heating!). If the user wishes to have more than one vacuum/back-fill cycle the operator can repeat appropriate steps described above
17. The test environment setup (air, vacuum or gas purge) must be completed prior to heating the specimen.

If the low integrity enclosure is fitted, items 1 through 6 and 8 from the above sequence are applicable. Once the gas supply is ready the 'inert gas valve' is opened and the 'high integrity vessel' is back-filled and gas is then vented from the enclosure.

In all modes with either enclosure, the front door must be fitted to the front of the enclosure before the system can be enabled and allow electrical current to flow through the specimen. The requirement of fitting the front door and having split electrical covers around the loadstring ensures the operator does not come in contact with the high current components.

**NB**

Do not use specimen protect if vacuum is to be applied, since it will cause loading on the specimen due to vacuum being applied to load string.

**NB**

If test is to be performed under vacuum, then the load cell must be balanced once the system is at maximum vacuum to remove the effects of the vacuum on the loadstring.

# Instron 8800 ETMT thermal loop tuning

## Current Control

1. Ensure vessel door is closed
2. Load frame in high force mode
3. Load frame in Load control
4. Monitor temperature axis 'servo drive' output (must be around 0 %)
5. Ensure water cooling is running
6. Enable heating by pressing the 'Enable' switch on the front of the control system
7. Open PID control panel within console software
8. Set 'P-term' to -20 dB
9. Select 'haver-triangle' in loop tuning page and enter 40 A as the amplitude and the frequency as 0.1 Hz
10. Start waveform and monitor command and current feedback. Ideally the feedback will mimic the command signal, but with a phase lag
11. Initially the feedback will be very low, but will increase as the 'P-term' is increased.

## Temperature Control

1. Ensure vessel door is closed
2. Load frame in high force mode
3. Load frame in Load control

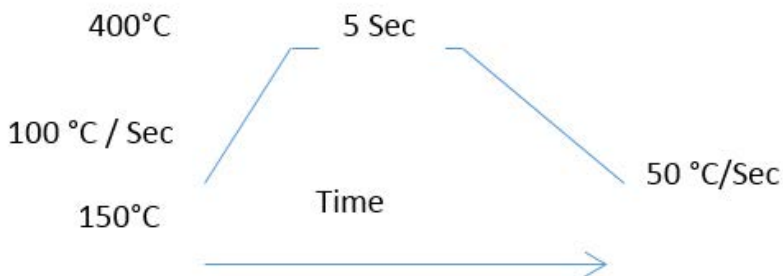


**Figure A1:** Schematic representation of a typical trapezoid cycle.

4. Monitor temperature axis 'servo drive' output (must be around 0 %)
5. Ensure water cooling is running
6. Enable heating by pressing the 'Enable' switch on the front of the control system
7. Open PID control panel within Console Software
8. Set 'P-term' to -20 dB
9. Use 'ramp generator' to create a trapezoid. A typical cycle is shown in Figure 69
10. Start waveform and monitor command and current feedback. Ideally the feedback will mimic the command signal, but with a phase lag (NB)
11. Initially the feedback will be very low, but will increase as the 'P-term' is increased. Integrator may be necessary to achieve adequate temperature feedback.

**NB**

The required cooling rate may not be achieved due to insufficient cooling (heat loss). The higher the operating temperature the higher the maximum cooling rate.

Appendix B

# Instron ETMT Technical Specifications

- Technical specifications

# Technical specifications

| Test Frame and Loadstring            |                                                                                                                                                                              |
|--------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Maximum Load                         | 3 kN                                                                                                                                                                         |
| Load Accuracy                        | Accurate to $\pm 0.005$ % of load cell capacity or 0.5 % of indicated load, whichever is the greater                                                                         |
| Maximum Speed                        | 1000 mm min <sup>-1</sup> (dependent on configuration)                                                                                                                       |
| Minimum Speed                        | 0.01 mm min <sup>-1</sup> (dependent on configuration)                                                                                                                       |
| Speed Accuracy                       | $\pm 0.1$ % of setting (zero or constant load)                                                                                                                               |
| Maximum Cyclic Frequency             | 3 Hz (specimen dependent)                                                                                                                                                    |
| Maximum Actuator movement            | 50 mm                                                                                                                                                                        |
| Total Strain Accuracy (LVDT)         | $\pm 2.5$ micron over $\pm 2.5$ mm LVDT stroke (0.1 % linearity), & maximum cyclic operation 1 Hz                                                                            |
| Maximum Heating Rate                 | 200 °C s <sup>-1</sup> as a ramp (specimen dependent)                                                                                                                        |
| Total Strain Accuracy (Extensometer) | 0.5 % strain (5 mm GL) over range + 2.0 mm, -1.0 mm, maximum cyclic operation of 1 Hz and distance from extensometer measuring tips to extreme end of extensometer of 100 mm |
| Grip Head Material                   | Brass or Stainless steel                                                                                                                                                     |
| Loadstring material                  | Stainless steel                                                                                                                                                              |
| Specimen Gripping                    | Manual screw clamp                                                                                                                                                           |
| Specimen Geometry                    | Round or Flat                                                                                                                                                                |
| Loadstring- Water Cooling            | Up to Grip heads                                                                                                                                                             |
| Test Frame                           | H1690 mm × W468 mm × D530 mm (In vertical mode)                                                                                                                              |

**Table B1:** Instron ETMT Test Frame and Loadstring specifications.

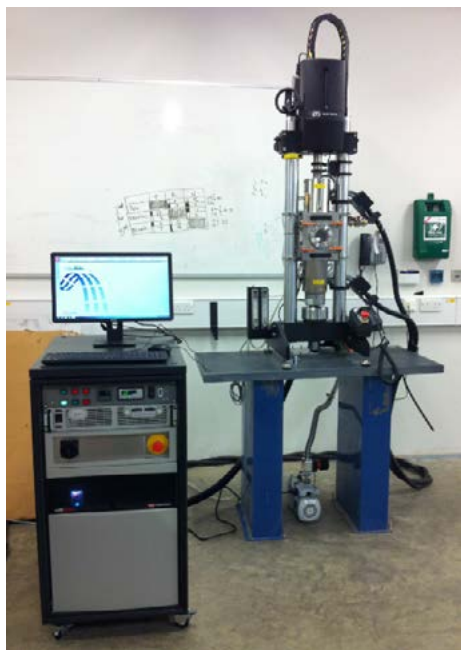


**Figure B1.** Instron ETMT system.

## Low Integrity Inert Atmosphere Capsule

|                     |                                                                |
|---------------------|----------------------------------------------------------------|
| Material            | Poly carbonate enclosure                                       |
| Vacuum              | Not possible                                                   |
| Maximum pressure    | 0.3 PSI                                                        |
| Stroke accommodated | ± 5 mm                                                         |
| Access              | Front door only, no rear access                                |
| Dimensions          | H200 mm × W150 mm × D183 mm (Internal)                         |
| Door interlock      | Front door (inductive safety switch)                           |
| Operation           | Inert gas purging                                              |
| Gas inlet           | Ø 6 mm plastic tubing                                          |
| Gas outlet          | Gas escapes around loadstring (top and bottom)                 |
| Cable access        | Simple cable gland and extensometer through slot in front door |

**Table B2:** Low Integrity Inert Atmosphere Capsule specifications.

## High Integrity Inert Atmosphere Capsule

|                        |                                                                         |
|------------------------|-------------------------------------------------------------------------|
| Ultimate vacuum        | 10 <sup>-3</sup> mbar (Dependant of specification of vacuum pump)       |
| Dimensions             | H200 mm × W150 mm × D183 mm (Internal)                                  |
| Material               | Stainless 316                                                           |
| Capsule Mass           | 3 kg                                                                    |
| Loadstring Interface   | Stainless steel bellows top and bottom                                  |
| Vacuum level obtained  | 0.05 mbar Using standard vacuum pump 1.8 m <sup>3</sup> h <sup>-1</sup> |
| Vacuum sensor          | Edwards active gauge (thermal conductivity sensor)                      |
| Vacuum pump location   | Below load frame on the floor                                           |
| Vacuum pump line       | 1.5 m long, NW25 Flexible Stainless steel hose                          |
| Maximum pressure       | 0.3 bar G                                                               |
| Inert gas inlet        | 'Tee' in to vacuum pumping port                                         |
| Inert gas outlet       | Pressure relief valve                                                   |
| Stroke accommodated    | ± 5 mm                                                                  |
| Vacuum port            | NW25                                                                    |
| Multi-pin feed-through | 18 pin, sealed (T/Cs, LVDT's, Extensometer and Specimen Voltage)        |
| Optical Window         | Mounted in front door                                                   |
| Clear diameter         | 68 mm                                                                   |
| Material               | Fused Silica                                                            |
| Surface Quality        | 40-20 scratch and dig                                                   |
| Thickness              | 6.4 mm                                                                  |
| Rear Access Plate      | Blank panel                                                             |
| Door interlock         | Front door only (inductive safety switch)                               |

**Table B3:** High Integrity Inert Atmosphere Capsule specifications.

| Temperature Control System                           |                                                                               |
|------------------------------------------------------|-------------------------------------------------------------------------------|
| DC Power Supply                                      |                                                                               |
| Nominal Power Rating                                 | 3.2 kW                                                                        |
| Current controller                                   | Instron 8800 controller, axis 2, channel 1                                    |
| DC Power Supply (mounted inside 20U control cabinet) | H90 mm × W423 mm × D443 mm                                                    |
| 400A, DC Power Supply                                | 13 kg                                                                         |
| Maximum Output Current                               | 400 A                                                                         |
| Maximum Output Voltage                               | 8 V                                                                           |
| Ripple (RMS)                                         | 12 mV                                                                         |
| Noise (P-P)                                          | 60 mV                                                                         |
| Regulation                                           | 6.2 mV/450 mA                                                                 |
| Temperature Control                                  |                                                                               |
| Maximum Temperature                                  | 1500 °C (specimen dependent)                                                  |
| Temperature Measurement                              | Typically type 'R' or 'S' thermocouple(s)                                     |
| Temperature controller                               | Instron 8800 controller, axis 2, channel 2                                    |
| Specimen Voltage monitoring                          |                                                                               |
| Voltage monitoring                                   | Instron 8800 controller, axis 2, channel 3 (monitoring only, NOT controlling) |

**Table B4:** *Temperature Control System specifications.*

| Dimensions and Weights                        |                                                              |
|-----------------------------------------------|--------------------------------------------------------------|
| Dimensions (Approximate)                      |                                                              |
| 19", 20U Control Cabinet (mounted on castors) | H 1000 mm × W500 mm × D800 mm                                |
| 8800MT Controller                             | H500 mm × W200 mm × D450 mm                                  |
| Weights                                       |                                                              |
| Test Frame                                    | 180 kg (including high integrity capsule, load string, etc.) |
| 8800 Controller                               | 25 kg                                                        |
| 19", 800 mm Deep Cabinet                      | 100 kg                                                       |

**Table B5:** *Approximate dimensions and weights specifications.*

| Environmental         |                               |
|-----------------------|-------------------------------|
| Operating Temperature | 10 °C to 38 °C                |
| Storage Temperature   | –40 °C to 65 °C               |
| Humidity              | 10 % to 90 % (non condensing) |

**Table B6:** *Environmental specifications.*

| Service Requirements |                                                                                                                                                       |
|----------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------|
| Power                |                                                                                                                                                       |
| Voltage              | 410 to 420 VAC 3 phase and neutral,                                                                                                                   |
| Current              | 16 A                                                                                                                                                  |
| Frequency            | 50 Hz                                                                                                                                                 |
| Cooling Water        | 1 to 4 l min <sup>-1</sup> ; inlet temp 10 to 25 °C; filtered to 50 µm; corrosion inhibitor preferred. Alarm trips at about 0.7 l min <sup>-1</sup> . |
| Argon Purge Gas      | 0.6 to 4 l min <sup>-1</sup> ; pressure regulated to 0.1 bar.                                                                                         |

**Table B7:** *Service Requirements.*

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# **Good Practice Guide for Miniature ETMT Tests**

Good Practice Guide No. 137

## **What is it about?**

This guide discusses various aspects of testing miniaturised testpieces of representative engineering alloys, especially at high temperatures with the Electro-Thermal Mechanical Test system (ETMT) developed by NPL and Instron UK. Measurement techniques are reviewed and comments made on uncertainties of measurement and best measurement practice. Typical comparative results with those from macroscopic test systems are presented, as are example case studies illustrating research applications.

## **Who is it for?**

This guide is intended for researchers, scientists and engineers who may be new to the field of miniaturised mechanical testing with the ETMT and are starting, or planning, to use the ETMT in the study of high-temperature alloys.

## **What is its purpose?**

The purpose of the guide is to provide technical information to users of the ETMT system, and to illustrate examples of applications of this measurement system for the analysis of mechanical properties of alloys, in order to support the use of this novel miniaturised technique to generate high quality mechanical and microstructural data.

## **What is the prerequisite knowledge?**

The guide assumes that the reader has some familiarity and experience in high temperature mechanical testing; has a good technical knowledge about temperature, strain and load measurement techniques; an understanding of mechanical properties of materials and experience performing mechanical tests using standard Instron application software.