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Guide to Ramped Miniature Thermomechanical Tests for Accelerated Material Evaluation in Powder Metallurgy Tool Steels

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 has 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 PM tool steels.

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INTRODUCTION

The purpose of thermomechanical fatigue (TMF) studies is twofold. First and foremost, it is to obtain engineering relationships and mathematical models for macroscopic material behaviour, allowing the design, evaluation and validation of engineering components. Second, it is 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:

± 2 °C at 200 °C - 400 °C
± 3 °C at 400-1000 °C

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 with 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^{o}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

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

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

Materials, Testpieces and Tests

PM high speed steels

Five grades of PM high speed steel were tested, based on the ASP30 gas atomised and hot isostatically pressed grade. Some grades were used as HIPed, others were tested after being subjected to post-HIP hot working. Two of the grades were tested in the HIPed condition, but also had small quantities of ceramic admixed prior to HIPing. All samples were austenitised, quenched and triple tempered to 875HV30 prior to testing. The material codes processing and additives were as follows:

LAH: HIPed
LAJ: admixed ZrO_2; HIPed
LAL:admixed ZrO_2 and TiB_2; HIPed
LEJ: HIPed + hot worked (longitudinal)
LEL HIPed + hot worked (transverse)

The high speed steels were provided by John Saveker Steels Ltd., Four Oaks, Sutton Coldfield, West Midlands. The first three batches were produced in-house, the latter two were purchased from Erasteel UK Ltd, Darnall, Sheffield.

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
- Fatigue load, ramp fatigue temperature
- Constant 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

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

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 ^{o}C/s while maintaining zero load on the 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 unusual behaviour of the LAL material (an experimental grade) is thought to be due to the tempering behaviour of areas in the microstructure peripheral to TiB_2/ZrO_2 clusters (these being denuded of alloying elements during HIPing; the HIPing temperature being approximately that of the Fe-B eutectic).

In the second type of ramping experiment the sample ramping temperature fatigue with in-phase mechanical fatigue (0-250MPa); the test regime is outlined in Figs 5a-5b. Initially the sample expands as in the above case, but then at higher temperatures the tool steel begins to deform and the displacement/ temperature, and displacement/ time curves deviate from approximately linear behaviour observed in the initial part of the test. Typical data are shown in Figs 6a-6b.

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 strain data for the high speed steels in the temperature ramping tests are shown in Fig 7. 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 8 following the expression (2) above.

The third type of ramping test consisted of a ramp fatigue test at a constant temperature of 650°C. A schematic outline of the test is shown in Fig 9. The initial load cycle is 0-250MPa. With the peak load (i.e. 250MPa) being increased by 50MPa every 1000 cycles. Two different cycle durations (6s at load followed by 6s at zero load; and 3s at load followed by 3s at zero load, respectively) were used to investigate dependence of cycle duration on fatigue lifetimes at this temperature.

The results (illustrated in Figs 10a-10c) show that in the case of the fatigue tests at a constant 650°C, shorter lifetimes were produced by the higher cycling frequency. In general, under given conditions, material LAH gives the longest fatigue lives, followed by LAJ and LEJ/LEL. The latter portions of the displacement-time plots seem to indicate that LAH fails in a more ductile manner than the other batches. However, fatigue lives of the LAJ material appear to show more repeatability under these conditions and are possibly not as sensitive to frequency as LAH. Strain rate–temperature and strain rate–peak applied fatigue stress relationships (Figs 11a-d) appear however to be largely unaffected by processing route or addition of zirconia. In the case of the strain rate – temperature data (Fig 8), the scatter in the results precludes any meaningful discussion on effects of processing route and/or addition of the ceramic. It can be seen that the constant (650°C) temperature ramp fatigue tests were better in terms of material discriminability than the ramping fatigue temperature fatigue load (i.e. 0-500N) tests.

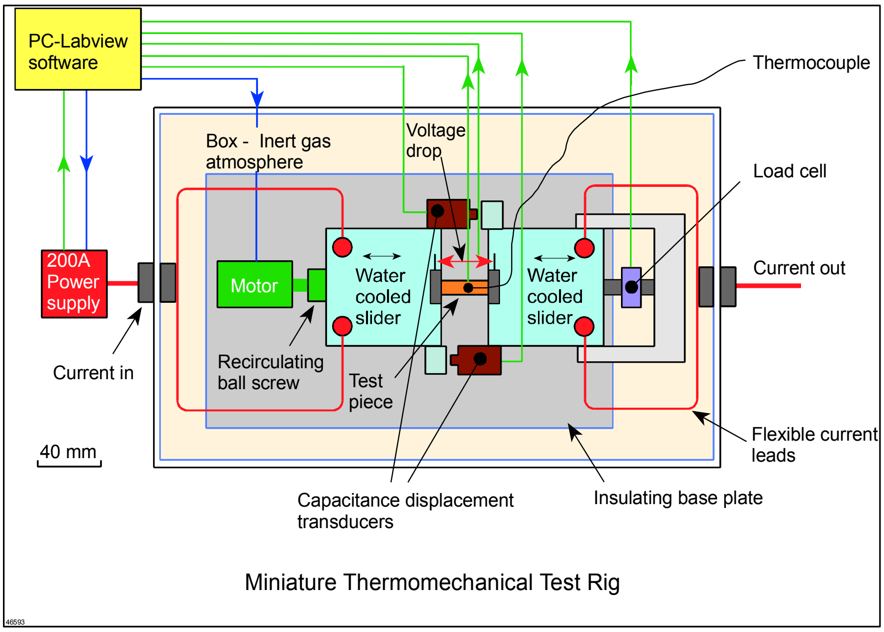

Fig 1 Schematic diagram of ETMT test system.

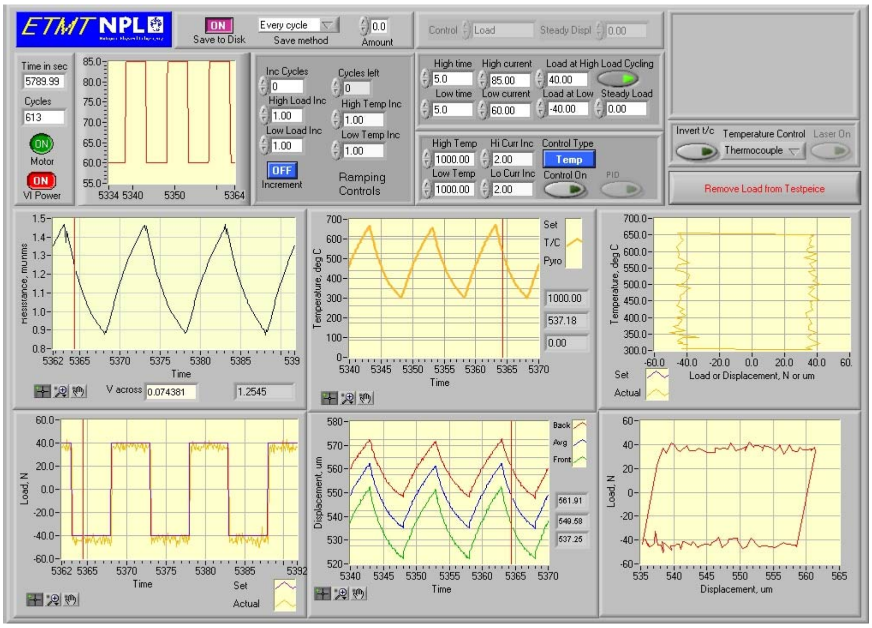

Fig 2 ETMT front panel control options

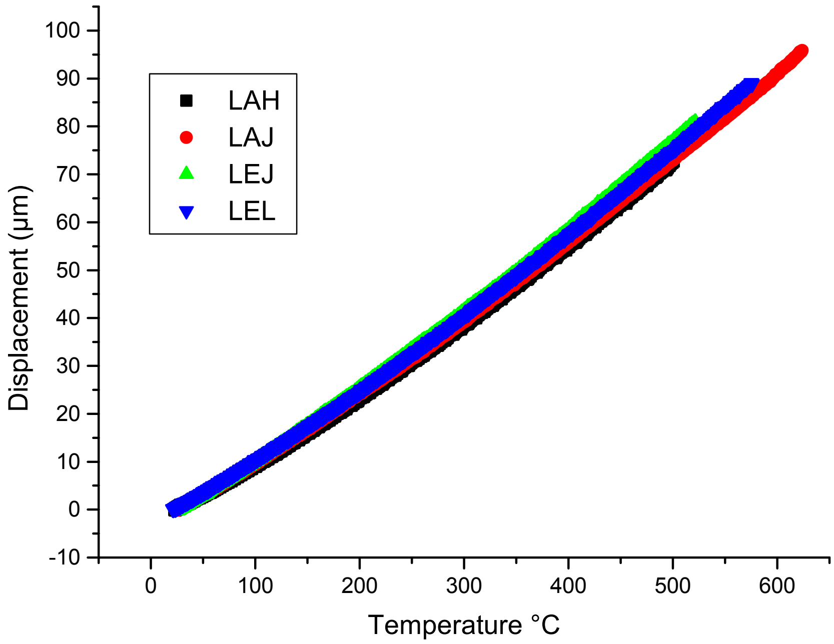

Fig 3 Expansion characteristics of four of the PM high speed steels.

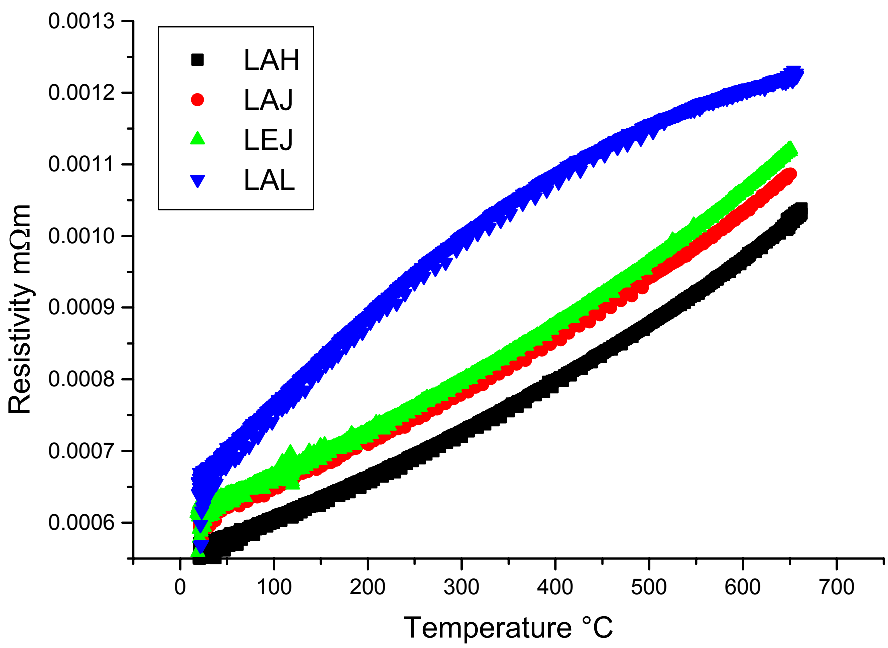

Fig 4 Resistivity temperature dependence of four of the PM High speed steels (LAL contains regions depleted in alloying elements; the annealing of these regions may explain the different resistivity-temperature behaviour in this material).

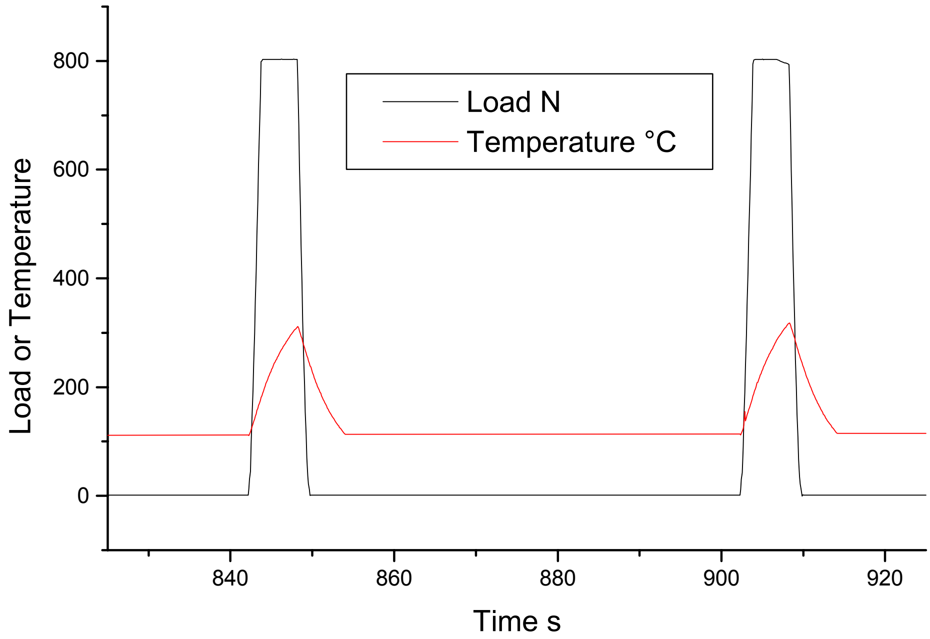

Fig 5a Outline of ramp cyclic temperature, cyclic load cycling tests.

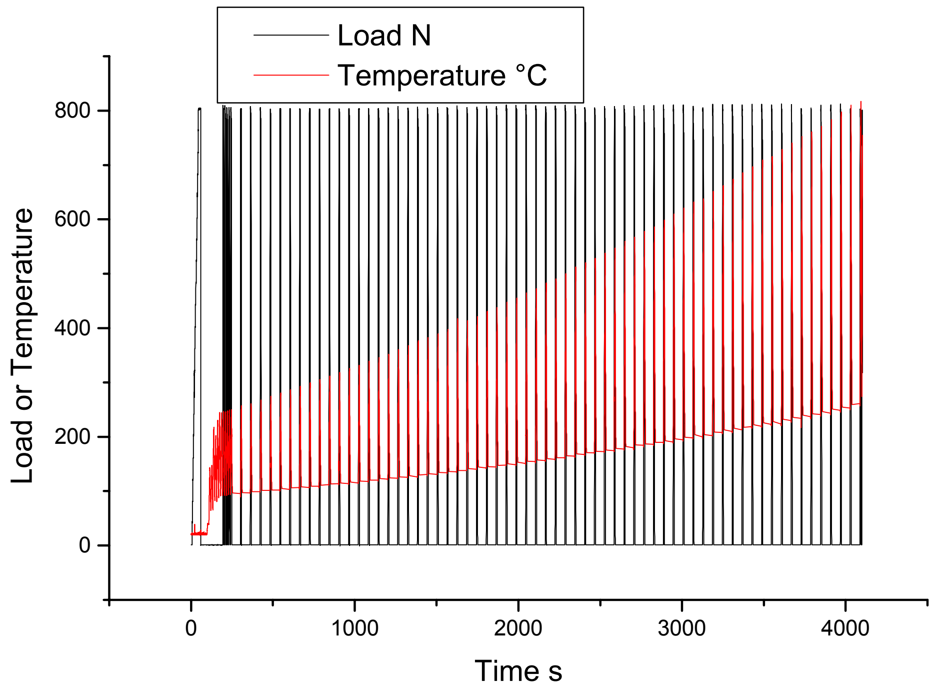

Fig 5b Outline of ramp cyclic temperature, cyclic load cycling tests.

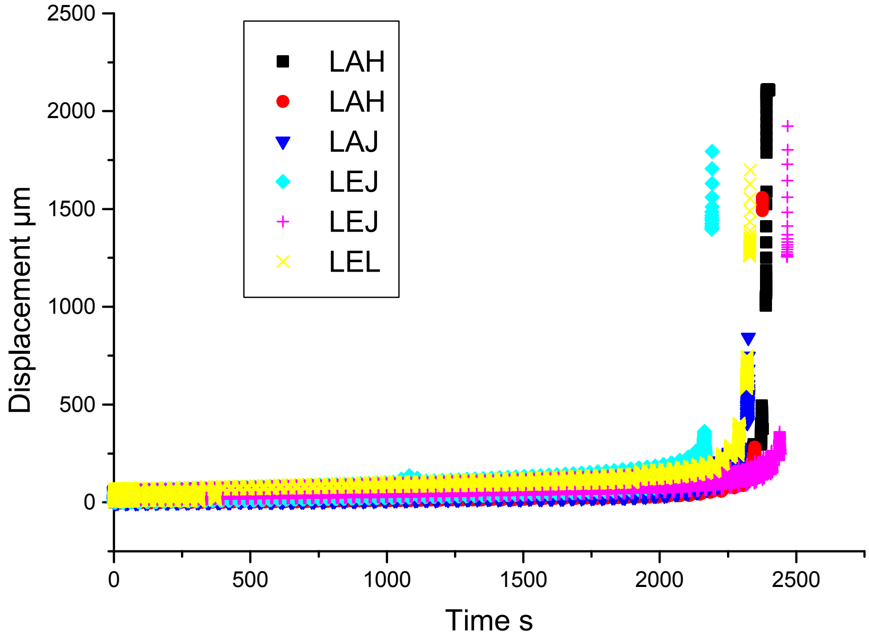

Fig 6a : Displacement/time plots for ramping fatigue/ in-phase fatigue load tests.

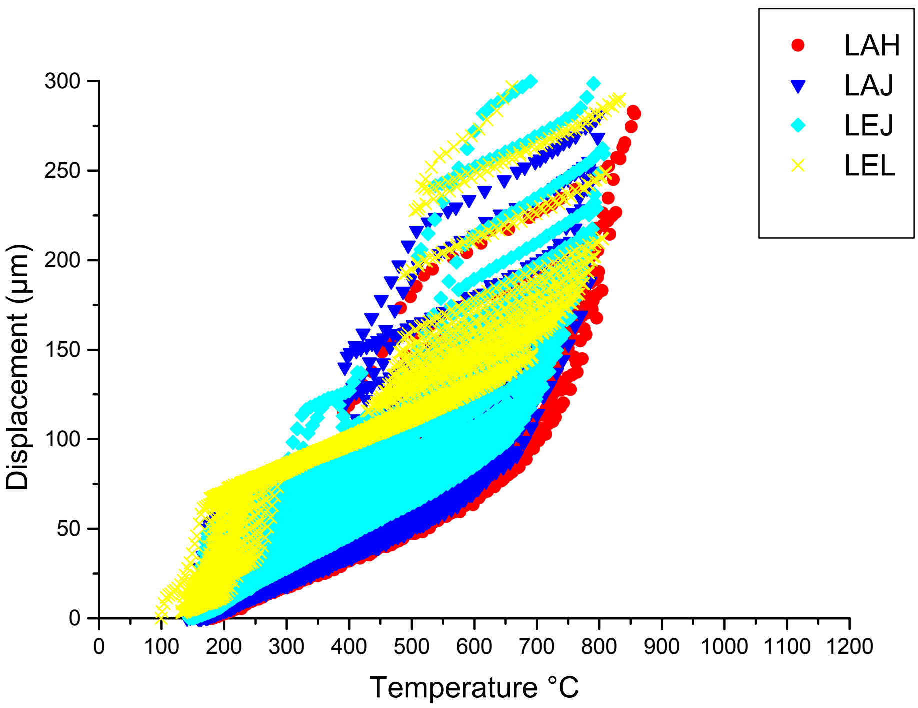

Fig 6b: Displacement/temperature plots for ramping fatigue/ in-phase fatigue load tests.

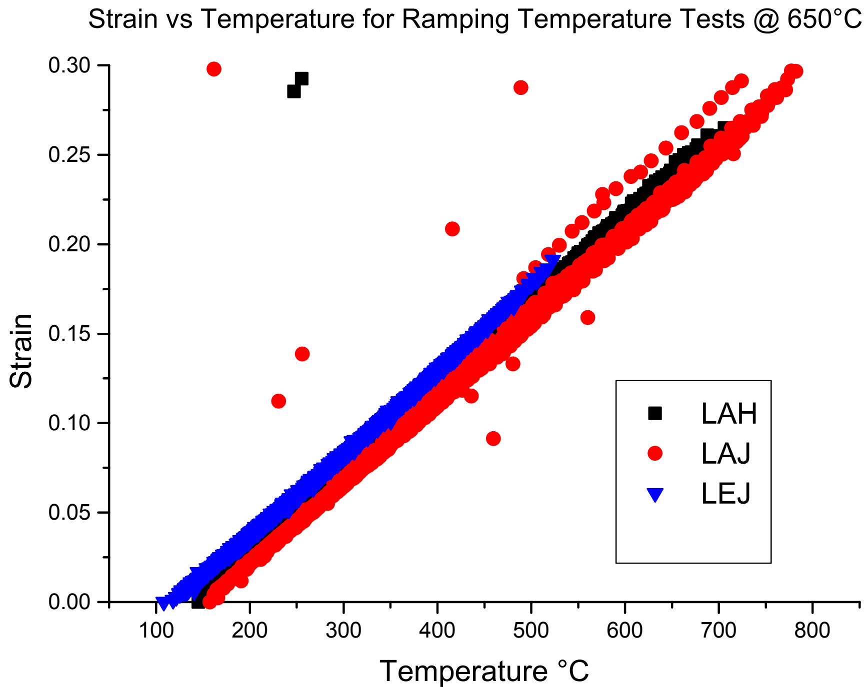

Fig 7: Typical plots of strain against temperature from ramping fatigue temperature/ in-phase fatigue load tests (stray data points are in fact noise).

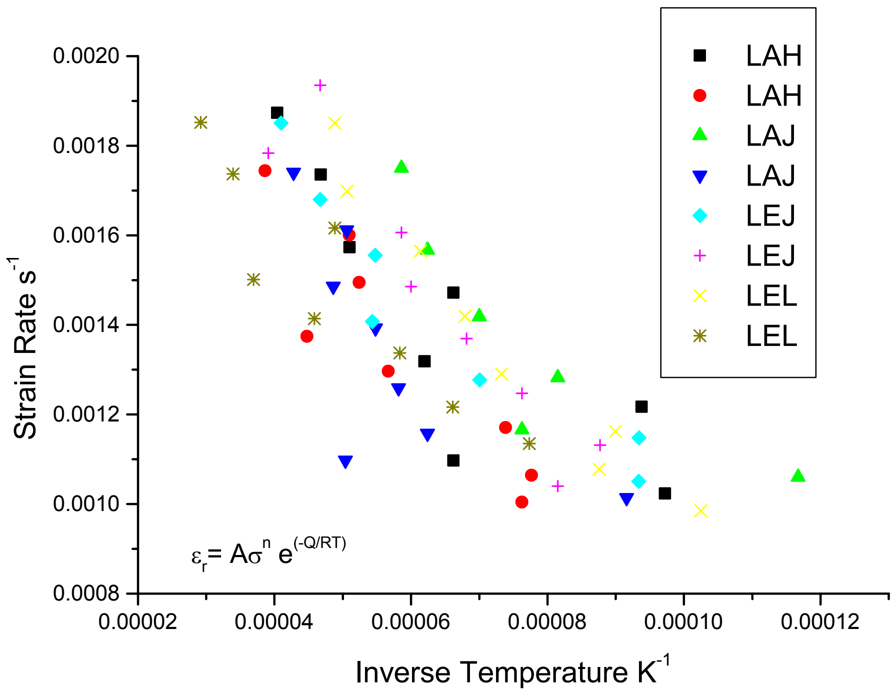

Fig 8: Plots of strain rate against temperature from ramping fatigue temperature/ in-phase fatigue load tests. Strain rates were computed from strains measured at the peak applied load (500N).

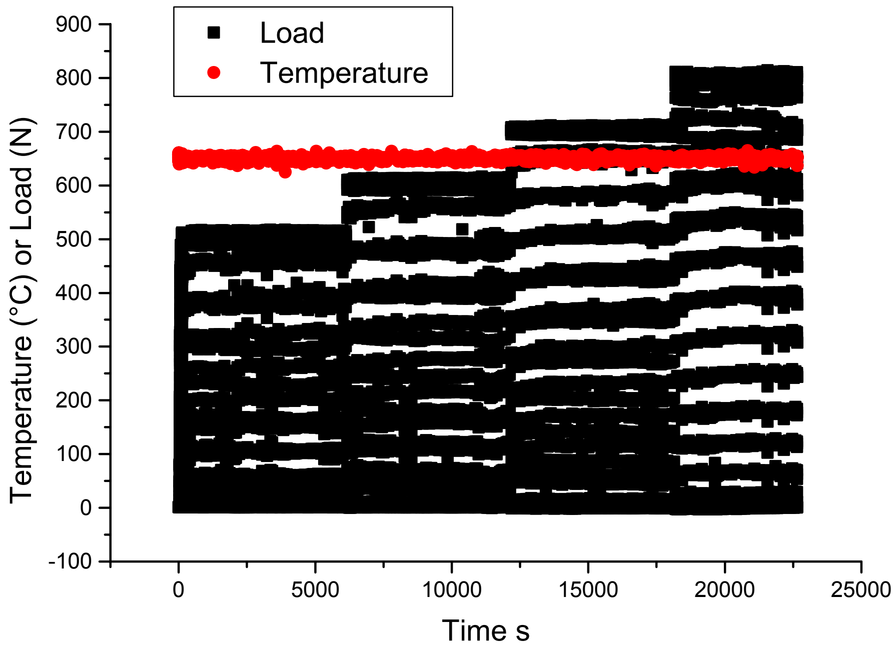

Fig 9: Outline of constant temperature (650°C), step ramp (i.e. load) fatigue tests.

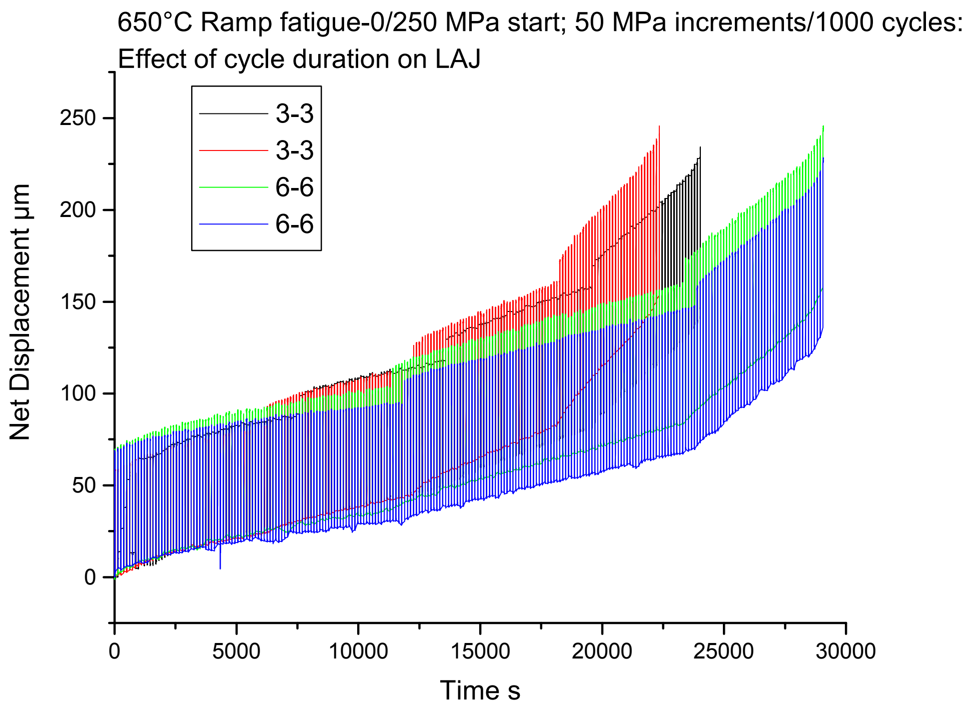

Fig 10a: Effect of cycle duration on LAJ (0/250MPa start) at 650°C.

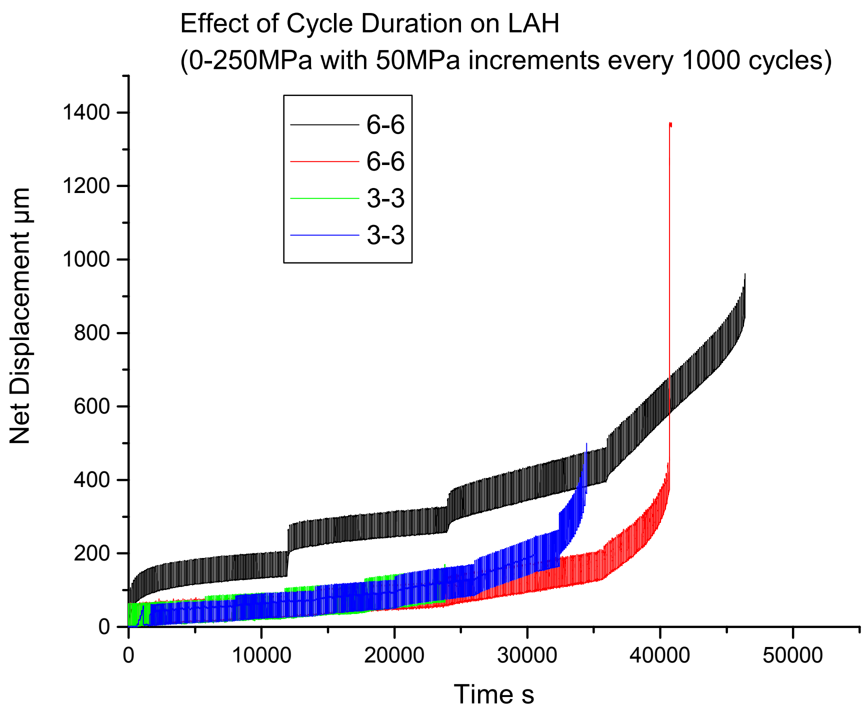

Fig 10b: Effect of cycle duration on LAH (0/250MPa start) at 650°C.

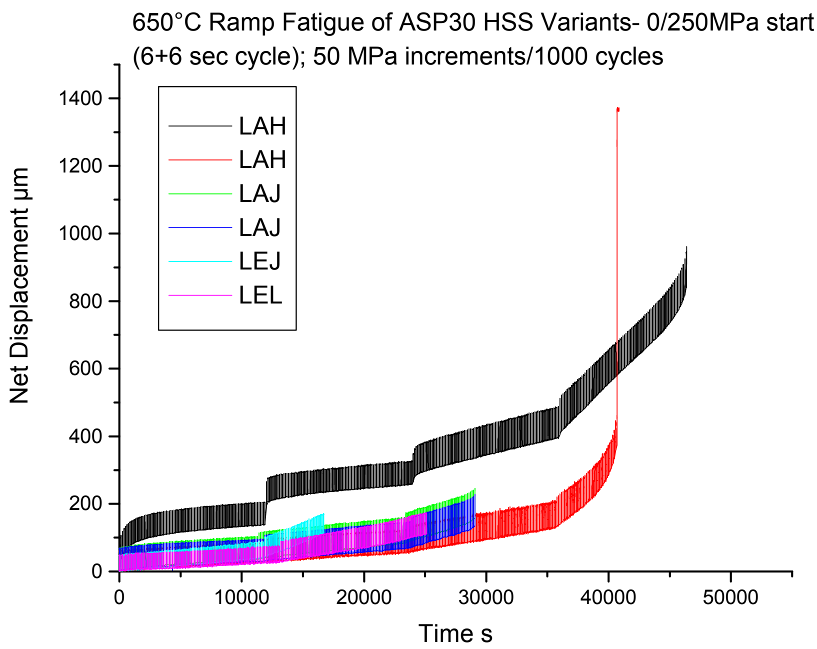

Fig 10c: Effect of composition/processing route on lifetimes when subjected to the 6-6s cycle regime.

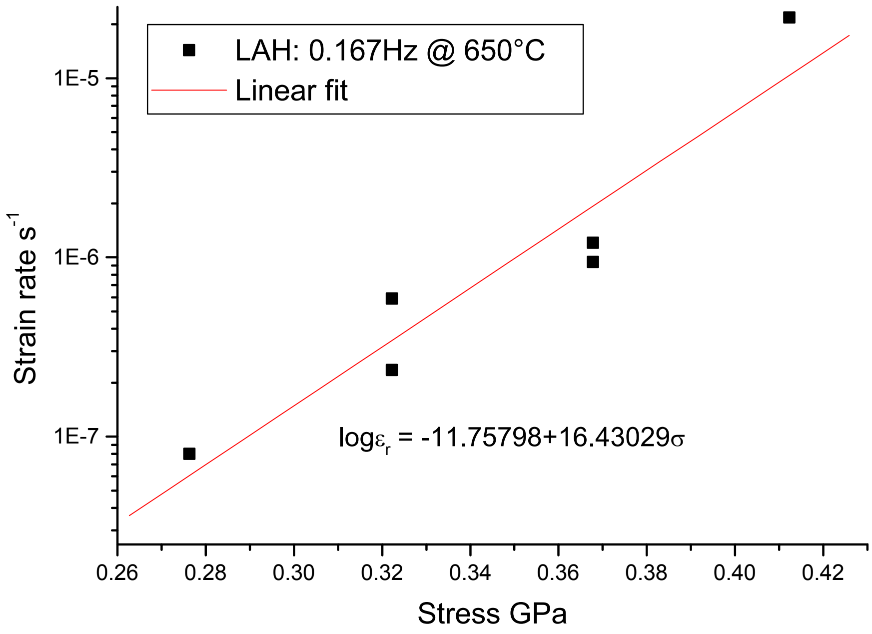

Fig 11a: Strain rate dependence on stress from step ramp test at constant temperature; LAH fatigued at 650°C (Initial 0-250MPa/0.167Hz waveform). Strain rates computed from strains measured at peak loads.

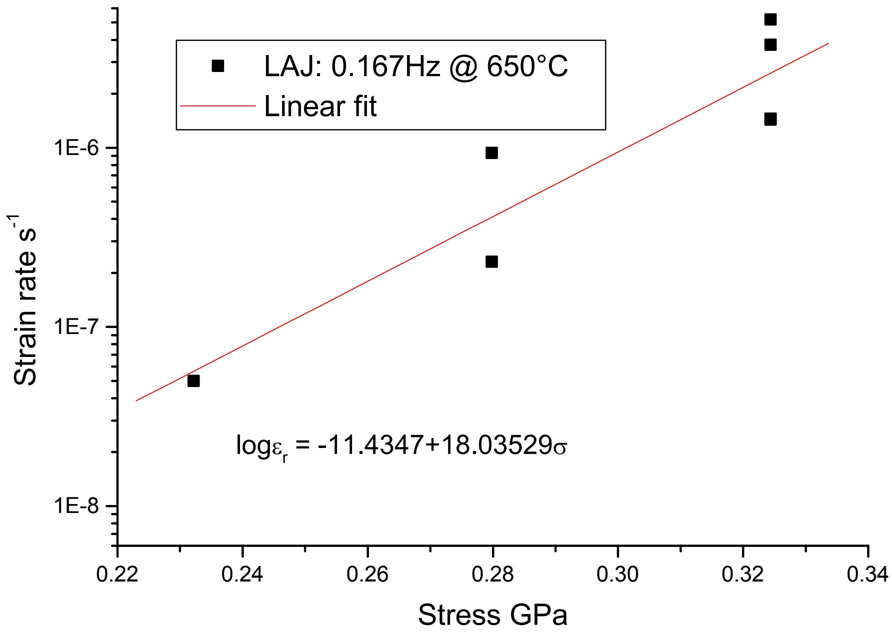

Fig 11b: Strain rate dependence on stress from step ramp test at constant temperature; LAJ fatigued at 650°C (Initial 0-250MPa/0.167Hz waveform). Strain rates computed from strains measured at peak loads.

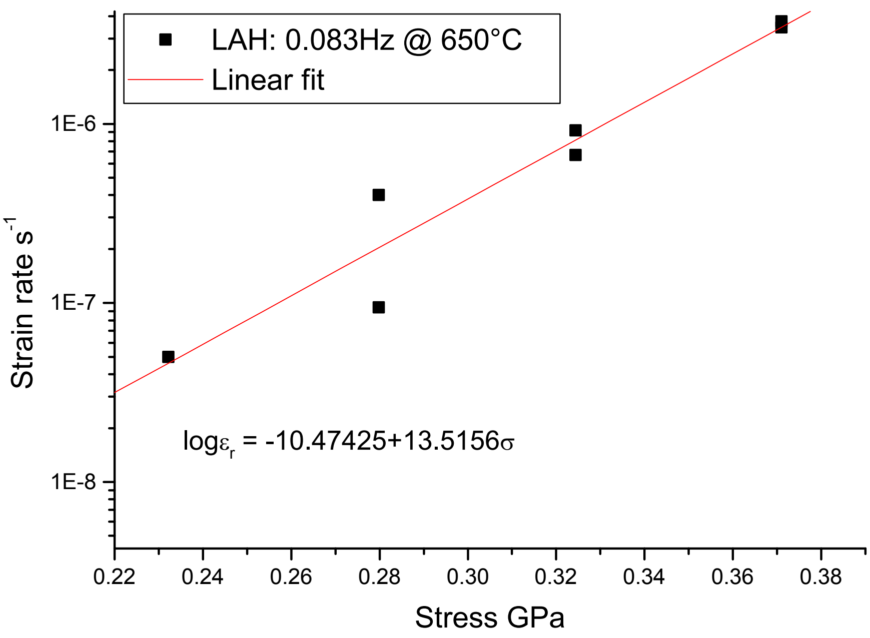

Fig 11c: Strain rate dependence on stress from step ramp test at constant temperature; LAH fatigued at 650°C (Initial 0-250MPa/0.083Hz waveform). Strain rates computed from strains measured at peak loads.

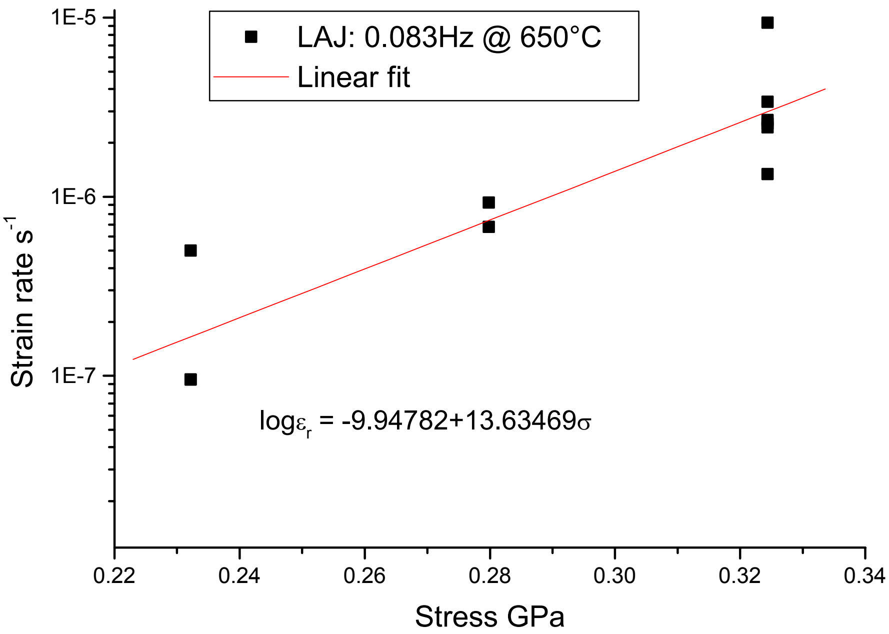

Fig 11d: Strain rate dependence on stress from step ramp test at constant temperature; LAJ fatigued at 650°C (Initial 0-250MPa/0.083Hz waveform). Strain rates computed from strains measured at peak loads.

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