

NPL REPORT
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**Uniaxial Tests Using A
Miniaturised Test Method**

p11 and p12 alloy steels

B ROEBUCK and M BROOKS

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Uniaxial Tests Using A Miniaturised Test Method *p11 and p12 alloy steels*

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ABSTRACT

The estimation of remnant life of materials in plant and structures is often assessed using destructive tests on specimens taken from the actual component. New and improved miniaturised testing procedures are needed in order to reduce the required sample size removed from the plant. This report details the work done at NPL using a new miniature test system, the ETMT [1-3]. Uniaxial tests have been conducted on two alloy steels, p11 and p12, with the objective to evaluate the utility of the system for conducting miniature uniaxial tests, in both monotonic uniaxial tensile tests and high stress short term constant load tests. The tests on steel p11 were conducted at 580 °C while the tests on steel p12 were conducted in the range 580-640 °C.

One of the steels, p11, was provided by University of Wales, Swansea in both the as-received and thermally exposed conditions. The thermal exposure comprised 300 h at 580 °C at 135 MPa. The time to failure under these conditions was 536 h. The other steel, p12, was provided by E.ON in two plant exposed conditions, one for 200 kh and one for 250 kh.

The report also contains three Appendices that give a test chronology for current tests (Appendix A); that provide additional background information on conducting uniaxial tensile tests in the ETMT (Appendix B) and test and calibration procedures (Appendix C) developed for the detailed use of the ETMT.

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1 INTRODUCTION

Materials in plant and structures, particularly those operated by power generation companies, are expected to endure in service far longer than the period available for testing or evaluating new systems. Although design procedures against creep and fatigue failure are well established, they contain sufficient uncertainty to make continual monitoring of plant necessary, and estimation of remanent life becomes essential. This is often assessed using destructive tests on specimens taken from the actual component. Therefore new and improved miniaturised testing procedures would be very welcome. Measurement research on this topic is being conducted in partnership with the IRC in Computer Aided Engineering at the University of Wales, Swansea. This report details the work done at NPL using a new miniature test system, the ETMT [1-3], aimed at obtaining uniaxial data and providing miniature test procedures for this purpose. In parallel, research is also being conducted on the Small Punch disc test [4] supported by modelling studies at the University of Wales, Swansea [5].

Uniaxial tests have been conducted on two alloy steels, p11 and p12, using the NPL miniature test system – the ETMT. One of the steels, p11, was provided by University of Wales, Swansea in both the as-received and thermally exposed conditions. The thermal exposure comprised 300 h at 580 °C at 135 MPa. The time to failure under these conditions was 536 h. The other steel, p12, was provided by E.ON in two plant exposed conditions, one for 200 kh and one for 250 kh.

The objective of the ETMT tests was to evaluate the utility of the system for conducting miniature uniaxial tests, in both monotonic uniaxial tensile tests and high stress short term constant load tests. The tests on steel p11 were conducted at 580 °C while the tests on steel p12 were conducted in the range 580-640 °C.

The report also contains three Appendices that give a test chronology for current tests (Appendix A); that provide additional background information on conducting uniaxial tensile tests in the ETMT (Appendix B) and test and calibration procedures (Appendix C) developed for the detailed use of the ETMT.

2 MATERIALS AND TESTS

Steel p11 is a nominal	1 Cr ½ Mo	low alloy steel.
Steel p12 is a nominal	½ Cr ½ Mo ¼ V	low alloy steel.

The p11 steel was provided by the University of Wales, Swansea in the form of round bar (nominally creep test pieces) about 15 mm in diameter. ETMT samples were edm sliced from these blanks and then lightly diamond ground to a final nominal size of 40 × 2 × 1 mm. Steels p11 ar and p11 exp refer to as received (ar) and exposed (exp) condition respectively.

The p12 steel was provided by E.ON as plant exposed material. The ETMT samples were prepared in the same way as those from the p11 steel. The sampling orientation was in a circumferential direction from pipe section. Steels p12 200 and p12 250 refer to the different plant exposure times.

It is important to characterise the resistivity of the testpieces because resistance values (see Appendix A) are used to measure strain during mechanical tests. The room temperature resistivity was measured using a conventional 4 probe dc current method. A current of about 2A was passed through the sample, i.e. at a level insufficient to heat it, and the potential drop along a length of about 6.5 mm was measured with forward and reversed current flow. An average value of this potential drop (pd) was used to calculate the resistivity. The temperature dependence of resistivity was also determined in the ETMT system – this time using controlled dc current to heat the sample.

Some background details of the mechanical test procedures applied during the investigation in the ETMT are given in Appendix A and a test chronology (Appendix B) is listed for each steel at the end of the report. The uniaxial tensile tests were conducted under conditions of constant loading rate. Both the uniaxial and constant load tests were undertaken in flowing argon.

3 RESULTS

3.1 RESISTIVITY MEASUREMENTS

The Room Temperature resistivity values are shown in Table 1 and illustrated with ranked plots in Fig 1. The temperature dependences of the resistivities are shown in Figs 2-5. The measurements showed good repeatability on different testpieces and the data can be fitted using a second order polynomial with coefficients, for example, as specified on one of the graphs (e.g. Fig 2). Thermal exposure to steel p11 has resulted in small but significant differences in resistivity at room temperature (Fig 1), whereas at elevated temperatures there is little change. For steels p12 different RT resistivities were noted, but it is not known whether this is solely due to differences in tool exposure or differences in composition since the original unexposed material has not been investigated.

3.2 UNIAXIAL TENSILE TESTS

The results of tensile tests at different temperatures for the p12 200 and 250 samples are shown in Fig 6 at a nominal constant stressing rate of 5 MPa/s. It can be seen that at this deformation rate (5 MPa/s) temperature has a significant effect on mechanical properties, with a 50% decrease in strength observed as the temperature is raised from 580 to 640 °C. The strains depicted in Fig 6 are plastic strains derived from measurements of change in resistance (see Appendix B). A Ramberg-Osgood power law was used to fit the data and representative data are shown in Figs 7 and 8. The Ramberg-Osgood power law relates measured strain, ε , to applied stress (σ) as follows

$$\varepsilon = \frac{\sigma}{E} + \left(\frac{\sigma}{\sigma_0} \right)^n \quad (1)$$

where E is the Young's Modulus and σ_0 and n are constants that reflect the shape of the curves.

The stress/strain curves at different test temperatures (580-640 °C) for the plant exposed p12 steel are compared in Fig 8. The data show little difference at the nominal stressing rate of 5 MPa/s. Loading rate significantly affects the shape of the tensile curves and this can be seen in Fig 9 where a factor of 100 difference in stressing rate (from 5 MPa/s to 0.05 MPa/s) results in a difference in flow stress of about 50-60 MPa at a strain of 5%. At the lower loading rate of 0.1 N/s there is a small difference in the tensile curves of p12 200 and 250 with p12 250 being slightly stronger (Fig 10).

The stress/strain curves for the p11 as-received and thermally exposed materials are compared in Fig 11. There is a clear effect of deformation rate for both materials, i.e. the higher the loading rate the stronger the material. The as received material (p11 ar) is stronger than the thermally exposed steel (p11 exp), particularly at low deformation rates (Fig 12).

For comparison, tensile curves for p11 and p12 are shown in Fig 13. Clearly steel p11 is significantly stronger than steel p12. The plots do not give any indication of ductility values as the p11 test was interrupted well before failure.

3.3 UNIAXIAL CONSTANT LOAD TESTS

The results of the constant load tests (at stresses of 95, 110 and 130 MPa) at 620 °C on the p12 steel are shown in Fig 14. The results are expressed either as strain values calculated from resistance changes or as direct measurements of grip displacement. The curve shapes are similar and it can be seen that there are significant differences between the different levels of plant exposure. The p12 200 sample is less resistant to load than p12 250. The results in Fig 14 are converted to the more typical strain rate vs time plots in Fig 15 and strain rate vs stress plots in Fig 16. Figure 16 also includes additional data from tests at 80 MPa. The strain vs time and strain rate vs time plots for this lower stress experiment are shown in Fig 17. It can be seen that one of the tests failed prematurely, possibly due to oxidation as some difficulties are being experienced with the ETMT test system in recent experiments in maintaining inert gas atmospheres in experiments.

A similar set of results for constant load tests on the p11 ar and p11 exp samples are shown in Figs 18 and 19. The results for steel p11 shown in Fig 19 are not as consistent a set as those shown in Fig 16 for steels p12. Consequently future tests are planned at lower applied stress levels to extend the data set to lower values of strain rate.

All the data from the ETMT tests have been combined in stress vs time to failure (Fig 20) and minimum strain rate vs time to failure (Fig 21) plots. Again, good trends are indicated but they also need extending to lower values of strain rate. The data point from the prematurely failed testpiece, p12 200 (80 MPa) has also been included. The correlation between minimum creep rate and time to failure (Fig 20) is even more consistent, clearly showing the effects of premature failures in two of the p12 steel samples. But again, further tests at lower stresses would be helpful. As would comparison with data obtained in macroscopic tests. These latter activities are both planned for the next stage of the work.

Table 1
Room Temperature Resistivity

Testpiece Code	Width mm	Thickness mm	Current A	pd separation mm	pd mean value mV	Resistivity, ρ nWm
p11 ar e	1.99	1.00	1.86	6.5	1.918	317
p11 ar f	2.00	1.00	1.86	6.5	1.916	317
p11 ar g	2.00	1.00	1.86	6.5	1.921	319
p11 ar h	2.01	1.00	1.86	6.5	1.935	324
p11 exp a	1.97	1.00	1.84	6.5	1.905	314
p11 exp b	1.97	1.00	1.84	6.5	1.903	313
p11 exp c	1.99	1.00	1.84	6.5	1.886	314
p11 exp d	1.97	0.99	1.84	6.5	1.911	312
p11 exp e	1.97	1.00	1.84	6.5	1.873	309
p11 exp f	1.98	0.99	1.84	6.5	1.904	312
p12 200 a	2.00	1.01	1.86	6.5	1.133	188
p12 200 b	2.00	1.00	1.86	6.5	1.131	187
p12 200 c	1.99	0.99	1.86	6.5	1.143	187
p12 200 d	2.00	1.00	1.86	6.5	1.120	185
p12 200 e	2.00	1.00	1.86	6.5	1.121	186
p12 200 f	2.00	1.00	1.94	6.5	1.200	190
p12 200 g	1.99	1.00	1.94	6.5	1.196	188
p12 200 h	1.96	1.00	1.94	6.5	1.183	184
p12 200 l	1.99	1.00	1.94	6.5	1.187	187
p12 200 j	1.99	1.00	1.94	6.5	1.178	186
p12 250 a	1.99	1.01	1.86	6.5	1.110	185
p12 250 b	1.99	1.01	1.86	6.5	1.110	184
p12 250 c	1.99	1.01	1.86	6.5	1.112	184
p12 250 d	1.99	1.00	1.86	6.5	1.114	184
p12 250 e	1.99	1.01	1.86	6.5	1.110	184
p12 250 f	1.99	1.01	1.94	6.5	1.171	186
p12 250 g	1.99	1.00	1.94	6.5	1.170	184
p12 250 h	1.99	1.00	1.94	6.5	1.164	184
p12 250 l	1.99	1.01	1.94	6.5	1.189	188
p12 250 j	1.99	1.01	1.94	6.5	1.173	187

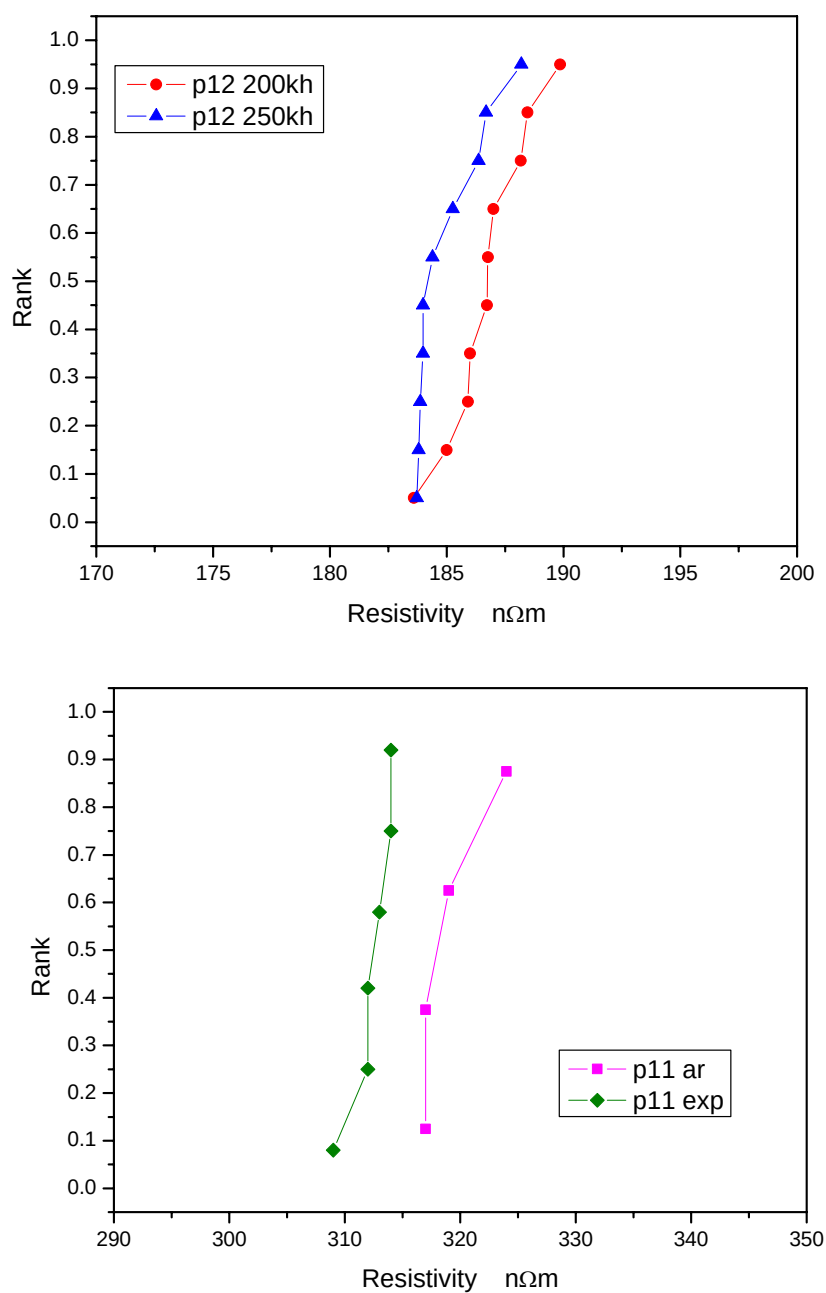


Fig 1 Ranked plots of RT resistivity.

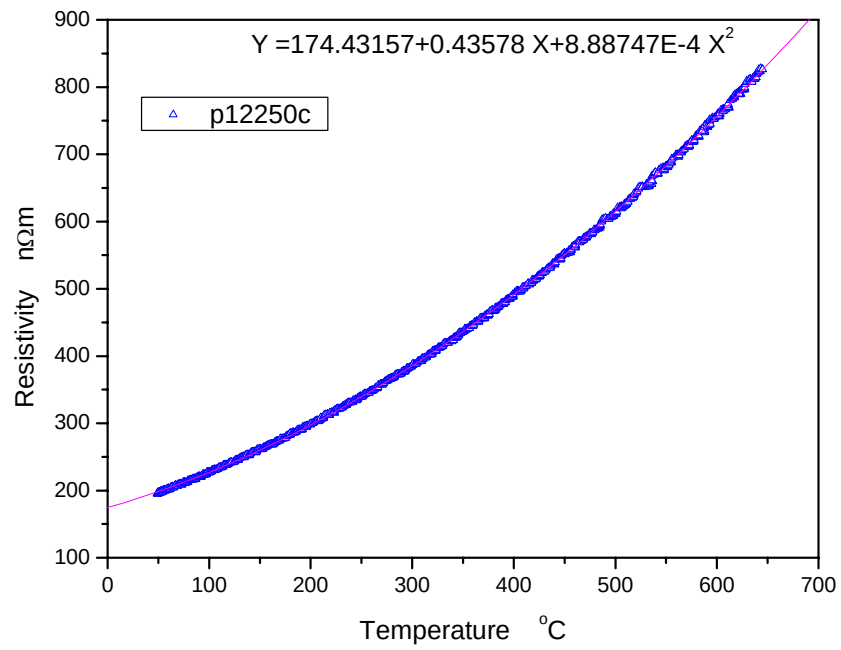


Fig 2 Temperature dependence of resistivity of steel p12 200.

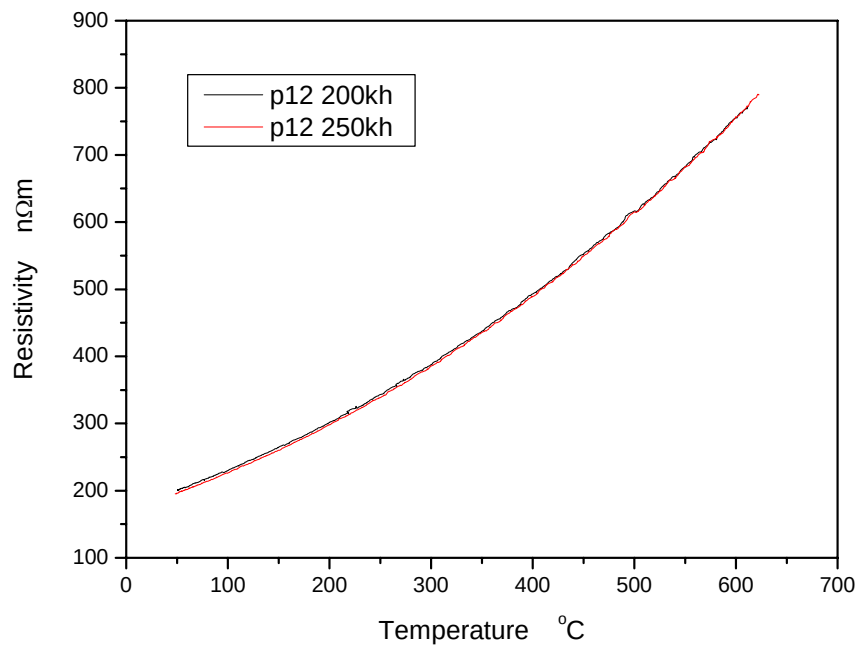


Fig 3 Temperature dependence of resistivity of steel p12 250 and p12 200.

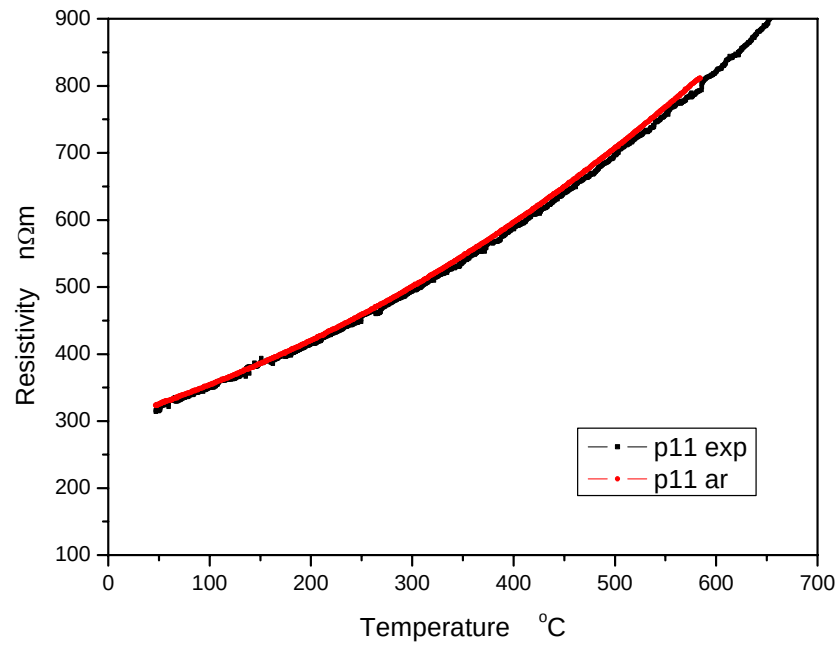


Fig 4 Temperature dependence of resistivity of steel p11 as-received and steel p11 thermally exposed.

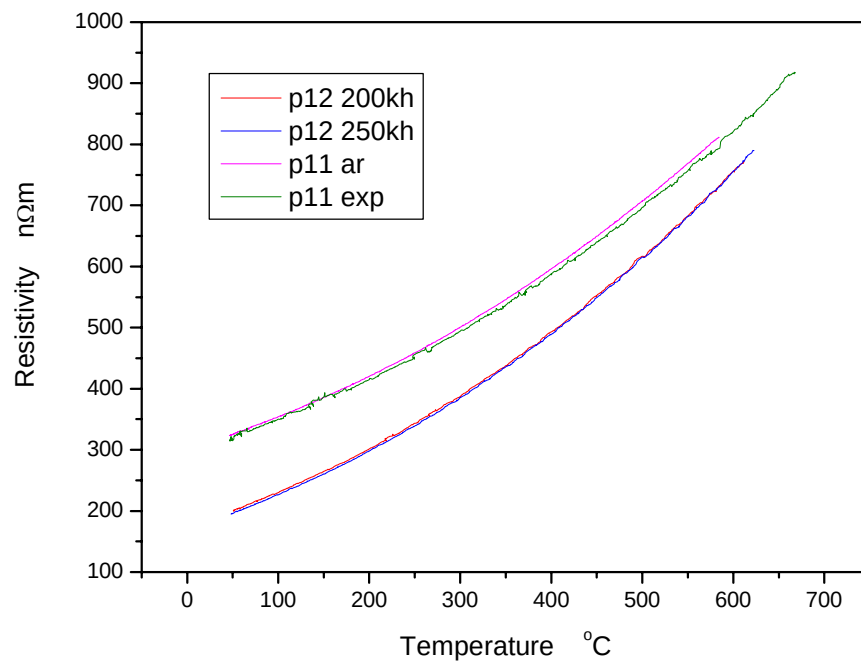


Fig 5 Temperature dependence of resistivity of steel p11 compared with steel p12.

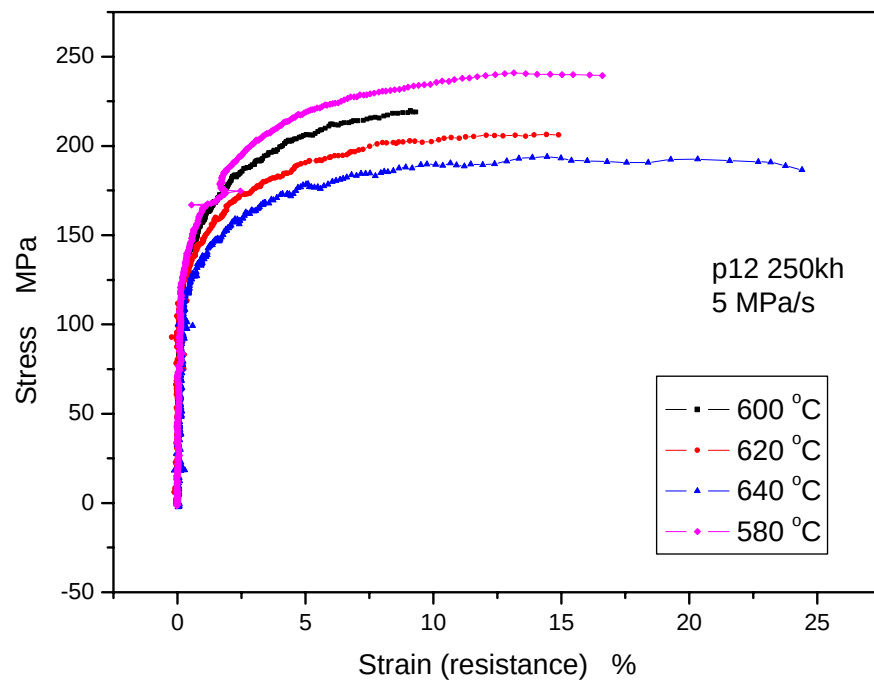
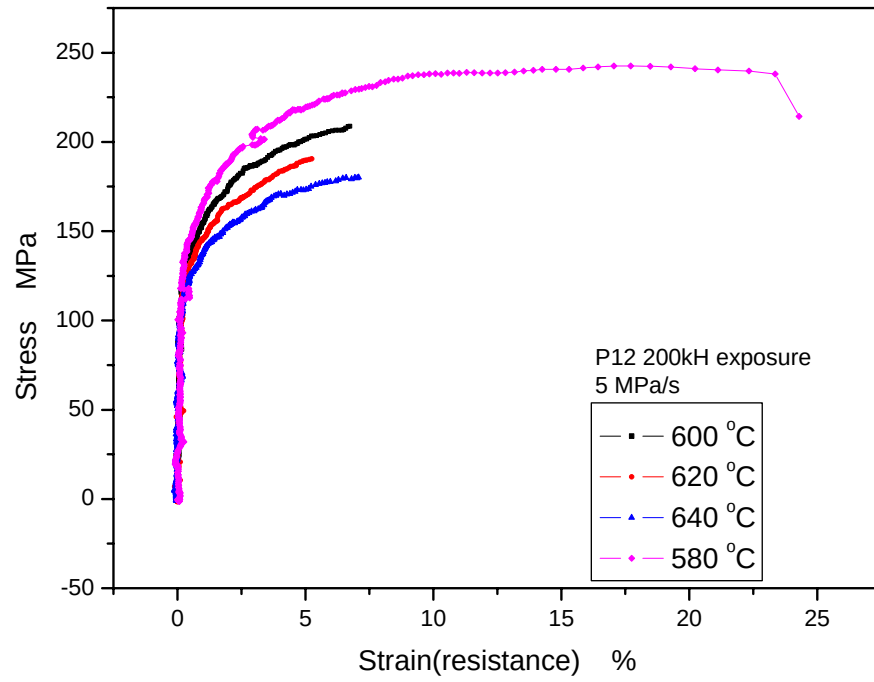


Fig 6 Tensile test results for p12 200 and 250.

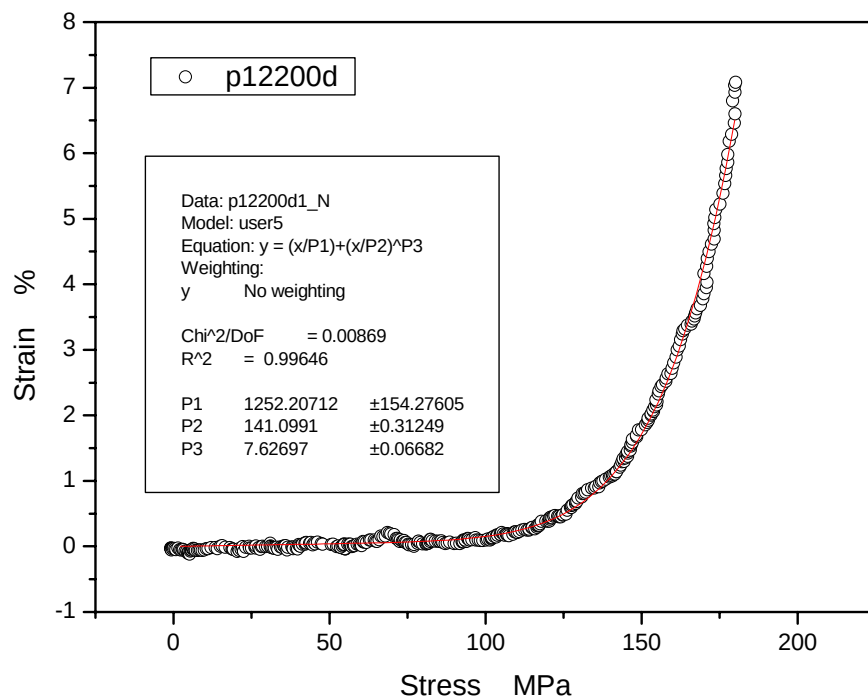
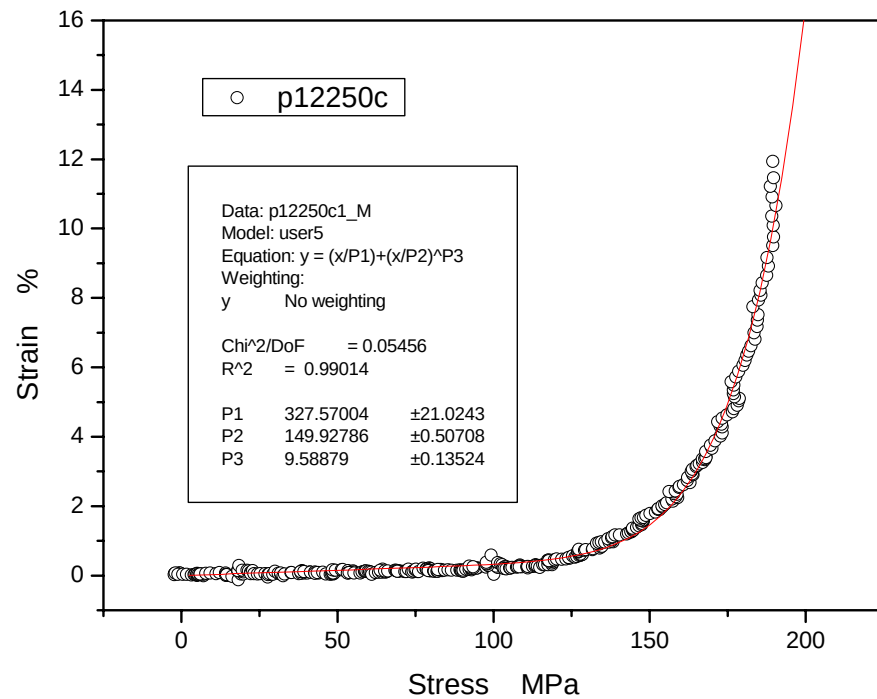


Fig 7 Representative Ramberg-Osgood fits to tensile data for p12 200 and p12 250.

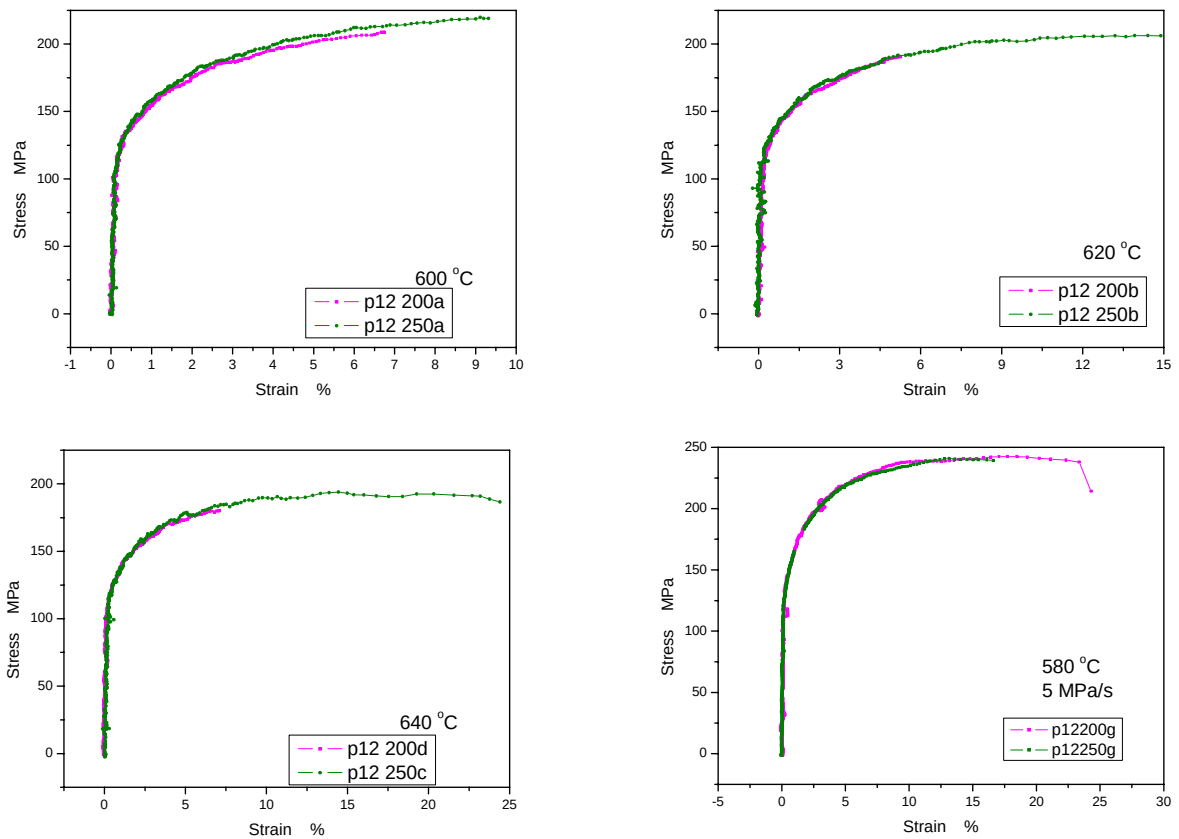


Fig 8 Comparison of tensile curves at different temperatures for steels p12 200 and p 12 250.

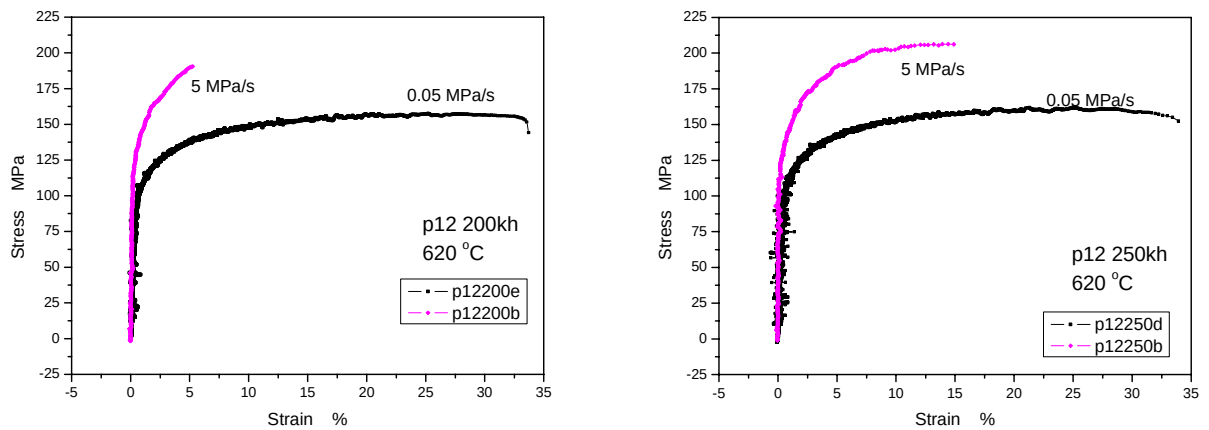


Fig 9 Effect of loading rate on tensile curves at 620 °C for steels p12 200 and p12 250.

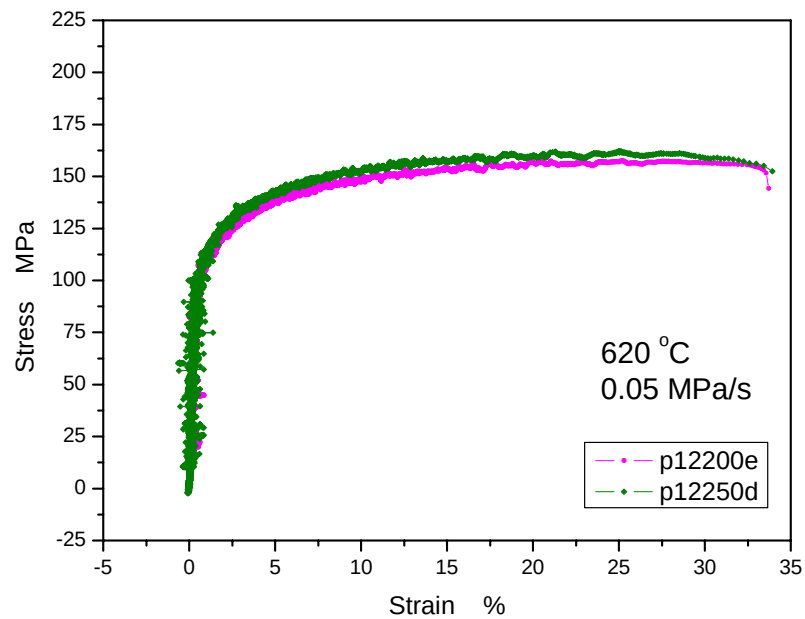


Fig 10 Comparison of tensile plots for p12 200 and 250 at the low nominal stressing rate of 0.05 MPa.

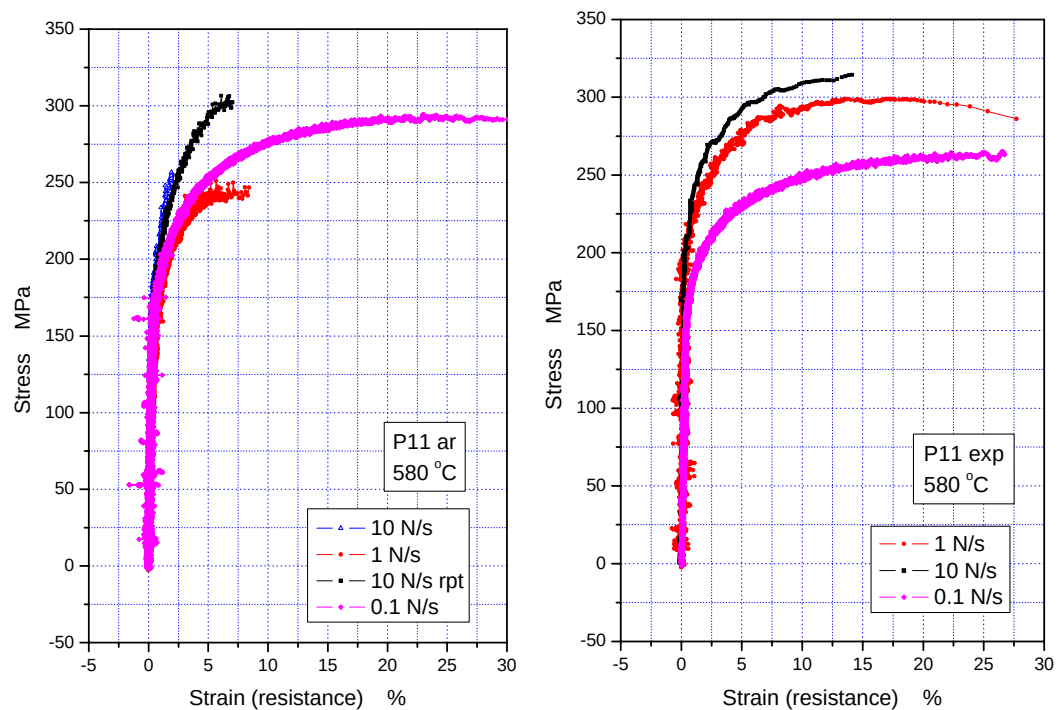


Fig 11 Comparison of tensile curves for p11 ar and p11 exp in tests at 580 °C.

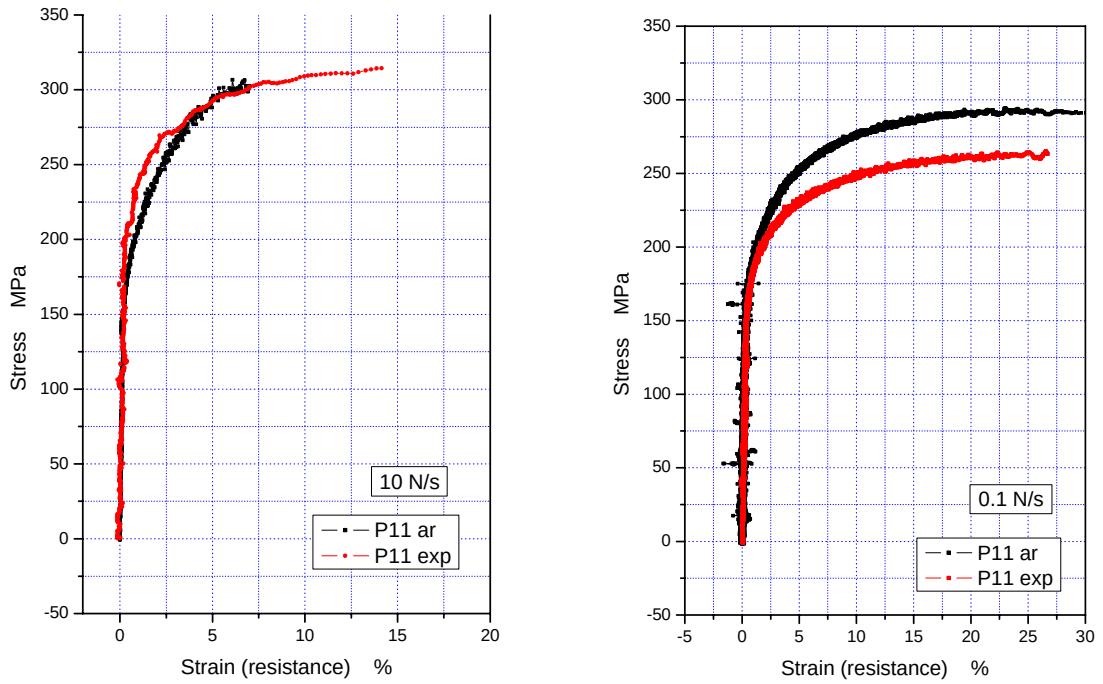


Fig 12 Comparison tensile curves at 580 °C for p11 ar and p11 exp showing effect of deformation rate.

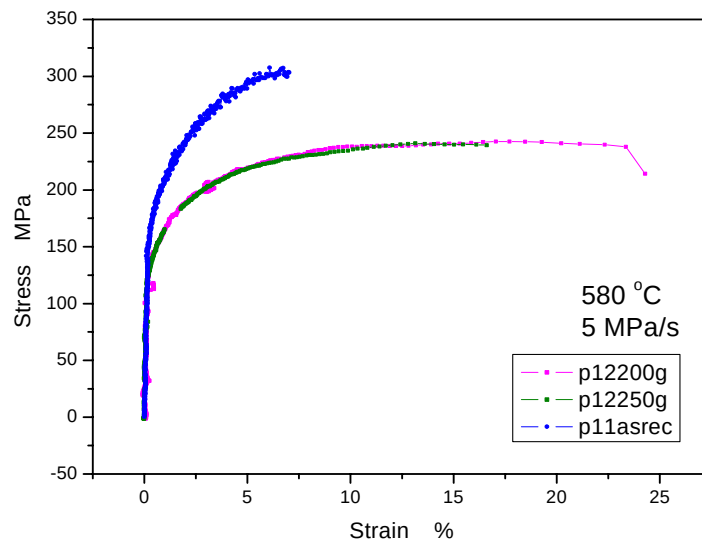


Fig 13 Comparison stress/strain curves for p11 and p12.

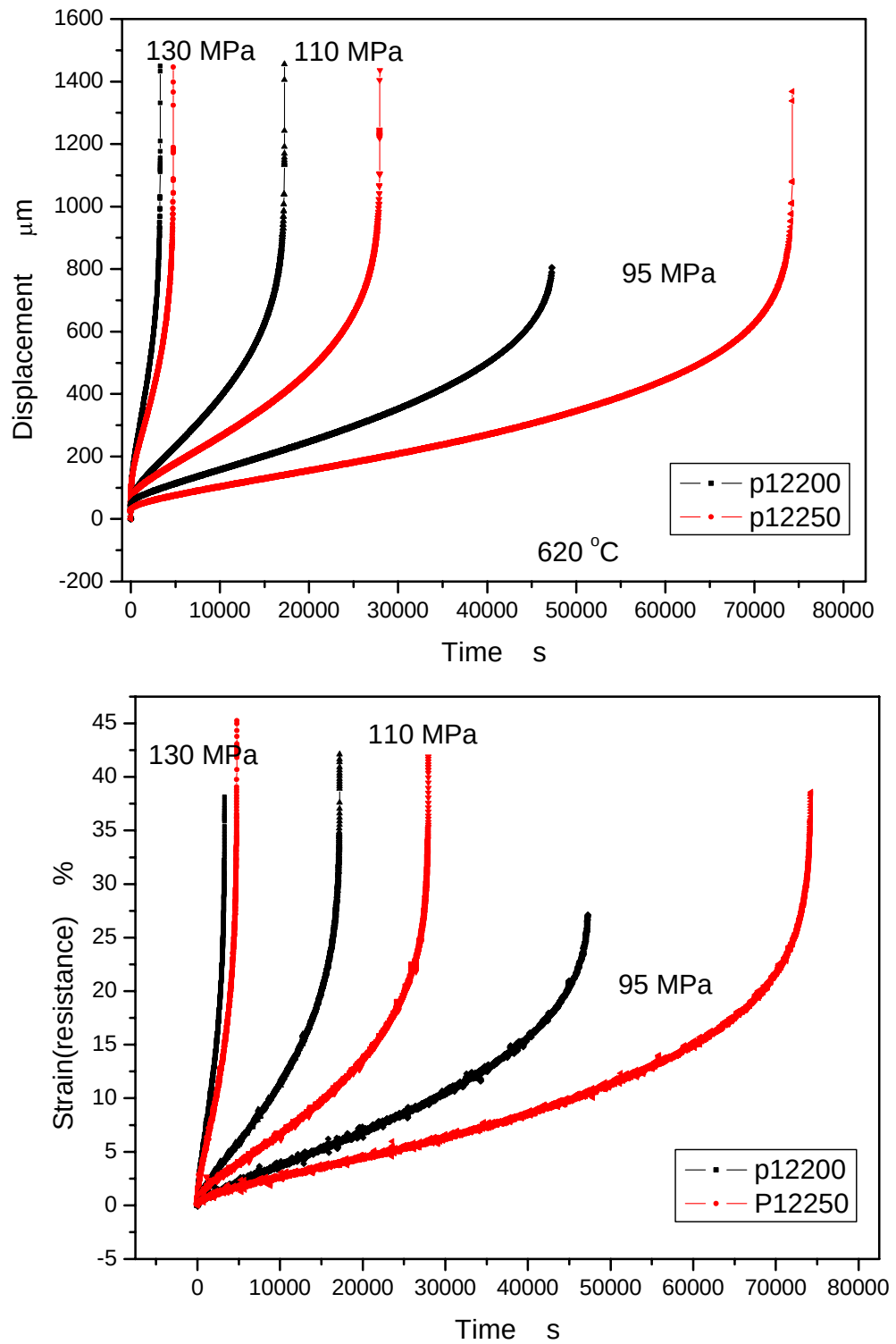


Fig 14 Displacement and strain plotted against time for constant load tests at 620 °C on steels p12 200 and p12 250.

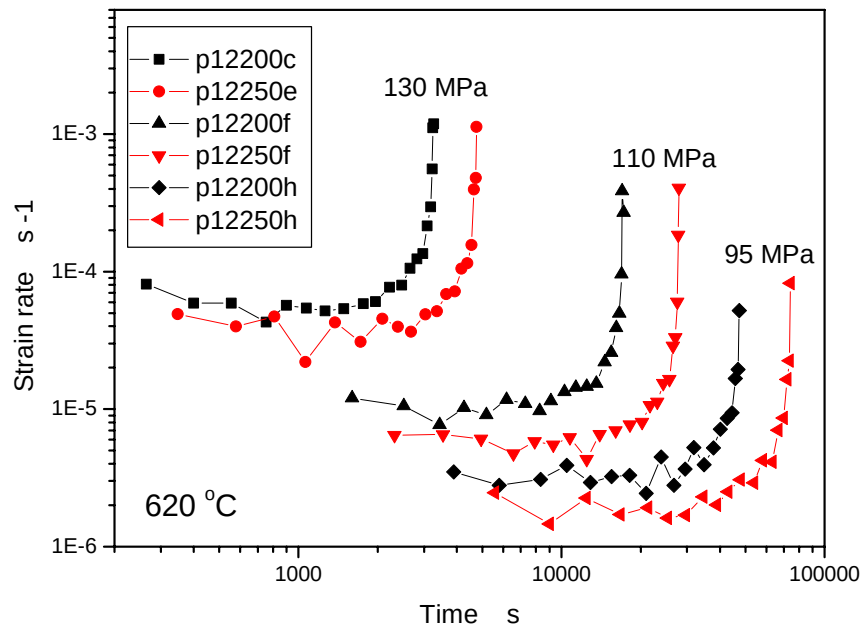


Fig 15 Strain rate plotted against time for constant load tests on steels p12 200 and p12 250.

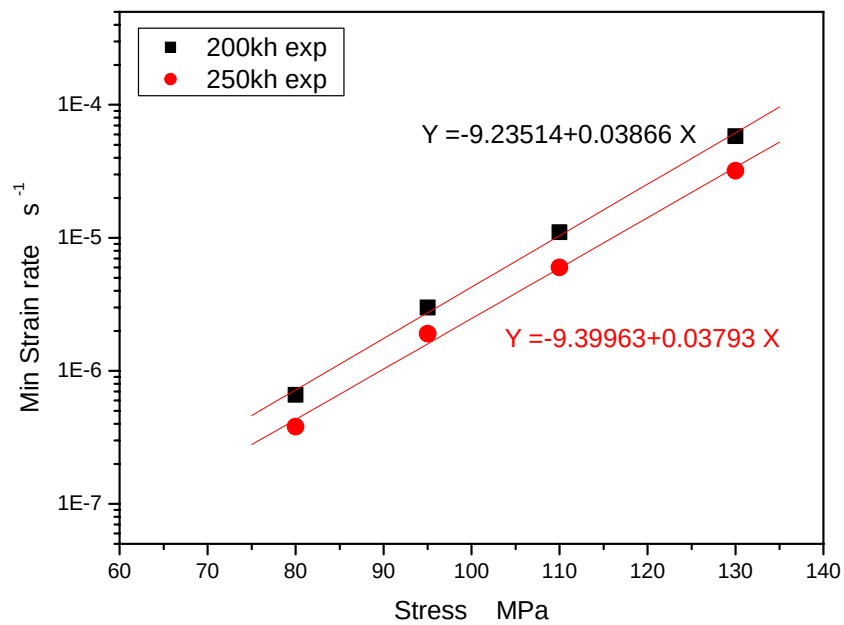


Fig 16 Strain rate plotted against stress for constant load tests on steels p12 200 and p12 250.

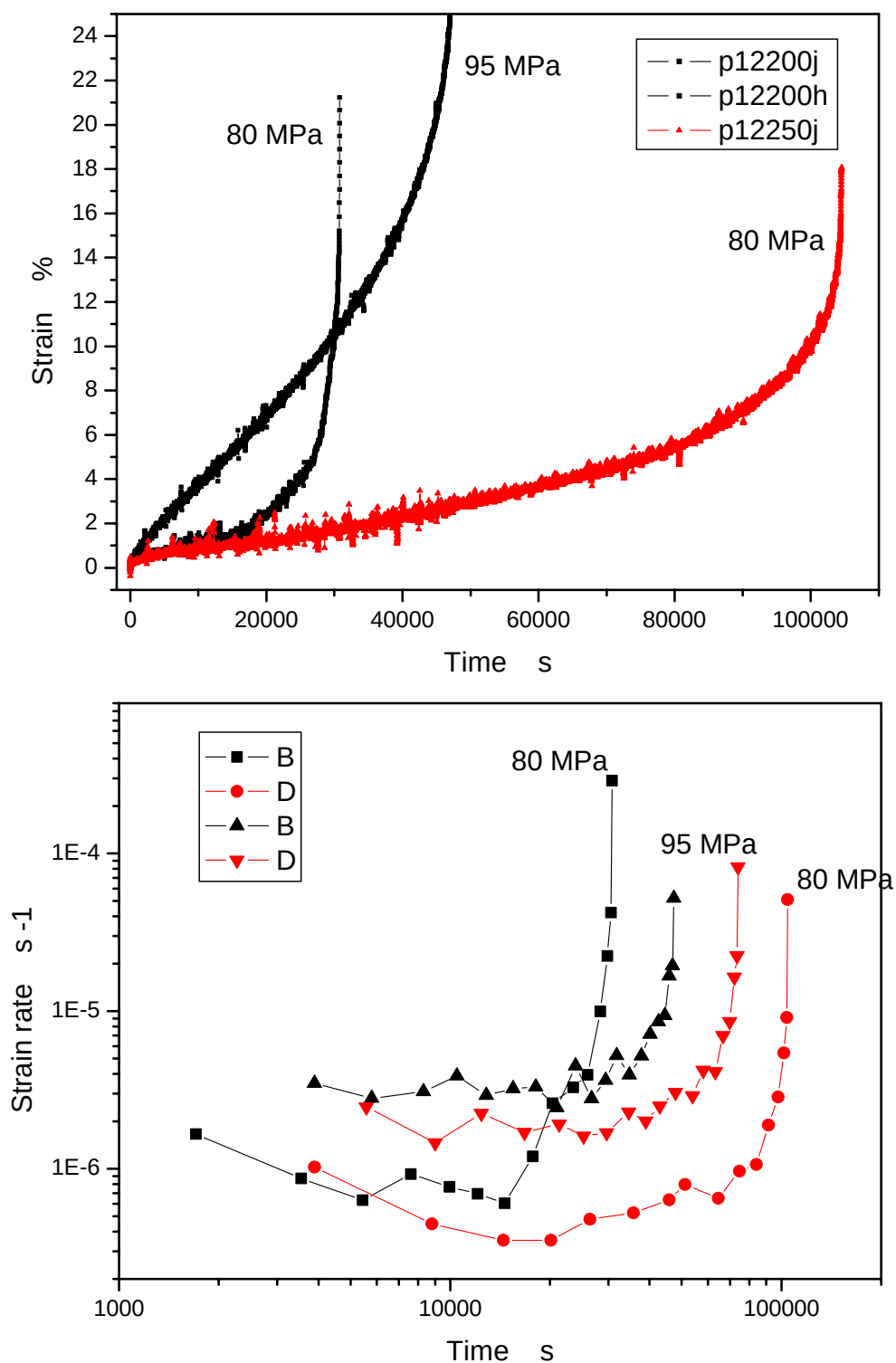


Fig 17 Strain and strain rate plotted against time for lower stress experiments on steels p12 200 and p12 250.

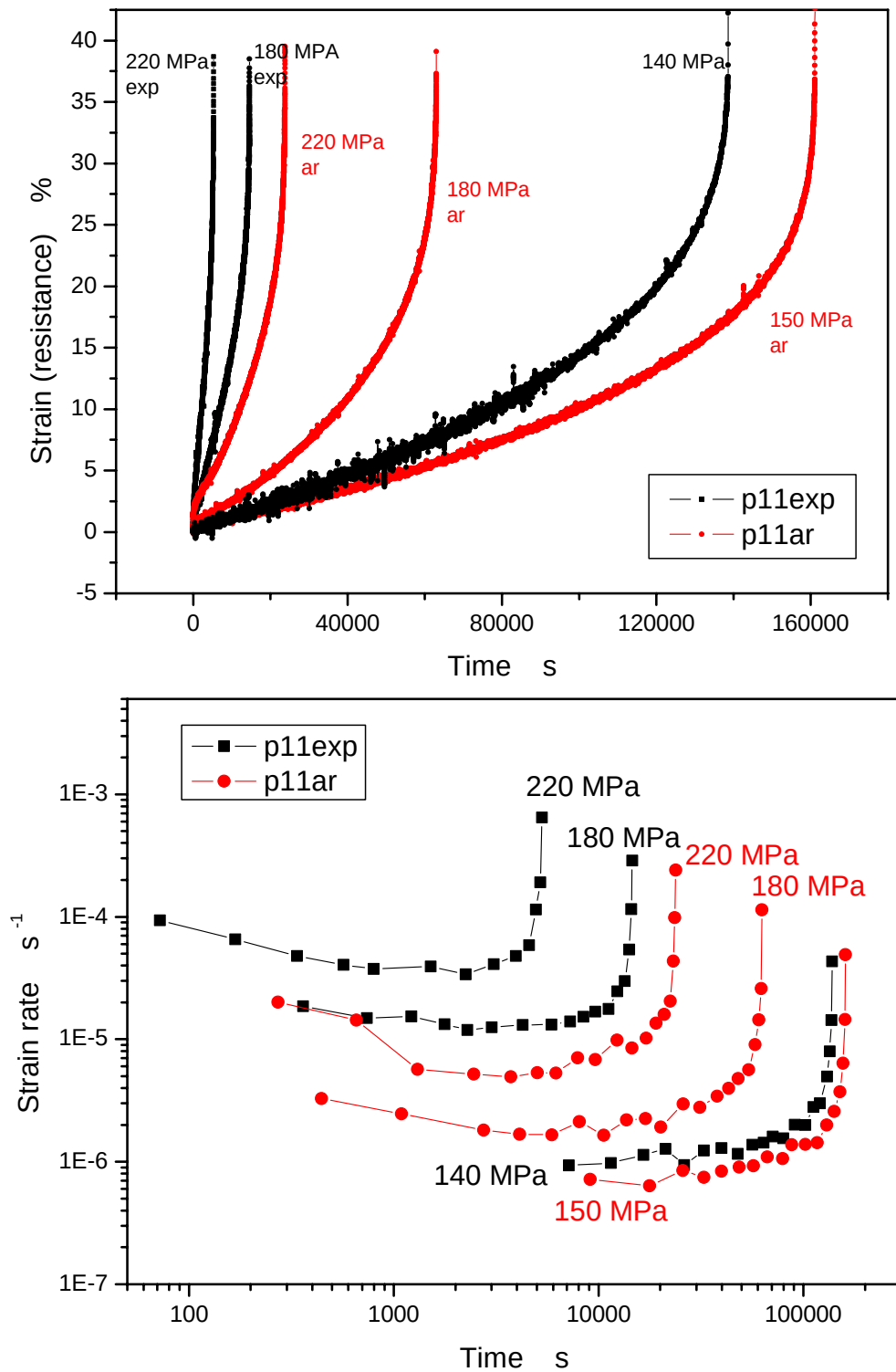


Fig 18 Strain and strain rate plotted against time for constant load experiments on steels p11 ar and p11 exp.

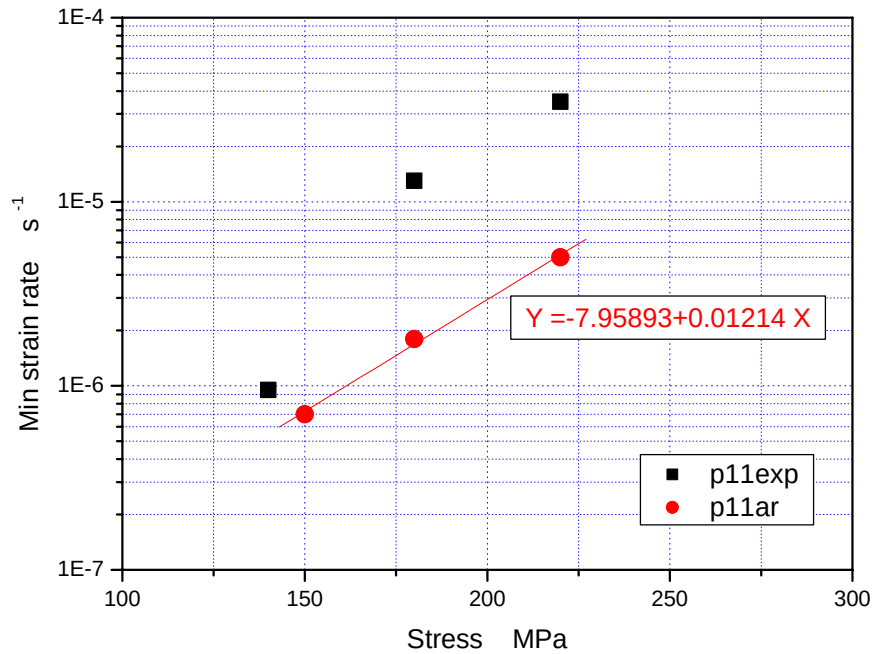


Fig 19 Strain rate plotted against stress for constant load tests on steels p11 ar and p11 exp.

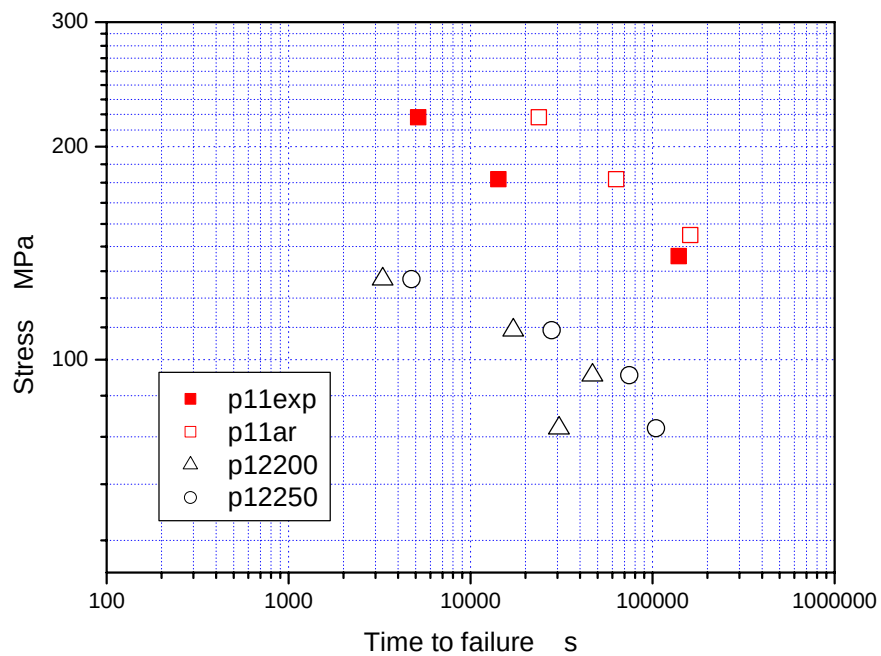


Fig 20 Stress plotted against time to failure for steels p11 and p12.

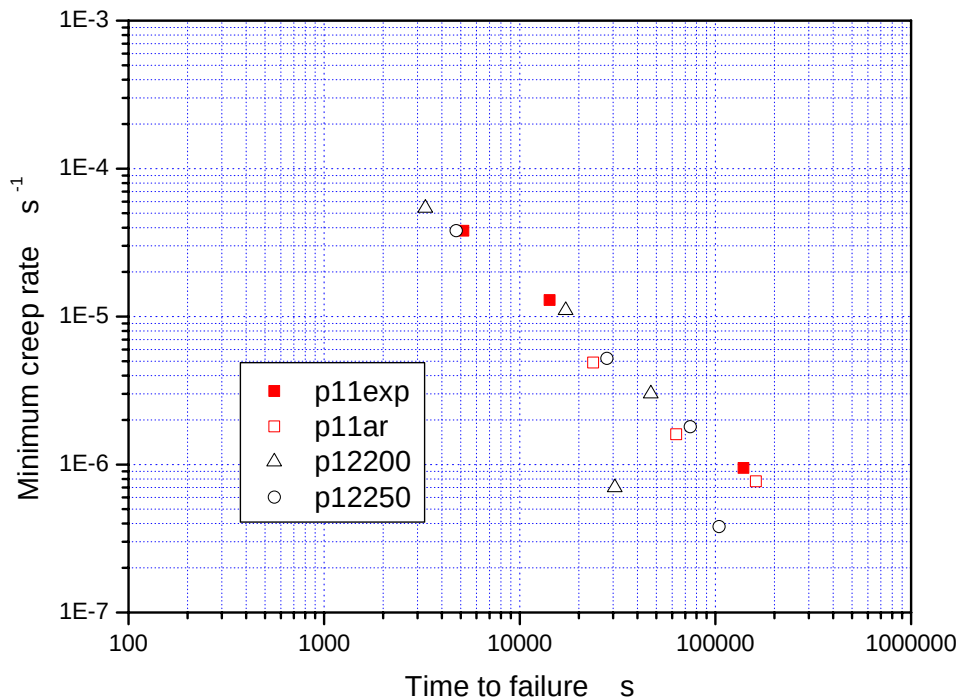


Fig 21 Minimum strain rate plotted against time to failure for steels p11 and p12.

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APPENDIX A – TEST CHRONOLOGY**ETMT Miniature Tests****Test Chronology – p11****p11 as received**

Test Code	Test	Comments
p11 ar a	Heat to 580 °C at 5 °C/s: Deform at 10 N/s to about 10% strain. Unload. Heat to 1050 °C at 5 °C/s. Cooled to RT at -5 °C/s.	
p11 ar b	Heat to 580 °C at 5 °C/s: Deform at 1 N/s. Unloaded. Cooled at 10 °C/s.	Possible slip in grips at 500 N.
p11 ar c	Heat to 580 °C at 10 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p11 ar d	Heat to 580 °C at 10 °C/s. Trial expts in sequential compression/tension.	Sample buckled at -460 N.
p11 ar e	Heat to 580 °C at 5 °C/s: Apply 300 N for creep test.	Taken to fracture.
p11 ar f	Heat to 580 °C at 5 °C/s: Apply 440 N for creep test.	Unloaded just before failure.
p11 ar g	Heat to 580 °C at 5 °C/s: Apply 360 N for creep test.	Taken to fracture.
p11 ar h	Heat to 580 °C at 5 °C/s: Deform at 0.1 N/s.	Taken to fracture.

p11 thermally exposed

Test Code	Test	Comments
p11 exp a	Heat to 580 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	Some slight problems with temperature control. Noisy.
p11 exp b	Heat to 580 °C at 5 °C/s. Apply 440 N for creep test.	Taken to fracture.
p11 exp c	Heat to 580 °C at 5 °C/s. Apply 360 N for creep test.	Taken to fracture.
p11 exp d	Heat to 580 °C at 5 °C/s. Deform at 1 N/s.	Taken to fracture. Overheated to 680 °C before test (5 °C/s up and down).
p11 exp e	Heat to 580 °C at 5 °C/s. Apply 300 N for creep test.	Software failure after 10 k s.
p11 exp f	Heat to 580 °C at 5 °C/s. Deform at 0.1 N/s.	Taken to fracture.
p11 exp g	Heat to 580 °C at 5 °C/s. Apply 300 N for creep test.	Taken to fracture: some signs of oxidation.
p11 exp h	Heat to 580 °C at 5 °C/s. Apply 280 N for creep test.	Taken to fracture: some indication of oxidation.

ETMT Tests**Test Chronology – p12 200 and p12 250****p12 200**

Test Code	Test	Comments
p12 200 a	Heat to 600 °C at 5 °C/s: Deform at 10 N/s: Unload. Cool at 10 °C/s.	
p12 200 b	Heat to 620 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 200 c	Heat to 620 °C at 5 °C/s: Apply 260 N for creep test.	Taken to fracture.
p12 200 d	Heat to 640 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 200 e	Heat to 620 °C at 5 °C/s: Deform at 0.1 N/s. Unload. Cool at 10 °C/s.	Taken to fracture.
p12 200 f	Heat to 620 °C at 5 °C/s: Apply 220 N for creep test.	Taken to fracture.
p12 200 g	Heat to 580 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 200 h	Heat to 620 °C at 5 °C/s: Apply 190 N for creep test.	Taken to fracture.
p12 200 i	Heat to 620 °C at 5 °C/s. Apply 190 N for creep test.	Taken to fracture: oxidised.
p12 200 j	Heat to 620 °C at 5 °C/s. Apply 160 N for creep test.	Taken to fracture: slight oxidation.

p12 250

Test Code	Test	Comments
p12 250 a	Heat to 600 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 250 b	Heat to 620 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 250 c	Heat to 640 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 250 d	Heat to 620 °C at 5 °C/s. Deform at 0.1 N/s.	Taken to fracture.
p12 250 e	Heat to 620 °C at 5 °C/s: Apply 260 N for creep test.	Taken to fracture.
p12 250 f	Heat to 620 °C at 5 °C/s: Apply 220 N for creep test.	Taken to fracture.
p12 250 g	Heat to 580 °C at 5 °C/s: Deform at 10 N/s. Unload. Cool at 10 °C/s.	
p12 250 h	Heat to 620 °C at 5 °C/s: Apply 190 N for creep test..	Taken to fracture.
p12 250 i	Heat to 620 °C at 5 °C/s. Apply 190 N for creep test.	Taken to fracture: oxidised.
p12 250 j	Heat to 620 °C at 5 °C/s. Apply 160 N for creep test.	Taken to fracture: some oxidation?

APPENDIX B - ETMT TENSILE TESTS

Data

A tensile test can be performed at RT 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

Analysis

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

At present ETMT testpieces are too small to attach conventional extensometers so mechanical properties are evaluated in one of two ways by plotting:

- A load or stress against displacement
- B load or stress against plastic strain (calculated from change in resistance [1-2]).

For method A the displacement measured is a combination of displacement due to the testpiece extension and displacement due to the compliance of the test rig itself (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 E or large cross-section, that does not extend significantly; this can be calculated for whatever material is inserted between the grips). Thus the corrected load/displacement curve can be calculated (Fig A.1).

The corrected stress (or load)/displacement curve can be used to estimate proof stress by drawing a line parallel to the 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. 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 associated with the defined central temperature. This effective gauge length 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 Fig A.2 from the data in Fig A.1.

For method B plastic strain, ϵ_p , is calculated from

$$\epsilon_p = \ln(\text{sqrt}(R_s/R_t)) \quad (\text{A1})$$

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 assumption is reasonably correct and the curve of stress/load against plastic strain is more or less vertical initially (Fig A.3). Proof stress is then obtained by drawing a vertical line at the required offset strain (Fig A.3). A comparison of the load(stress)/strain from displacement and resistance (Type I) calculations is shown in Fig A.4.

Type II – it has been observed in some tests at elevated temperature that stress/strain plots obtained by this method of analysis have a pseudo elastic region (Fig 5), probably because the assumption re elastic behaviour and Poisson's ratio is incorrect. Further work is needed to explore the possibility of deriving a new expression that allows elastic behaviour to be calculated as well as plasticity. 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 (Fig A.5). Initial calculations allowing for elastic changes in dimensions, which remain to be validated, indicate that total strain, ε_T , is given by

$$\varepsilon_T = \frac{M-1}{1+2M\nu} \quad (A2)$$

where ν is Poisson's ratio and M is the resistance ratio, R_s/R_t . Thus, for $\nu = 0.5$:

$$E = \frac{M-1}{1+M} \simeq \ln(\text{sqrt}(M)) \quad \text{for } 1.5 > M > 1 \quad (A3)$$

Finally

$$\nu = \frac{EM - E - \sigma}{2M\sigma} \quad (A4)$$

indicating that ν can be measured from the pseudo elastic slope if E is known. These expressions need to be confirmed by tests on material with known values of ν and E at elevated temperatures.

Calibration Tests

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 are shown in Fig A.6 compared with the results of tests on conventional macroscopic testpieces that used extensometers to measure strain. The agreement is good and confirms that plastic strain measurement through changes in resistance is an acceptable method. Further work is needed to develop an understanding of the pseudo-elastic region observed in tests on some Ni alloys. For this purpose research is under way to develop a linescan non-contact optical system to measure strain in the central 2 mm of the hot zone. The system has been

developed successfully at room temperature but needs additional research to validate its utility at elevated temperatures.

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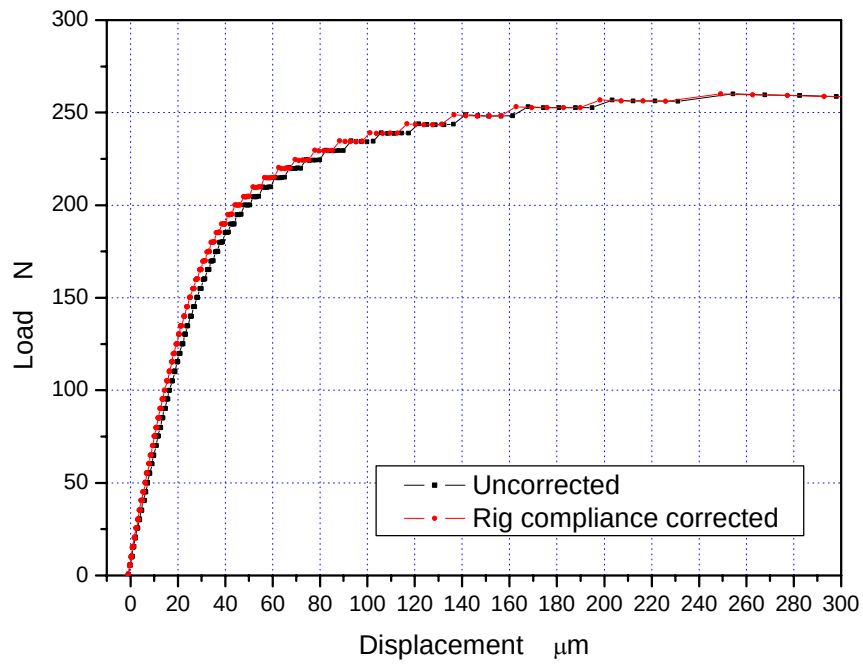


Fig A.1 Typical load (stress)/displacement curve uncorrected and corrected for rig compliance.

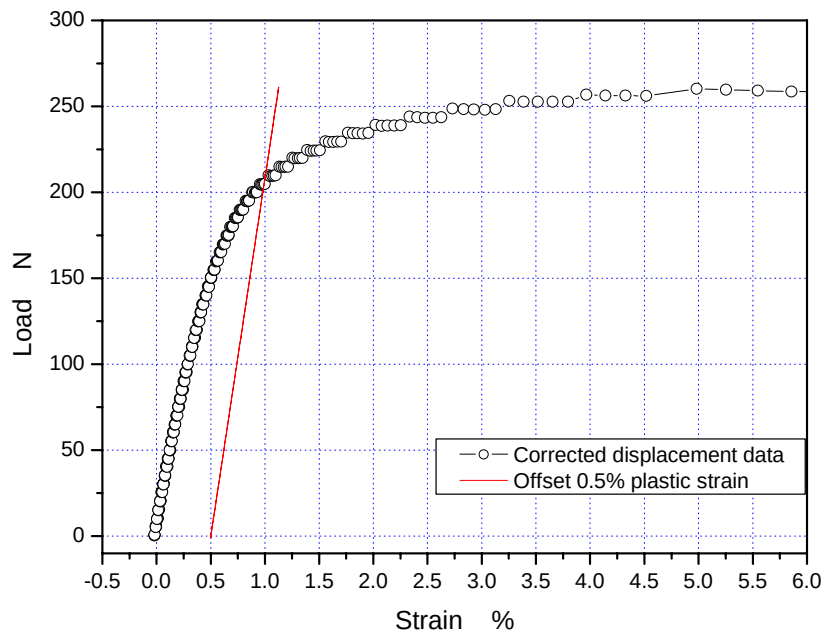


Fig A.2 Estimated load (stress)/strain curve in test at 1000 °C indicating 0.5% proof stress determination.

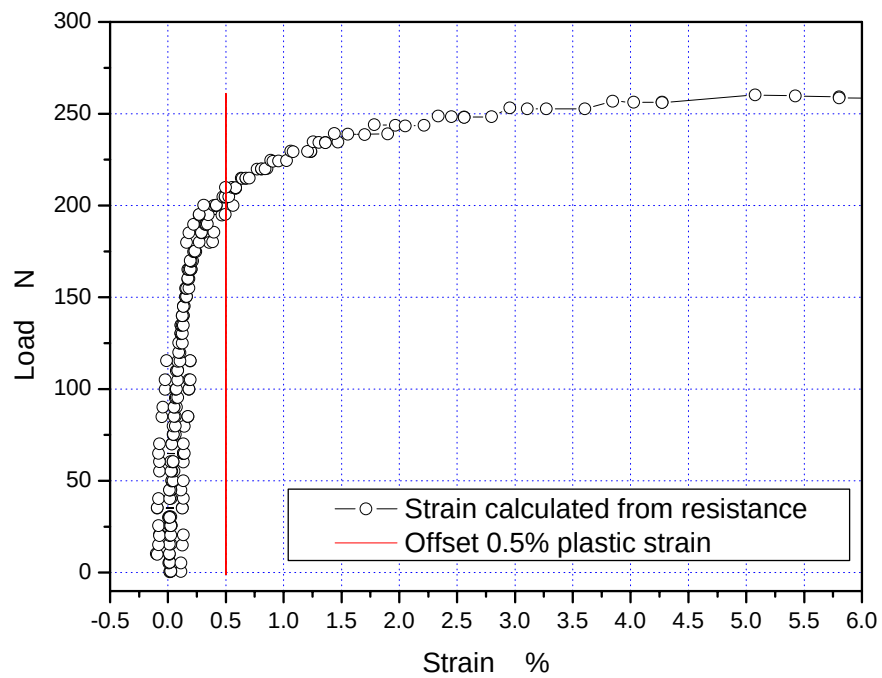


Fig A.3 Type I Load (stress)/strain curve from resistance plot.

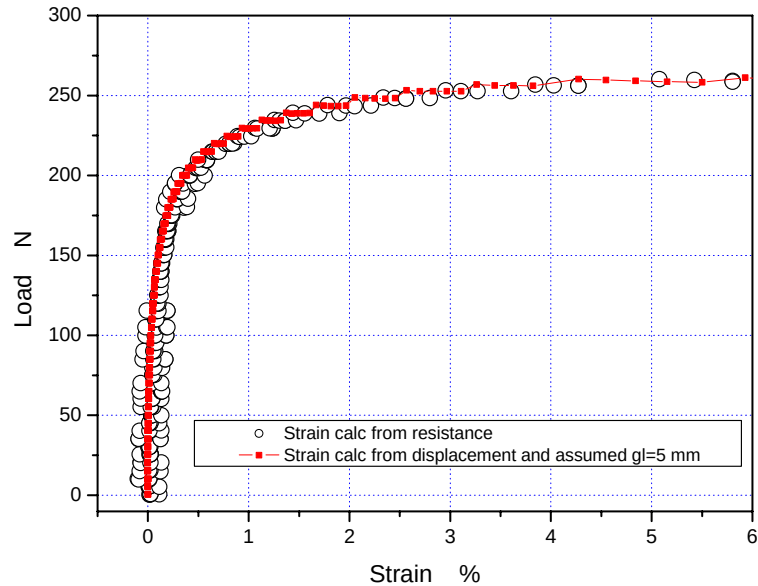


Fig A.4 Comparison of load (stress)/displacement plots from displacement and resistance calculations.

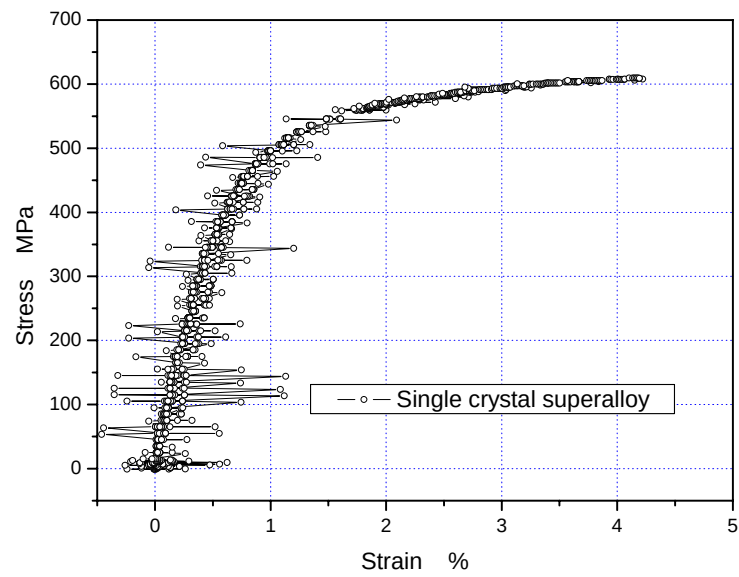


Fig A.5 Type II Stress/strain curve from resistance plot shown 'pseudo' elasticity in test at 1000 °C on superalloy single crystal material.

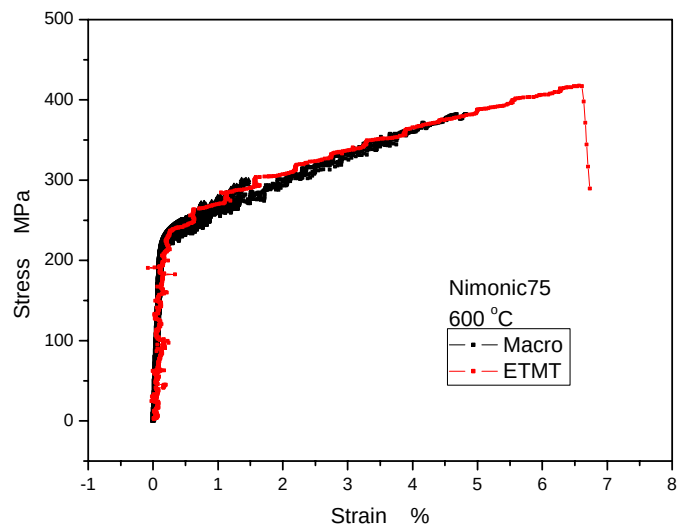


Fig A.6 Comparison of miniature ETMT uniaxial tests with macroscopic tests at 600 °C on a Nimonic 75 reference material.

APPENDIX C – ETMT TEST AND CALIBRATION PROCEDURES

This Appendix details the operating procedures for the NPL ETMT. Details of the Labview data acquisition and control software used to control the rig and record data are included in the form of printouts of the source code (diagrams). The document contains information on the background to the use of the ETMT, operating guidelines for performing ‘multi-property’ (e.g fatigue and uniaxial) tests and calibration procedures for the measurement of load, displacement and temperature, and the Data Acquisition hardware.

NPL has developed a miniaturised electrothermo-mechanical test rig (ETMT). The rig is fully computer controlled and uses electrical resistance heating. It can be operated in such a way that either ‘multi-property’ experiments, e.g. uniaxial, creep or fatigue deformations, with either in-phase or out-of-phase applied stresses, or TMF experiments may be performed. Analyses and models have also been developed to acquire thermal property and modulus data as a function of varying temperature. The rig has been used to measure and study the thermal performance of a number of different composite material and monitor phase transformations and electrical resistivity changes with varying temperature of steels, Ti alloys and TiAl intermetallics. In addition, it has been shown that the thermal fatigue properties of hard tool materials can be measured together with yield in compression and tension at temperatures up to 1600 °C. In principle it can also be used to study kinetics of microstructural changes.

The system comprises:

- An environmental chamber with electrical lead-throughs, water cooling for the testpiece grips and a facility for inert gas supply.
- A signal conditioning, amplification and control interface electronics unit, allowing computerised control/data acquisition while providing duplicate amplified signal outputs.
- Computer controlled DC heating power supply with multiple testpiece resistance and thermocouple measurement inputs. An adapter is available for infrared (pyrometer) temperature measurement. The upper temperature limit of tests is governed by the melting point and the resistivity of the test material.
- Mechanical loading assembly ($\pm 100\text{N}$, $\pm 1000\text{N}$ or $\pm 4000\text{N}$; depending on load cell fitted and ETMT version number) 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 (PC running at least MS windows 2000), which can then be later analysed with conventional PC programs.

The ETMT system is controlled using software written in a commercially available development environment from National Instruments – Labview[®]. This software system uses a modular approach to coding, and the principle of Virtual Instruments (VIs) to control experimental hardware and acquire data.

The system 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). The software to run each of these modes can be found in *ETMT with TMF.llb*

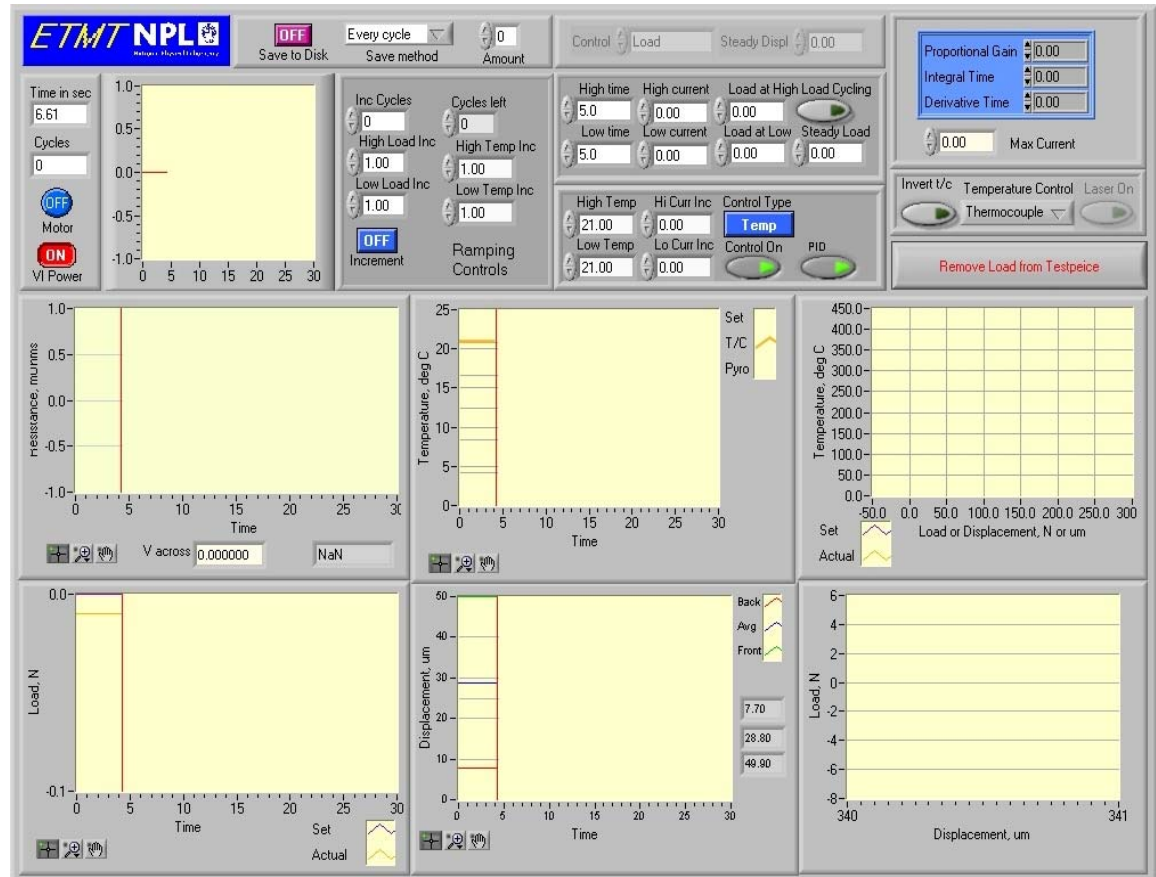


Figure C1: Front panel for ETMT software, operating in Multi property mode

Testpiece temperature control is provided via a one of two options, depending on selected mode of operation, i.e. either by direct manipulation of the applied Direct Current to the testpiece, or via the use of a PID control algorithm. In the case of using PID control, material dependent Proportional, Integral and Derivative parameters are required to be supplied by the user. Testpiece temperature measurement is via either a type R (Cp Pt – Pt13%Rh) thermocouple, or via a pyrometer.

Note : the Pyrometer for which the software is currently designed to work with has a minimum temperature of 350 °C. It should thus be noted that temperatures below this will be logged as 350 °C.

Figures C1 (above) and C2 (below) show the front panels for the two operating modes of the control software. In both cases, the screen is split approximately into thirds, with the top third containing the system controls and the lower two thirds, the real time plots of load, temperature, displacement, resistance for the test in progress, together with plots of Load or Displacement vs Temperature or each other, updated every cycle.

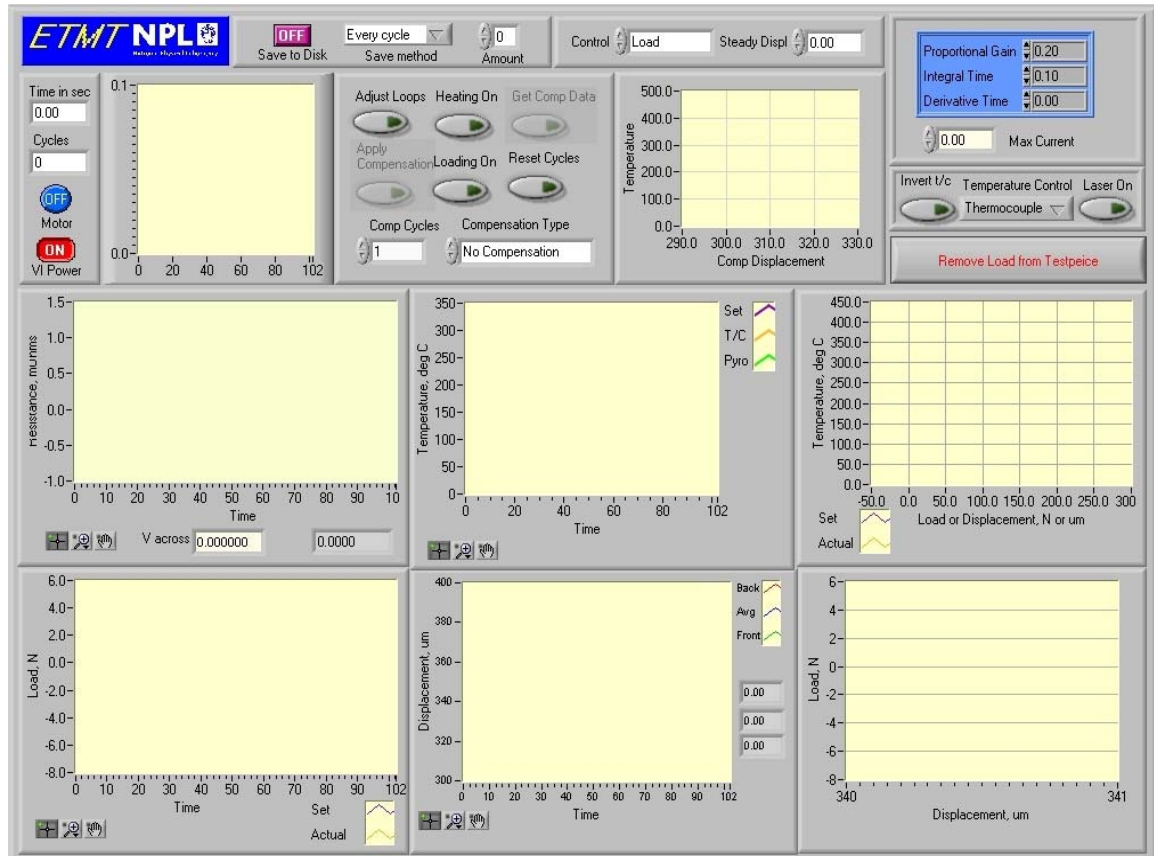


Figure C2: Front panel for ETMT Software operating in TMF mode.

The following steps should be taken to set up the equipment for an experiment.

Switch on power to computer and ETMT signal conditioning electronics.

The electronics, particularly the load cell conditioner, require several hours to stabilise. It is recommended that signal conditioning electronics are permanently energised, except when long periods of non-use are anticipated.

Limits and offsets

Within the software control program, there are three user set limits that control the operating boundaries of the ETMT. All three limits work by halting the execution of the software control program if exceeded. The limits are: Temperature; Load; Displacement. The user has the option to adjust these limits when the program is run. If a limit trips, re-run program to reset.

Note : The temperature limit will be tripped, if the thermocouple circuit is not closed, i.e. no thermocouple present, or broken lead etc. While preparing the system for testing it is recommended that the thermocouple circuit is temporarily closed via the use of crocodile clips.

Each time the program is run, the software compensates for temperature and voltage drop offsets. The line voltages for both channels are read for a specified time, an average taken and this value set to zero for the voltage drop, and 21 °C for the temperature.

Zero the load cell indicator with no testpiece in place.

Load the ETMT software control program by double clicking the shortcut icon on the computer desktop. If the software control program has already been loaded and is not visible, an icon will be present on the taskbar. Click this icon to restore the program to a visible state.

Run Program

i.e. click white arrow (run icon) at the top left corner of the window

Program Initialisation

When run, the program will prompt the user to input an operator, filename, load and temperature limits, select mode of operation, temperature measurement method and to set the material specific PID coefficients (see figure C3, below). Irrespective of the testing method to be used, while setting up the ETMT, the mode of operation should be set to multi-property, and the filename to 'test'.

Test details
Please fill in test details below

Operator

Test identifier name

Filename & path

Max Load Compression -4000.00	Max Load Tension 4000.00
Maximum Displacement 3500.00	Room Temperature 21.00
Max Temperature Limit 1600.00	Test Type Multi-property

Temperature Measurement:

PID parameters:

Figure C3: Test details dialogue box.

At this stage, it is not necessary to select either the material dependent PID coefficients, or the temperature measurement device

Setting up testpieces

Always have the dc current power switched off when changing or setting up testpieces.

The grips are moved to the appropriate position before gripping the testpiece by running the program and moving the grip attached to the motor by applying a nominal tensile or compressive load. When the correct position is reached the motor is turned off.

The capacitance displacement transducers can be moved independently from the grips to allow a choice of starting positions to be adopted for a range of testpiece length. A typical starting position is 16.5 mm grip separation, equating to a 500 μm transducer separation. Check the separation with slip gauges, which can themselves be checked using the calibrated micrometer.

To prevent damage to the test system, safety switch(es) are present which cut power to the motor once a pre-set hardware limit has been reached. Manual override button(s) can be found on the side of the box.

Position grips to give appropriate spacing using motor on/off switch.

For movement left or right a virtual load setting (set tension or compression, usually about $\pm 20\text{N}$) is required.

Turn motor off

Use the motor off switch on software front panel, or the motor power switch on the front of the signal conditioning unit to stop the motor moving the grips when in the correct position.

Insert testpiece.

Make sure dc power supply is switched off during this operation to avoid short circuits.

- a) Check that thermocouple weld looks clean with no secondary wire cross-over or contacts. It is recommended that this be done using a X10 magnifying glass.
- b) Pencil marks on the testpiece - equidistant from the thermocouple adjacent to the grips, measured and drawn before insertion, will help in making sure the thermocouple is centred.
- c) Loctite glue can be used, **on one surface only**, to aid gripping, particularly if loads up to 1000N and above are to be applied. The other surface must remain clean for the current to pass without additional resistance heating at the grips.

Zero load using software control.

Input 0 steady load and turn on motor. If Load does not adjust, check the manual override (if present) on the front of the signal conditioning unit is set to ON.

Turn on water to grips.

Stop the program.

Clicking on the 'VI Power' switch will stop the program at the end of the current cycle. When the program has stopped running, connect the thermocouple and resistance leads.

If the thermocouple circuit becomes open, a software trip is activated which halts the program at the end of the next cycle. Resistance leads can be attached locally on the testpiece via 0.1mm Pt or Pt-13% Rh wires or at the grips. If the latter method is used then the resistance measured is a combination of the integrated change in resistance along the testpiece with a temperature distribution plus resistance at the gripping points.

Fit lid to ETMT and start Argon flow.

leave the argon flowing for 15-30 min to purge the environmental chamber if required. It is advisable to have the internal fans (switch on the front of the signal conditioning unit) operating to ensure good circulation of the Argon within the ETMT.

Re-start the program.

Switch on dc power supply.

During power supply start-up, the control program will temporarily freeze. This is due to the power supply communicating with the computer, and is considered normal operation.

Check thermocouples are connected correctly; i.e. does the temperature goes positive when current applied. If not click on the "invert thermocouple" switch. Stop the control program, by clicking on the 'VI Power'.

Note: The program is re-run because experience has shown that the control software can become unstable and crash, if left running after switching on the dc power supply.

The System is now ready for testing to commence.

Run the ETMT Multi Property software.

Click on the white arrow at the top left corner of the window.

Enter test details

When prompted, see Figure 3, provide test details; i.e. Operator, test identifier, filename, and the software limit values for maximum load, temperature and displacement. The Filename and path can be either be typed directly into the space provided, or it can be selected by clicking on the folder icon, and using the windows 'file save as' dialog box.

Note: these software limit values are the limits which when exceeded the software 'trips' and shuts down the current experiment. The default values for these limits can be found in the file Thermal4.ini.

Set the test type to 'Multi-property', and select the method of temperature measurement. At this stage, material and test dependent PID values may be selected or entered by clicking the 'Select PID parameters' button.

Click OK to continue.

Testing options.

The following are a typical list of options available for performing a test, when in multi-property mode, more combinations are possible

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 load	PID manipulation incrementing current values

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

Note : 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.

If at any time during testing in multi-property mode, the user desires to instantaneously remove all loading from the testpiece, this can be achieved by clicking on the button labelled ‘Remove Load from Testpiece’. This also resets the high and low load control values to zero, and turns off load cycling. High and Low Current values remain unaltered.

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 :

Save every cycle.	Saves all data.
Save every n cycles.	Every n cycles, saves all data logged for that cycle.
Save every displacement..	Saves all data for cycle, when displacement at end of high cycle changes by more than the set value in μm .

The user can elect to start and stop saving data as often as they wish during an experiment, the new data always being added 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 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
- Turn off the cooling water to the grips
- Remove testpiece.

Proportional Integral Derivative (PID) Temperature Control

The current version of the ETMT software provides the facility to control the temperature of a testpiece, whether being tested in the TMF or Multi-property mode of operation using the PID control algorithms supplied by National Instruments as an add on tool kit to the Labview development environment. The nature of the PID control, is such that each material under test will require different coefficients for the user adjustable parameters, and possibly depending on the nature of the test being undertaken. Thus, the ETMT program provides a feature whereby material dependent PID parameters can be saved to and loaded from a text file, and edited.

PID Parameters

The parameters that can be adjusted are as follows :

Parameter	Description
Proportional Gain	Controls the current through testpiece in proportion to the error between the set point temperature and the actual temperature. Increasing this value causes a greater change in the current output for a given error.
Integral time	Causes the current through the testpiece to change at a rate proportional to the error between the set point and the actual temperature. The longer the integral time, the slower it changes.
Derivative time	Mathematically the opposite to Integral time. A Proportional + derivative controller responds to step change in error by adding to the proportional component that decays with time. The longer the derivative time, the longer the decay time. The same controller will respond to a ramp change by adding to the proportional only response. The amount added will increase as the derivative time is increased.
Maximum current	This is the Full scale current output (i.e. 100%) required to give maximum temperature.
Maximum temperature	This is the maximum temperature the material under test can be heated to i.e. its melting temperature.

The PID parameters can be used in almost any combination, except Derivative only. This is because the derivative action only knows that the error is changing, not the set point and thus cannot control to a set point.

Note 1: The maximum temperature is related to maximum current, the two values having an effect on the efficiency of the PID control algorithm. They do not relate to maximum temperature as the software defined limit at which the program trips, and could be significantly less than the maximum temperature required for control purposes.

Note 2: When using PID control in combination with Pyrometer temperature measurement and control, the maximum current should be initially set to zero (0) when the PID control is activated and gradually increased until the pyrometer registers a temperature above its minimum value (350 °C). The maximum current can then be increased to the value appropriate for the experiment.

The PID parameters, with the exception of maximum temp can be edited at any time during TMF testing and any time so long as PID control is active in Multi-property testing. The controls can be found at the top right corner of the main window (see Figures C1, C2). These controls are initialised with the values the user selects when prompted for test details. Any changes to these values during testing take effect immediately.

Recalling, Saving and editing Material dependent PID parameters

During the initialisation stage of the ETMT Thermal fatigue software, when the user is being prompted for test details, an option exists to select PID parameters. Clicking the button 'PID parameters – Select' will bring up a new window, see Figure C4, below.

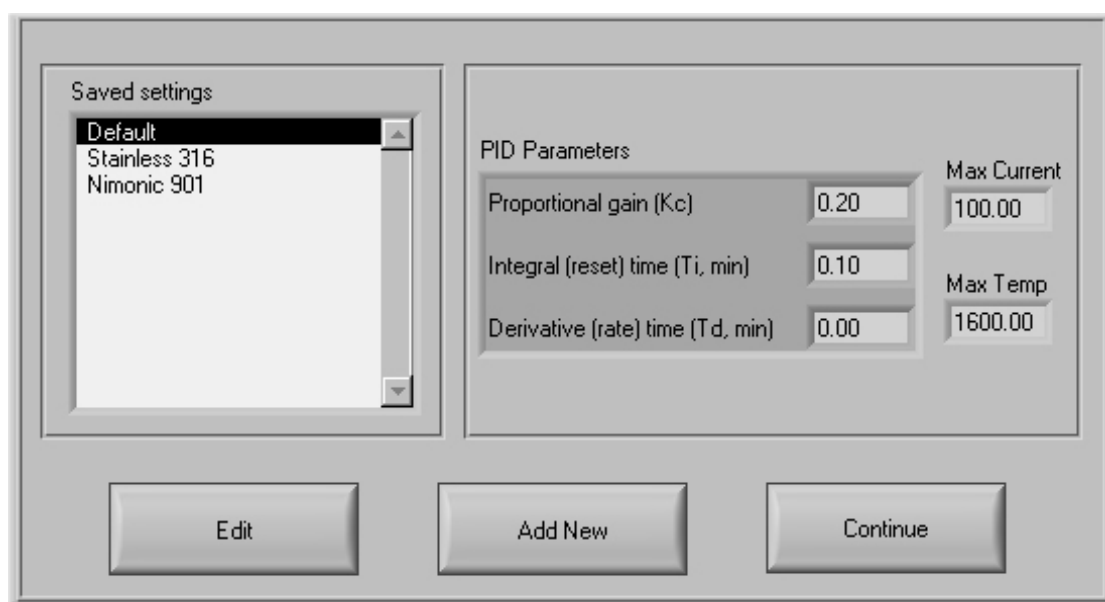


Figure C4: PID parameters initialisation front panel

The panel as shown in Figure C4, above, enables users to select one of many saved set material/test dependent PID parameters. Clicking on any of the saved settings in the dialogue box on the left will load the parameter values from a file (ETMT_PID

parameters.ini located in the computers system directory) and display them on the right. By clicking continue, the user is returned to the 'Enter test details screen', and the selected PID parameters loaded into the ETMT control software.

The user also has the option of editing an existing set of PID parameters, and creating a new set, to save to the data file. Clicking on 'Edit' will bring up the screen shown in Figure C5, below. The existing values will be loaded into the relevant controls, and the user can now edit them as they see fit. Clicking OK saves the edited values to the data file, and returns the user to the screen shown in Figure C4.

Figure C4: Editing PID parameters.

Figure C5: adding a new set of parameters

If the user clicks 'Add New', the screen shown in Figure C6 will appear. A material identifier is required for the parameters to be filed under in the data file, and is entered at the top of the screen. This is the identifier that will appear in the list box on the left of Figure C4. Values for the various parameters can be entered by the user. Clicking 'OK' saves these parameters to the data file, and returns the user to the screen shown in Figure C4. Clicking 'Cancel' also returns the user to the same screen, but without updating the data in the data file.

Note: Since the data file ETMT_PID_coeffs.ini is a text file, it can be opened and edited using any conventional text editor. Any changes made will be visible the next time the above screens are accessed.

Software front panel – Multiproperty tests

The panel enables a number of parameters to be selected and displays information in six graphs.

The six main graphs are

- Group 1. Resistance Vs time
- Group 2. Temperature Vs time
- Group 3. Resistance Vs temperature
- Group 4. Load Vs time
- Group 5. Displacement Vs time, from front or back transducer or an average of both
- Group 6. Load Vs displacement.

Graphs 1, 2, 4 and 5 are updated continuously
 Graphs 3 and 6 are updated after each cycle.

A small graph at the top of the program window displays the dc current history.

Data files

The data is saved into a tab delimited ASCII text file, with columns as follows :

Column	Parameter	Column	Parameter
1	Time, seconds	6	Displacement, μm
2	Cycles	7	Potential drop, Volts
3	Load, Newtons	8	Resistance, milliohms
4	Thermocouple Temperature, $^{\circ}\text{C}$	9	Current, Amps
5	Pyrometer Temperature, $^{\circ}\text{C}$		

Front Panel Controls

Cycle Controls (Multi property) :

Low current	Sets dc current at low time
High current	Sets dc current at high time
Low time	Sets time for low current and load
High time	Sets time on for high current and load
Load at High	Sets load value at high time
Load at Low	Sets load value at low time
Steady Load	Sets load applied to testpiece (Load cycling off)
Load cycling	On/off button for load cycling

Temp controls (Multi property) :

High temp	Temperature to control to at high time
Low temp	Temperature to control to at low time
High current inc	Amount by which to adjust current, to control temperature during high cycle, when not under PID control
Low current inc	Amount by which to adjust current, to control temperature during low cycle, when not under PID control
Control type	Select between temperature and resistivity control
Control on	On / off button for temperature control
PID	On / off button for PID control of temperature. PID controls not visible unless on.

Ramping controls (Multi property):

Inc Cycles	Apply increment every n cycles
Cycles left	No cycles left before increment applied
High load Inc	Increment to apply to high load control
Low load Inc	Increment to apply to low load control
High temp inc	Increment to apply to high temperature control
Low load inc	Increment to apply to low temperature control

Save data controls:

Save data	On / off switch to enable saving data to disc
Save method	Select save to disc method
Amount	Relative to save methods.

PID controls:

Proportional Gain	Adjust the amount of proportional gain.
Integral time	Adjust the Integral time.
Derivative time	Adjust the derivative decay time.
Max Current	Adjust maximum current that can be applied to the testpiece.

Temp Control

Thermocouple / Pyrometer	Select temperature measurement device to use to control temperature with
Laser on	Switches on the pyrometer measuring laser
Invert T/c	Invert thermocouple

Remove Load from Testpiece

Reset switch to remove immediately loading from testpiece

Motor

On / Off	Software On / Off switch to control the ETMT drive motor.
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CALIBRATION:

Calibration of the ETMT is achieved by the use of a set of ancillary calibration equipment and a set of four Labview VIs which are stored in the c:\ETMT\ETMT Calibration V2.llb library. A Shortcut to the library is present on the computer desktop. The purpose of calibration is to ensure that the ETMT system gives reliable traceable measurements. The calibration constants are stored in an initialisation file for the ETMT, thermal 3.ini which is stored in the c:\windows directory. A Printout of thermal*.ini can be found in the relevant calibration folder for the ETMT.

The different calibration procedures are:

1. DAQ card voltage measurements (Thermocouple, Voltage Drop).
2. Load.
3. Displacement.
4. Current.
5. Cycle time.
6. Reference sample
7. Dimensional measurements.

The calibration certificates, or a copy thereof, for all the transfer mechanisms used are kept in a folder near to the ETMT, titled 'ETMT Equipment and Calibration Records'

All calibration values are to be entered into a calibration record, kept in the same folder. Having completed a calibration run, the equipment record for the appropriate ETMT should be updated, and initialled.

The different calibration procedures are described below.

1 *DAQ Card Voltage Measurements*

Calibration of the DAQ (Digital Acquisition) for voltage measurements is carried out by the following procedure:

- 1.1 Locate the screw terminals inside the ETMT enclosure for the leads for potential drop measurements (Green and Light Blue), and for the thermocouple temperature measurements (Yellow and Grey).
- 1.2 Connect a voltage source,(such as Time Electronics DC Voltage Standard 2004) to the analogue input channel to be calibrated and switch on.
- 1.3 Connect a calibrated DVM in parallel to the voltage source and switch on.
- 1.4 Switch on ETMT signal conditioning unit (if not already energised) and leave for at least 30 minutes to warm up.
- 1.5 Load and PD / Thermocouple calibration VI, found in 'ETMT Calibration V2.11b'.
- 1.6 Select Channel to be calibrated (i.e. Thermocouple or Potential Difference), and run program by clicking on the white arrow at the top left corner of the window.
- 1.7 Starting at 0.0 V, increase the voltage supplied by the source in twenty evenly spaced increments to the maximum voltage for the channel being calibrated (1V for Potential drop, 20mV for Thermocouple).
- 1.8 At each voltage increment, read the DVM voltage and type in to the control labelled 'DVM Voltage'.
- 1.9 Press Use Value button on front panel.
- 1.10 When all increments have been completed, press Calculate Fit button, to generate a best fit plot of the data.
- 1.11 The calibration data will be automatically saved to a file of the kind '*P_Drop 3-6-99*' the exact name and date varying with channel calibrated and date carried out. If more than more than one calibration is carried out on the same channel on the same date, the user will be prompted to overwrite existing data file.
- 1.12 Print out current window.
- 1.13 Write your name on print-out and date.
- 1.14 Record Channel Voltage Offset and Channel Voltage Scaling values in the ETMT calibration record.
- 1.15 Store print-out in ETMT calibration and equipment record folder kept near the ETMT.

2 Load

The measurement of load, the correct setting of load from values specified on the front panel, and the read-out of the front panel on the conditioning unit are calibrated by using the load calibration VI, and a specially designed calibrated load cell.

This calibration procedure assumes that the ETMT load cell and conditioner have already been set up and calibrated at least once. If you are setting up a load cell for the first time, please refer to the RDP E308 manual, located in the miscellaneous manuals boxfile nearby the ETMT.

- 2.1 Switch on calibrated load cell signal conditioner.
- 2.2 Switch on ETMT signal conditioner unit, and leave to warm up for 0.5 hour.
- 2.3 Load and run load calibration VI (front panel and block diagram given in Figure C3).
- 2.4 Remove specimen clamps from ETMT.
- 2.5 Adjust grips for correct spacing to replace specimen clamps with calibrated load cell.
- 2.6 Press **Motor On** button to switch motor on, Switch changes colour from Green to Blue.
- 2.7 If gap is too small set **Control Load Voltage** to +0.5 and motor will move grips apart.
- 2.8 If gap is too large set **Control Load Voltage** to -0.5 and motor will move grips together.
- 2.9 When grip separation is correct switch motor drive off by pressing Motor On button again (Switch colour changes from Blue to Green).
- 2.10 Firmly clamp calibrated load cell into position.
- 2.11 Set **Control Load Voltage** on Front Panel to -9.5.
- 2.12 Press **Motor On** button to switch motor on. Reading on calibrated load cell and ETMT signal conditioner unit should decrease to close to -1000 N.
- 2.13 Note : If the ETMT load cell indicator differs wildly from the calibrated load cell, it will need reconfiguring. Refer to RDP E308 Manual.
- 2.14 Type exact readings from ETMT Front Panel (**Front Panel Load**) and calibrated load cell signal conditioner (**Actual Load**) into front panel of load calibration VI.

- 2.15 Press **Use Value** button
- 2.16 Press up arrow on **Control Load Voltage** (note increment has been pre-set to 0.5 V).
- 2.17 Wait until load readouts have stabilised, typically a few seconds.
- 2.18 Repeat steps 13-16 until **Control Load Voltage** reaches 9.5V.
- 2.19 Press **Calculate Fit** button, to generate a best plot fit of the data
- 2.20 The calibration data will be automatically saved to a file of the kind '*Load Calibration 3-6-99*' the exact date varying with the date the calibration was carried out. If more than one calibration is carried out on the same day, the user will be prompted to 'overwrite existing data file'.
- 2.21 Print out current window.
- 2.22 Write your name on print-out and date.
- 2.23 Record the **Load Computer Offset**, **Load Computer Scaling**, **Load Voltage Offset** and the **Load Voltage Scaling** in the ETMT calibration record.
- 2.24 Store print-out in the relevant ETMT calibration and equipment record folder kept near the ETMT

Having run the load calibration VI, it is now necessary to check the calibration, and if necessary manually adjust the scaling and offset calibration values. This is done using the Load Cal Check.VI. It is assumed that the calibrated loadcell is still in place, following the primary load cell calibration, and all the electronics have been energised for at least half an hour.

- 2.25 Load and the calibration program 'Load Cal Check.VI'.
- 2.26 Enter the calibration constants, Load Computer Offset, Load Computer Scaling, Load Voltage Offset and the Load Voltage Scaling .
- 2.27 Run 'Load Cal Check.VI', by clicking on the white arrow at the top left corner of the window.
- 2.28 Set the target load to either -1000 N or -100 N, depending on load cell fitted to the ETMT.
- 2.29 Compare the output load with the load displayed by the calibrated load cell.
- 2.30 If required, adjust the calibration constants until the output load is comparable with the calibrated load.

- 2.31 Set the Target load to 0 N, and compare the output load to the load displayed by the calibrated load cell.
- 2.32 If required, adjust the calibration constants until the output load is comparable with the calibrated load cell.
- 2.33 Set the target load to either 1000 N or 100 N, depending on load cell fitted to the ETMT, and compare the output load to the load displayed by the calibrated load cell.
- 2.34 Adjust the calibration constants as required.
- 2.35 Repeat steps 2.28 to 2.33 until there is a good agreement between the output load and the calibrated load over the entire calibrated range.
- 2.36 Record the adjusted calibration constants and offsets in the ETMT calibration record.

3. *Displacement*

The displacement transducers are calibrated using the displacement calibration VI, and an ASL SLVC displacement transducer, calibrated against a laser interferometer by the Centre for Length Measurement (CLM) at NPL.

The displacement transducers used in the ETMT are capacitance devices, and their output response is best described by a quadratic relationship between displacement and signal. For that reason, a quadratic fit with three coefficients is used to fit the output signal to the actual displacement in the displacement calibration VI. The calibration procedure is as follows:

- 3.1 Switch on calibrated displacement transducer signal conditioner, and set the readout to mm.
- 3.2 Switch on ETMT signal conditioner unit (if not already energised), and allow to warm up for 0.5 hours.
- 3.3 Load and run displacement calibration VI (front panel and block diagram given in Figure C6).
- 3.4 Remove specimen clamps from ETMT.
- 3.5 Adjust grips to correct spacing for calibrated displacement transducer.
- 3.6 Press **Motor On** button, changes colour from green to blue, to switch motor on.
- 3.7 If gap is too small select **widen grips**, changes from green to blue, and the motor will drive the grips apart.

- 3.8 If gap is too large deselect **widen grips** (changes colour from blue to green) and the motor will drive the grips together.
- 3.9 When grip separation is correct switch motor drive off by pressing Motor On button again.
- 3.10 Fit the transducer mounts, and clamp the calibrated displacement transducer into position, adjusting position of calibrated displacement transducer to give positive value close to zero.
- 3.11 Adjust position of ETMT displacement transducers so that they are touching their respective targets.
- 3.12 Null calibrated displacement transducer to zero.
- 3.13 Select **Widen Grips** (switch turns blue)
- 3.14 Briefly press the button Motor Nudge, until the calibrated displacement transducer readout is approximately 0.125 mm
- 3.15 Type the exact reading from calibrated displacement transducer readout (in mm), in the control labelled Actual Displacement on the front panel.
- 3.16 Press Use Value button.
- 3.17 Press Nudge Motor to move grips apart. Aim to obtain movements which will give at least 20 increments over the nominal ETMT transducer range of 2.0 mm.
- 3.18 Repeat steps 3.14-3.17 until calibrated displacement exceeds 1000 μm .
- 3.19 Press Calculate Fit button.
- 3.20 The calibration data will be automatically saved to a file of the kind '*Displacement Calibration 3-6-99*' the exact date varying with the date the calibration was carried out. If more than one calibration is carried out on the same day, the user will be prompted to 'overwrite existing data file'.
- 3.21 Print out current window.
- 3.22 Write your name on print-out and date.
- 3.23 Record the three Fit Back and the three Fit Front values (selected by clicking on array selection arrows) in the ETMT calibration record.
- 3.24 The displacement constants needed for the ETMT initialisation file (thermal4.ini) are given by:

Front Fit [0]	DispFrontOffset
Front Fit [1]	DispFrontLinConst

Front Fit [2]	DispFrontSquareConst
Back Fit [0]	DispBackOffset
Back Fit [1]	DispBackLinConst
Back Fit [2]	DispBackSquareConst

- 3.25 Store the printout in the relevant ETMT calibration and equipment record folder, kept near the ETMT.

4. *Current*

- 4.1 The current supplied by the power supply is calibrated by the following procedure:
- 4.2 Switch on ETMT signal conditioning unit (if not already energised) and warm up for ½ hour.
- 4.3 Inset Reference Testpiece of known resistivity into the ETMT, and fix into place.
- 4.4 Load 'Current Calibration.IV'.
- 4.5 Enter the Resistivity (in $n\Omega m$) for the test piece, along with cross-sectional area (mm^2), and reference length (mm) into the relevant spaces provided. The reference length is the distance over which potential drop is measured, if using the twin probes from the resistivity kit, this distance is 15.3 mm.
- 4.6 Enter the Load Comp Offset and the Load Comp Scaling values from the load calibration previously carried out in the spaces provided.
- 4.7 Connect a calibrated DVM to the potential drop probes, and place nearby the ETMT.
- 4.8 Turn on the HP Power supply, and wait for it to initialise.
- 4.9 Run 'Current Calibration.VI' by clicking on the white arrow at the top left corner of the active window.
- 4.10 Set the Control Current to zero.
- 4.11 Using the probes, measure the potential drop over the reference length and type the reading in the relevant space on the front panel. Two measurements are made, in the forward and reverse directions, to account for any thermal EMFs that may be present.
- 4.12 Click on the Orange button, Use Value
- 4.13 Increment the control current from 0 to a maximum of 200A, to give 20 increments.
- 4.14 Repeat steps 4.11-4.12 for each increment.

- 4.15 Press the Calculate Fit Button
- 4.16 The calibration data will be automatically saved to a file of the kind '*Current Calibration 3-6-99*' the exact date varying with the date the calibration was carried out. If more than one calibration is carried out on the same day, the user will be prompted to 'overwrite existing data file'.
- 4.17 Switch off power supply.
- 4.18 Print out current window.
- 4.19 Write your name on print-out and date.
- 4.20 Record the Control Scaling, Control Offset, Control Fit Error, Reading Scaling, Reading Offset, Reading Fit Error values in the ETMT calibration record.
- 4.21 Store the printout, and calibration record in the relevant ETMT equipment and calibration folder, kept near the ETMT.

5. *Cycle Time*

The cycle time calibration is made by comparing the time taken for 20 cycles of the Thermal Fatigue program to complete against the expected time. The procedure is as follows:

- 5.1 With ETMT signal conditioning unit switched on, load Thermal Fatigue program.
- 5.2 Set **High Time** and **Low Time** to 5 seconds.
- 5.3 Run program.
- 5.4 Time the duration of the program from when **Cycle** registers 5 until Cycle registers 25.
- 5.5 Record the elapsed time. Calculate the new **TimeAdjust** value by multiplying the old TimeAdjust value in the initialisation file c:\windows\thermal3.ini by 200/elapsed time.
- 5.6 Edit the value of Time Adjust in the initialisation file to the new value.
- 5.7 Record the new Time Adjust value in the calibration record, and store in the relevant ETMT equipment and calibration folder, kept near the ETMT.

6. *Reference Sample and Dimensional Measurements*

Reference Sample

The overall performance and the calibration of the ETMT system, including power supply are checked by the use of a pure titanium reference sample. Pure titanium is used because it undergoes a phase change at a fixed, well defined temperature (can be used to check thermocouple performance), and it is easy to spot weld leads to this material. The Titanium is supplied in the form of a thin strip (40x2x1 mm), with a Pt/Pt-Rh thermocouple (type R) and Pt or Pt-Rh potential drop leads spot welded to the sample.

- 6.1 Switch on the ETMT signal conditioner unit (if not already energised) and leave for ½ an hour to warm up.
- 6.2 Load Thermal Fatigue program.
- 6.3 Set High Time and Low Time to 1 second.
- 6.4 Run program.
- 6.5 Set High Current to 1A and Low Current to 1A.
- 6.6 Set Steady Load to 0.
- 6.7 Switch Motor On,
- 6.8 After a stable cycle pattern has been reached, Switch Save on.
- 6.9 Select save data for every cycle. The program will save data from the beginning of the next cycle to the end of the cycle after the Save button is deselected.
- 6.10 Set the temperature control panel to 1000 °C for high and 1000 °C for low. Set the High and Low Inc. to 1, and Switch ON.
- 6.11 When the reference sample temperature has stabilised at 1000 °C, Switch the temperature control OFF, and turn off the power supply.
- 6.12 When the reference sample has cooled to room temperature, deselect SAVE, and switch the VI power button OFF.
- 6.13 Print out plot of resistivity/temperature and date.
- 6.14 Using Microsoft Excel, compare resistivity/temperature, and temperature/current data with supplied data set in calibration folder.
- 6.15 An example of a typical data set can be found at the end of this section in C10.

7. *Dimensional Measurements*

In order to calculate stresses on testpieces it is necessary to know the testpiece width and thickness. This is done with a calibrated micrometer situated next to the ETMT. Take two sets of measurements before and after testing, at different points along the sample.

The calibration details for the micrometer are kept in the relevant section of the ETMT equipment record and calibration folder.