

Guide to Ramped Miniature Thermomechanical Tests for Accelerated Material Discrimination in Hardmetals

Conventional thermomechanical fatigue tests are usually performed under conditions of close control of strain amplitude, temperature cycle, loading rates and frequency, mainly for the purposes of developing data for design. However, it is also helpful to have a rapid assessment of the likely response to specific thermomechanical environments, particularly when new materials are being developed, or when material is in short supply or when material production processes have been changed.

For this purpose a miniature test system, using DC current heating, has been developed at the National Physical Laboratory. This been evaluated for its ability to discriminate between different materials under these general circumstances but with conditions of ramping load or temperature to accelerate material response and failure characteristics. This Measurement Note provides a guide to the test methodology adopted for hardmetals.

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1. INTRODUCTION

The purpose of thermomechanical fatigue (TMF) studies is twofold. First and foremost, to obtain engineering relationships and mathematical models for macroscopic material behaviour, allowing the design, evaluation and validation of engineering components. Second, to gain deeper understanding of deformation, damage initiation and growth as influenced by microstructure. Materials scientists conducting basic research are primarily interested in the second goal, while the first is pursued by engineers and designers who integrate this basic information and the experimental data to develop structural models for application in component design, as well as for the validation of the design.

Conventional TMF testing is aimed at simulating the thermomechanical loading history of critical volume elements in components in a laboratory test set-up. This is achieved by exposing the test specimen synchronously to a temperature cycle and to a mechanical strain cycle, applied at predefined rates between given temperature and strain limits. The phase angle between the temperature and strain cycles is chosen to represent the condition in a given volume element of the component. Spatial uniformity of the temperature and mechanical strain (that both change with time) over the volume of the test specimen's gauge length is critical and must be maintained at all times. Continuous measurement of the corresponding stress provides the unique relationship between stress, strain and temperature that controls the behaviour of the material in the selected volume element of the component.

There are several reasons for using miniature tests. For example, advanced materials are often in short supply and the use of small testpieces can accelerate the production of useful data. However, strain measurement becomes more difficult as the testpiece size diminishes. Extensometers are typically used on gauge lengths of 10-25 mm, but smaller testpieces present some problems in the use of conventional extensometers. An innovative method of strain measurement based on

resistance changes was developed in the current work to circumvent the extensometer problem.

Conventional thermomechanical tests usually apply a well defined strain-temperature cycle to the testpiece and measure various parameters as a function of number of deformation cycles, such as the drop in load that occurs as damage develops [1,2]. This procedure generates information vital for lifing components. But, there is another aspect to thermomechanical testing – that of assessing the relative abilities of new materials to resist thermomechanical deformation and that of characterizing the effects of changes in microstructure that occur during service life. In the current work this aspect of thermomechanical deformation has been evaluated using a new miniature system employing various forms of parameter ramping. This approach was chosen to accelerate the acquisition of data. There are many testing possibilities, for example:

- Temperature and stress ramping monotonically
- Either temperature or stress ramping with the other constant
- Temperature and stress cycling, but with increasing amplitude (ramping)
- Either temperature or stress cycling with one constant amplitude and the other with a ramping amplitude.

Aspects of this testing envelope were assessed in the current project on a number of hardmetals in a new thermomechanical test system (the ETMT) where the use of miniature testpieces allows more rapid heating and cooling rates than are possible in conventional systems. The test method involved the use of a miniature electro-mechanical test system in which direct current is used to heat the testpiece.

The primary objective of this work was to evaluate the versatility of the ETMT for performing accelerated ramping tests to assess the thermomechanical deformation resistance of materials for elevated temperature service performance.

Test System

The miniature electro-thermomechanical test system (the ETMT) has been developed at NPL [3-5] to obtain multiproperty data over a range of temperatures. The system uses DC electric current to heat rectangular cross section testpieces. Strain is measured using changes in electrical resistance. Both thermal and mechanical loads can be cycled.

The ETMT consists of an environmental chamber (500 x 250 x 120 mm) with electrical leadthroughs, water cooled grips and inert gas supply for prevention of testpiece oxidation (Fig 1). A computer-controlled DC heating power supply (200A) is used to heat the specimen, with an integral testpiece resistance and thermocouple measurement facility. Testpiece geometries are determined by testpiece resistance. Conducting testpieces, are typically 2 × 1 mm cross section, 40 mm long and heating rates up to 200 °C s⁻¹ are possible, dependent on the thermal characteristics of the testpiece. Cooling rates are determined by the thermal diffusivity of the testpiece and loss of heat to grips. This can typically be between 100 °C s⁻¹ or 10 °C s⁻¹ for good and poor conductors, respectively. The testpiece grips are held at a fixed temperature (room temperature) to provide a constant reference point. This results in a parabolic temperature distribution for testpieces smaller than about 20 mm in length with a central temperature up to 800 °C. Temperature distributions in testpieces heated to much higher temperatures are more uniform in the central 2-4 mm of the testpiece, typically less than ± 5 °C at a central temperature of 1250 °C. The system uses a mechanical loading assembly (± 4 kN maximum), with an in-line drive and a versatile gripping system, a load cell (0.5N resolution) and capacitance displacement transducers (0.4 micrometre resolution). A computer-controlled motor is included for null, mean, ramping or fatigue load capability (in or out-of-phase DC current cycle) or constant displacement tests for stress relaxation or thermal shock experiments. The motor response is set to about 200-1000 N s⁻¹ for nominally square wave fatigue experiments. Uniaxial tests can have variable (step loading) rates, typically

0.1-20 N s⁻¹. Full thermo-mechanical control is possible in load or displacement control modes with options for sinusoidal, triangular, trapezoidal or arbitrary waveforms for both load and temperature, together with any degree of phase lag. Full strain control is not yet possible due to the difficulty of strain measurement on such small testpieces. The system uses customised LabView[®] software to monitor and control tests and temperature cycles by appropriate feedback control (Fig 2). Because the testpiece is held in water cooled grips a non-uniform temperature distribution develops along the testpiece. Measurements and analysis [3-5] have shown that the steady state temperature profile along the testpiece is parabolic at temperatures up to about 800 °C. The indicated temperature of the central 2 mm of the testpiece is approximately within the range:

$$\begin{aligned} &\pm 2\text{ }^{\circ}\text{C at } 200\text{ }^{\circ}\text{C} - 400\text{ }^{\circ}\text{C} \\ &\pm 3\text{ }^{\circ}\text{C at } 400\text{--}1000\text{ }^{\circ}\text{C} \end{aligned}$$

The program software maintains control of temperature even if the testpiece changes in cross section, providing the strain rate is not greater than about 0.01 s⁻¹. For uniaxial tests the loading rate can be set at rates between 0.05 N s⁻¹ to 1000 N s⁻¹. During the test, changes in length are monitored by the capacitance transducers mounted on the grips and results are plotted as load against displacement. However, it must be noted that the displacement contains an element due to the compliance of the rig which is about 0.05 μm N⁻¹.

A further issue is the accuracy of temperature measurement, since thermocouples are made at NPL individually for each test, using 0.1 mm diameter wires of Pt and Pt-13% Rh, that are fusion welded to form a small bead. The bead is then spot welded to the testpiece. Furthermore the voltage is processed by a standard LabView[®] software routine to give a direct reading in °C. A sample of pure Ti was used in order to check the combined ETMT software and thermocouple. Ti has a well defined transformation temperature. Repeat measurements were made and the indicated temperature of the transformation was within 2 °C of the handbook value for pure Ti. The resistance was measured over a 3 mm length in the centre of the testpiece, using spot welded

0.1 mm diameter wires of Pt-13%Rh, where it is known that the temperature is reasonably uniform. A two-colour pyrometer has also been recently installed and can be used in the range 400-1200 °C in situations where it is difficult to spot weld a conventional thermocouple.

Strain Measurement

Because of the difficulties of deconvoluting the load/displacement data from rig compliance and with a temperature distribution, an alternative strain measurement procedure was developed based on the use of resistance measurements. Resistance is measured over the central 2-3 mm of the testpiece, where the temperature distribution is reasonably constant. The resistance in the central 2-3 mm of the testpiece changes significantly during a deformation test due to a change in the cross sectional area. The resistance increases in tension and decreases in compression. The following expression was developed to allow true stress/strain data to be obtained from these changes [3-5].

True strain, ε , is given by

$$\varepsilon = \ln \sqrt{R_t / R_s} \quad (1)$$

where R_s and R_t are the testpiece resistance before and during the test respectively.

Materials, Testpieces and Tests

WC hardmetals (cemented carbides)

Six grades of hardmetal (cemented carbides) were tested, based on WC as the hard phase. Some grades contained an alloy binder phase that was different to the Co that is conventionally present. The material codes, nominal wt% composition and hardnesses, HV30, of the six hardmetals were:

cwn8	:	1300	:	92 WC; 8 Ni
shmcn5	:	1950	:	95 WC; 5 Co/Ni
cwfnc20	:	1250	:	80 WC; 20 Fe/Ni/Co
k801	:	1065	:	WC; 6 Ni
n15	:	1250	:	WC; 15 Co
uc9	:	995	:	WC; 10 Co

The arithmetic mean linear grain size, μm , for each sample was

cwn8	:	0.85 (medium)	k801	:	2.70 (mixed)
shmcn5	:	0.37 (fine)	n15	:	0.49 (fine)
cwfnc20	:	-	uc9	:	4.97 (coarse)

The hardmetals were provided by members of the research group of the British Hardmetal Association.

Testpieces were wire electrodischarge machined (EDM) from larger blocks of material to a nominal, rectangular, 1 mm x 2 mm x 40 mm size. These were then lightly ground using a diamond wheel to remove EDM residues and ensure parallel dimensions (to ± 0.01 mm). Thermocouples and resistance leads were spot welded to the centre of the testpiece using as low discharge voltage as possible to minimise the likelihood of damage.

The following test types were examined:

- Zero load, ramp temperature
- Constant load, ramp temperature
- Constant temperature, step ramp load
- Cyclic temperature, ramp fatigue load.

A primary objective for each type of ramping test was to generate information on the dependence of thermomechanical strain rate on stress and temperature in ramping tests. The usual convention in high temperature deformation studies of plotting strain rate against stress or temperature was followed. For example, the most common expression relating deformation rate to applied stress is given by

$$\dot{\varepsilon} = A\sigma^n \exp [-Q/RT] \quad (2)$$

where $\dot{\varepsilon}$ is the strain rate, Q is an activation energy, T is the temperature, σ is the stress and A and n are constants. The data generated from the ramping tests was therefore analysed with this expression in mind.

Results and Discussion

An initial temperature ramping experiment was performed for each material at a heating rate of 2 °C/s while maintaining zero load on the

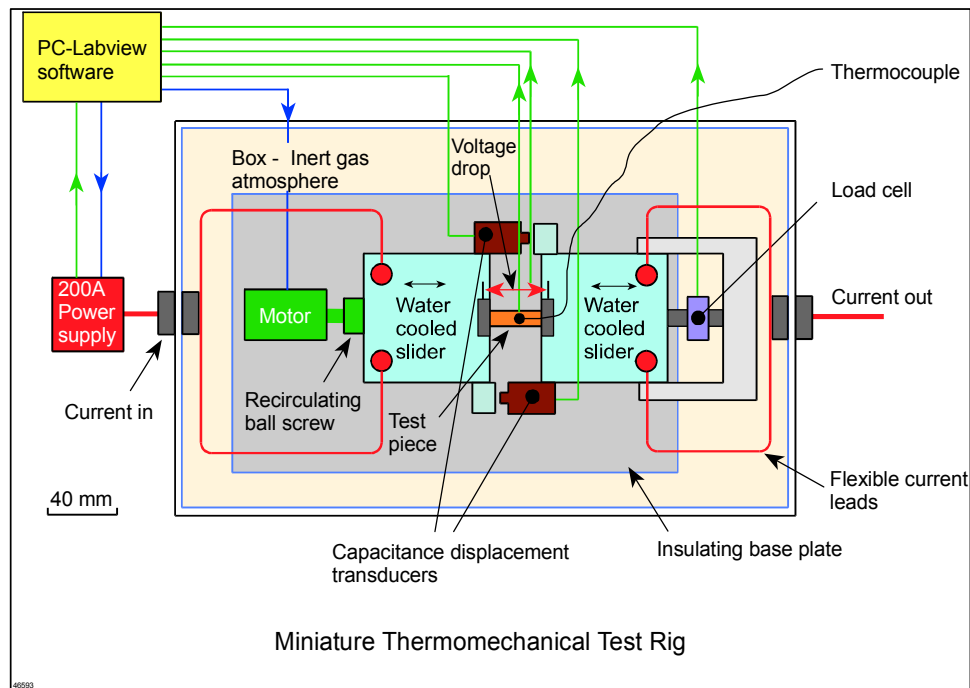


Fig 1 Schematic diagram of ETMT test system.

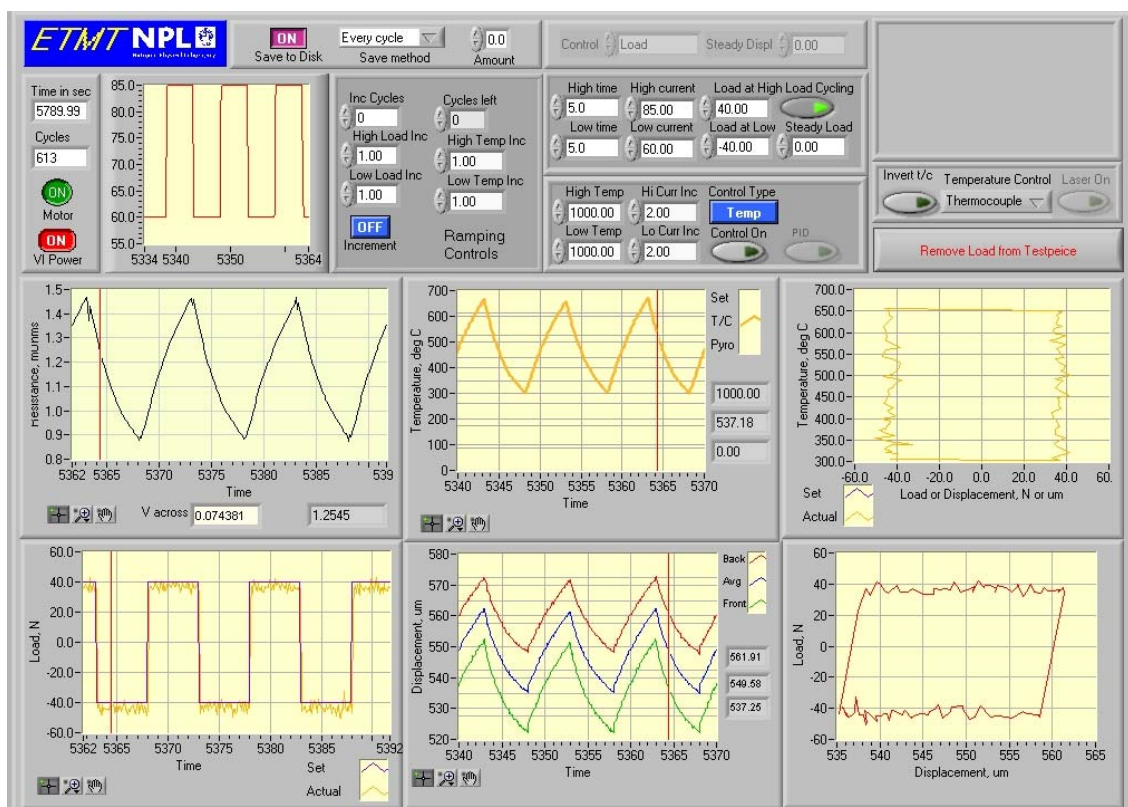


Fig 2 ETMT front panel control options

testpiece. This experiment generates data on the thermal expansion characteristics of the material and the dependence of resistance (resistivity) on temperature. Typical data for four of the materials are shown in Figs 3 and 4. The phase transformation associated with the Fe/Co/Ni material can be seen at about 600 °C in both the expansion and resistivity plots.

In the second type of ramping experiment the same heating rate was applied (about 2 °C/s) while maintaining a constant load on the testpiece. Initially the sample expands as in the above case, but then at higher temperatures the hardmetal begins to deform and the displacement/temperature curve deviates from that measured at zero load. Typical data are shown in Fig 5.

As each material deforms the resistance increases due to plastic deformation, over and above the increase due to increasing temperature. The resistivity increase is converted to strain to generate data on the variation of strain with temperature. Typical data for the hardmetals is shown in Fig 6. Because the same temperature ramping rate has been used for each test a strain rate can thus be determined from this data. Typical values are shown plotted in Fig 7 following the expression (2) above.

A gradient equivalent to an activation energy of 300 kJ mole⁻¹ is shown superimposed on the data. The results show good discrimination between the different hardmetals.

The third type of ramping test consisted of a step load ramp at a constant temperature of 850 °C. A schematic outline of the test is shown in Fig 8. The load was increased by a fixed value every 2500s. Different starting values were chosen for each material dependent on the results of preliminary tests to measure nominal strength.

The ramping test for the Ni and Co/Ni hardmetals was commenced at 400 N (200 MPa) and there was little deformation before what was essentially brittle fracture (Fig 9). The Fe/Co/Ni material was considerably more ductile and so the test in this case was started at 100 N (50 MPa). This

enabled a log strain rate/stress plot to be calculated (Fig 10) showing the typical linear correlation that is observed in high temperature deformation studies.

In the fourth type of test the temperature was cycled between 350 and 850 °C with a cycle time of 15s. A schematic of the test profile is given in Fig 11. The load was cycled at an R value of -1 (i.e. equal tension to compression) but step increasing the amplitude every 50 cycles. Typical results showing material discrimination are shown in Fig 12.

Summary

A new test system for miniaturised thermomechanical tests has been evaluated for its ability to discriminate between materials in ramping experiments. Tests on a range of materials have demonstrated its versatility. A number of different ramping experiments were explored with the objective of measuring the dependence of deformation strain rate on applied stress. This was successfully achieved for all the materials evaluated. However, it was necessary to use an unconventional method for monitoring strain, using the change in electrical resistance as the testpieces experienced significant plastic deformation. The ramping tests were shown to discriminate particularly well between materials with different binder phase types.

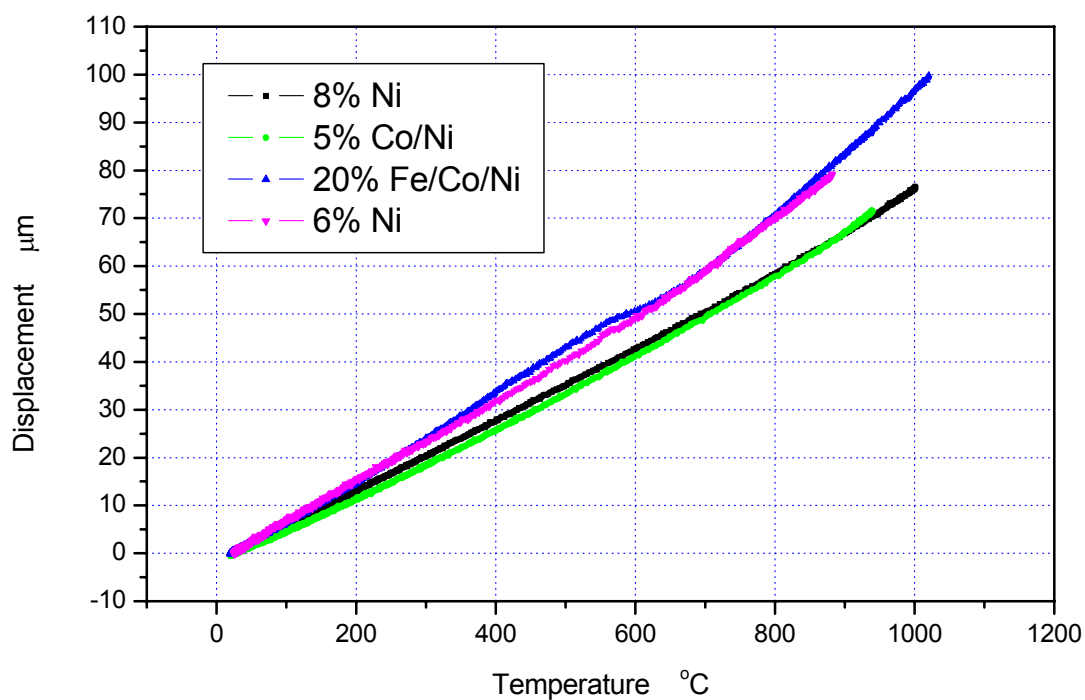


Fig 3 Expansion characteristics of four of the hardmetals.

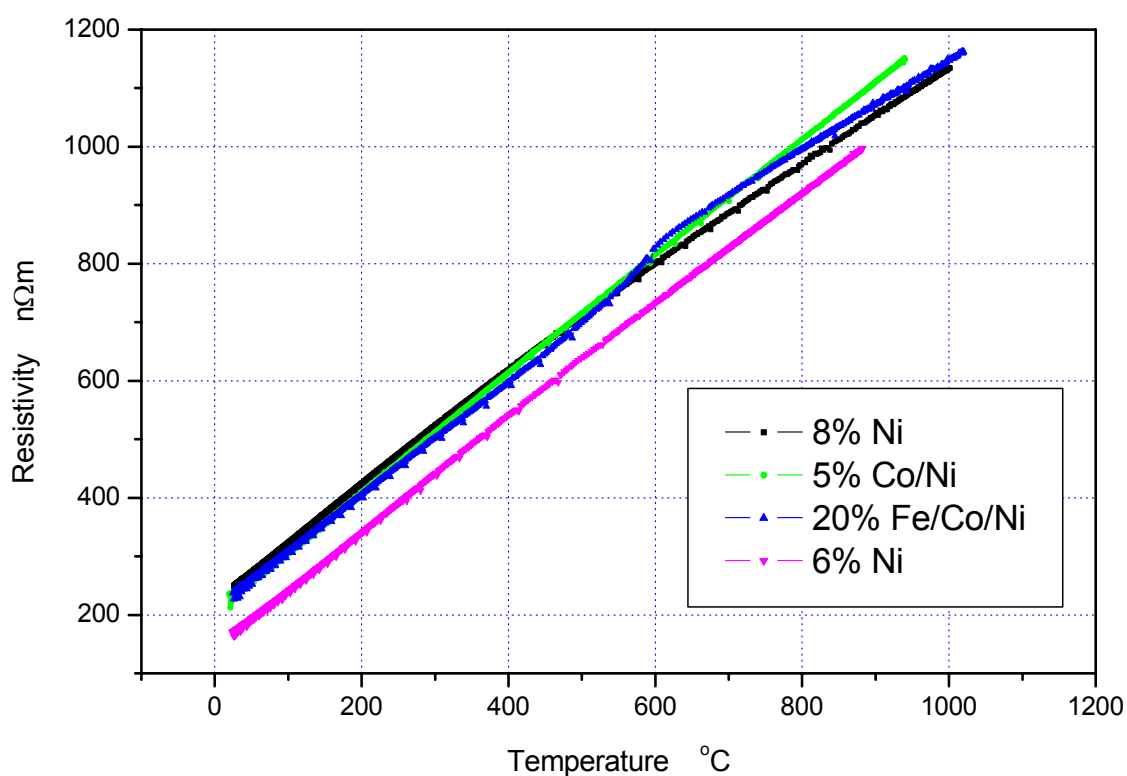


Fig 4 Resistivity temperature dependence of the hardmetals.

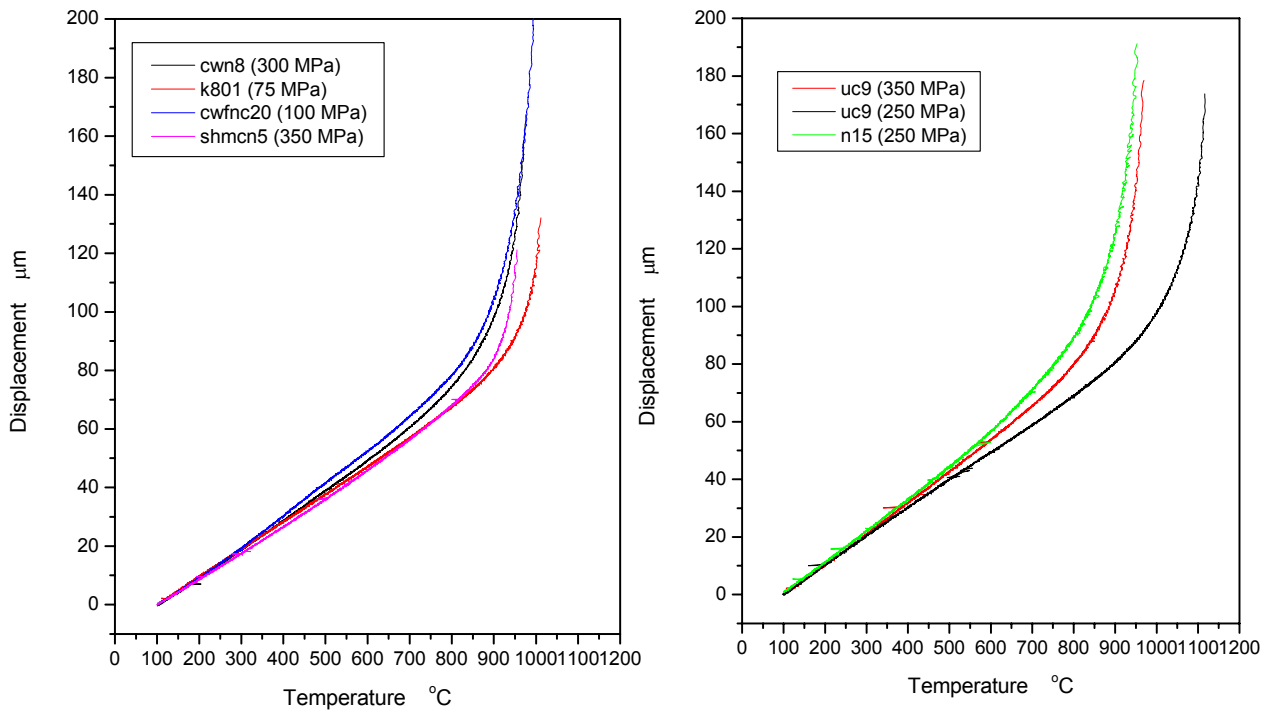


Fig 5 Ramping displacement/temperature plots for selected applied loads.

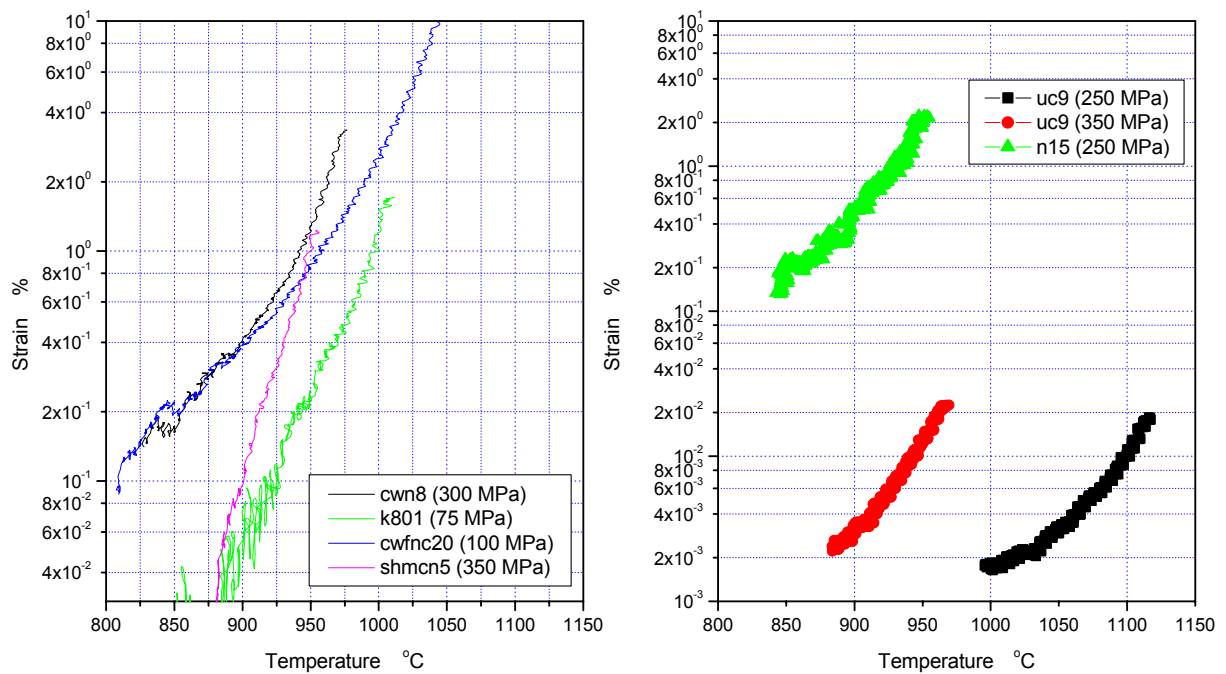


Fig 6 Typical plots of strain against temperature.

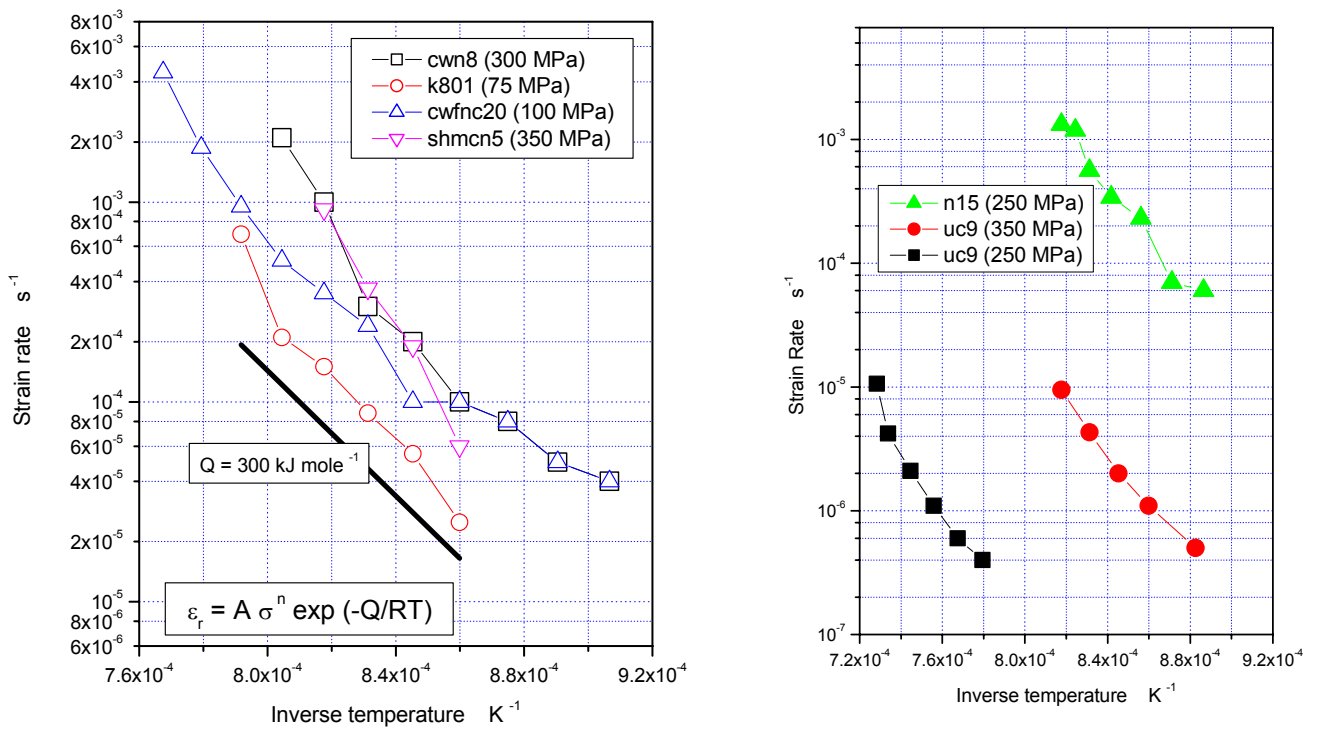


Fig 7 Typical plots of strain rate against temperature.

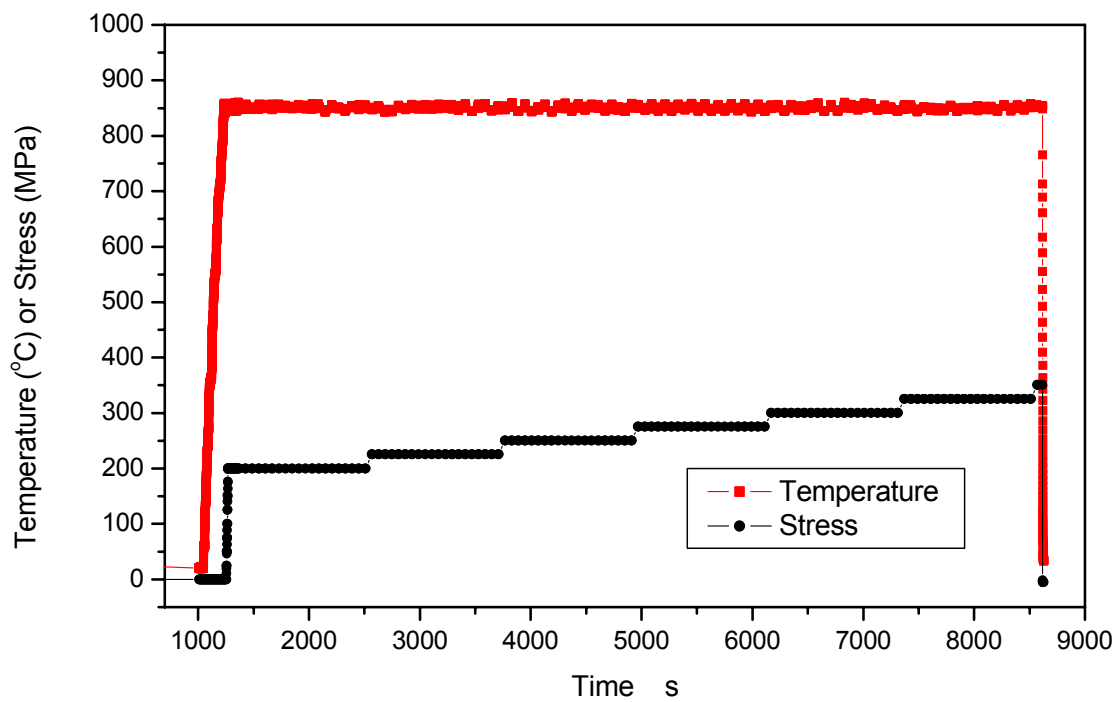


Fig 8 Outline of constant temperature, step load ramp test.

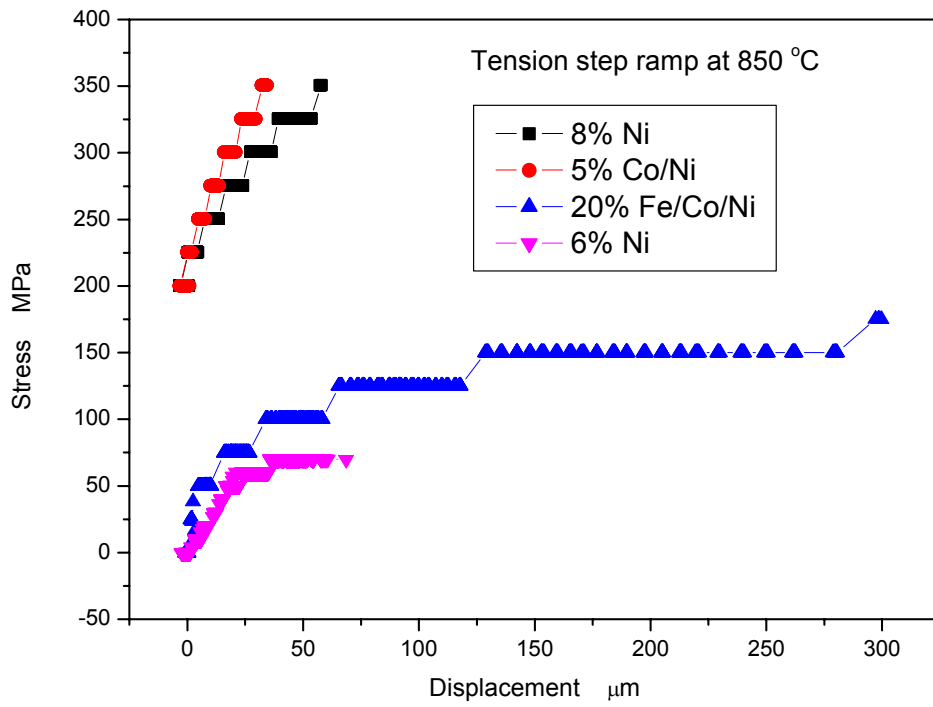


Fig 9 Load/displacement data from step ramp tests at constant temperature.

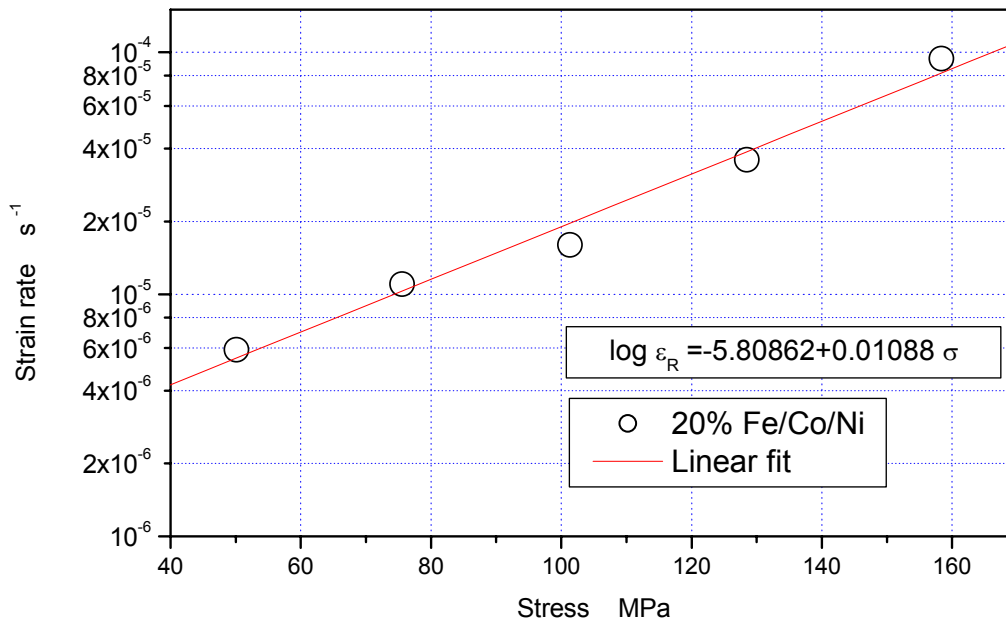


Fig 10 Strain rate dependence on stress from step ramp tests at constant temperatures.

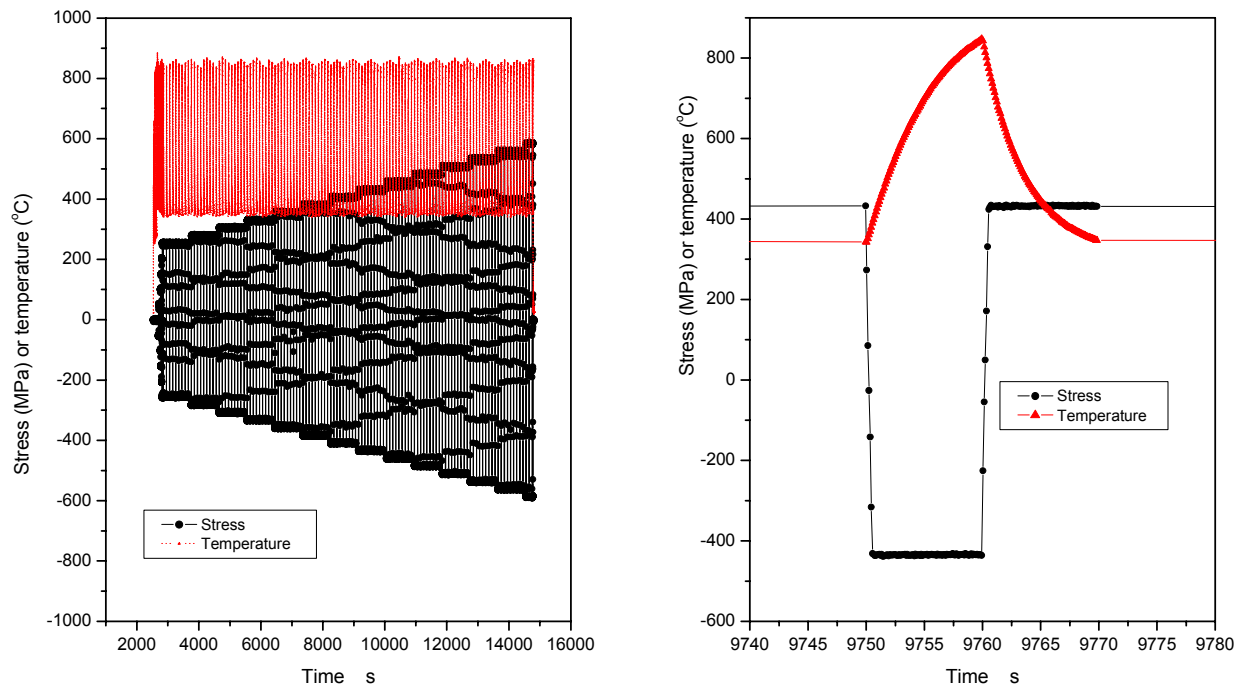


Fig 11 Outline of cyclic temperature, ramp load cycling tests.

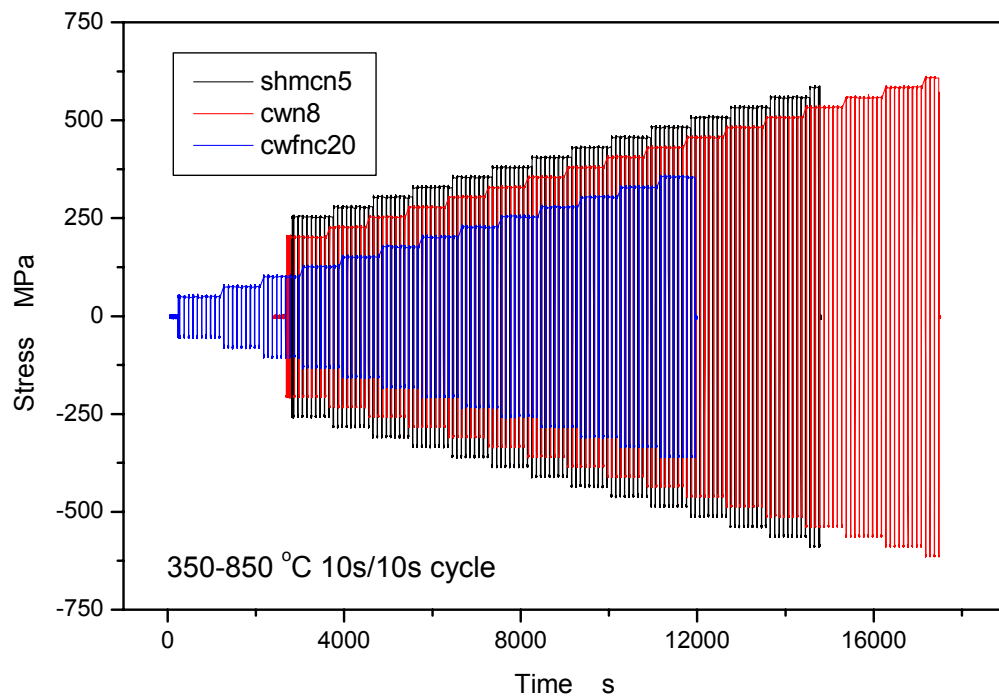


Fig 12 Typical step cycling load and temperature results.

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