CHAPTER THREE

3.0 EXPERIMENTAL PROCEDURES

3.1 Introduction

Experiments were designed to investigate the following mechanisms:

- Deformation and softening kinetics.
- Variation of activation energy with ageing
- Effect of creep on softening
- Comparison of tensile and creep data

Data from these tests were used to investigate different deformation mechanisms and to determine relevant material parameters. Some key equipment was used in the laboratory during the course of this works; namely, a Denison creep machine, automated tensile test machine and a furnace.

3.1.1 *Materials*

The material used was low alloy steel, 709M40, with chemical analysis shown in table 3.1. The material was machined into threaded end specimens having a gauge of 50mm and a diameter of 5mm, after which it was heat treated to the T-condition (diagram 3.1).

Table 3.1: Chemical Analysis of 709M40 steel by wt%

C	Si	Mn	Cr	Mo	Fe
0.39	0.23	0.77	0.96	0.28	Bal.

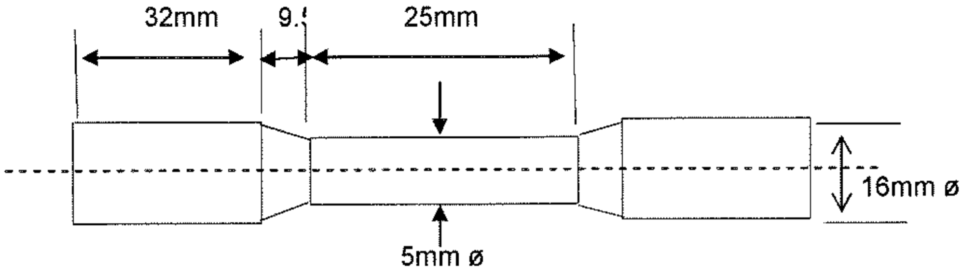

Figure 3.1: *Schematic diagram of test specimen*

3.2 Tensile Tests

3.2.1 Determination of Deformation Kinetics

Tensile testing was carried out at several temperatures and a number of strain rates, the temperature range being 17°C to 600°C. Tests were carried out at constant cross-head speed that produced strain rate at yield in the range 10^{-3} to 10^{-7} s^{-1} when allowance had been made for machine stiffness and temperature dependent module effects.

The purpose of these tests was to determine the deformation kinetics and relevant parameters. Stress related materials constant, $\hat{\sigma}$, the activation volume, *v,* and stress exponent, *n*. were determined. Ten sets of tensile tests were carried out at strain rates ranging from 10^{-1} to 10^{-7} 1/s. Temperature ranged from 17°C to 604°C.

3.2.2 Static Recovery Test

To quantify the ageing process a number of specimens were kept in a furnace for periods of up to two weeks at several temperatures from 560°C to 640°C. Tensile tests were carried out on these samples corresponding to a strain rate of 1.5 x 10^{-4} s^{-1} at yield. Data from these tests was used to investigate the softening mechanism.

3.2.3 Effect of Ageing on Activation Energy

Specimen aged at 603^{o}C for different times and tensile tested at 250 ^{o}C at strain rate of 0.001/s..

Specimen Code	Aging Temperature (^{o}K)	Aging time(hours)
A24t250b A38t250b	876	24 48

Table 3.2: *Test Data to determine the effect on activation energy*

3.3 Automated Tensile Machine

Tensile tests where carried out using a computer integrated Lloyd machine as shown in figure 3.2. The tensile materials testing system combines high performance, flexibility and exceptional ease of use. It is ideal for testing applications with capacities up to 20kN (4500lbf) in tension and direct compression.

The Lloyd machine features a twin column, twin lead screw frame and incorporates a crosshead guidance system to prevent side loading of the sample under test. The grips have internal threads to fit the two cross heads (the lower one being stationary and the upper head being moveable). A high-resolution encoder is used to measure sample extension and also to provide high accuracy speed control.

The Lloyd machine features a multi-position control console with membrane multi-function keypad and easy to read LCD display. Load and extension are displayed on highly legible LCD display, selected in variety of units. The machine may be programmed to perform up to ten different tests and the statistics for each test are automatically calculated.

Tests may also be performed using state of the art control and analysis software windowsR via the integral RS232 interface allowing full PC integration. A special two grip system was used to fit and hold the test specimen in a vertical position and in alignment with the cross heads of the Lloyd machine. The grips have internal threads to fit the two cross heads (the lower one being stationary and the upper head being mobile) and to ensure that the test specimen is subjected to tension – compression loading with little bending during the test. A range of highly accurate, interchangeable load cells and extensometers are available for tension, compression and cycling through zero force measurements.

Figure 3.2 Lloyd machine with auxiliary components including a desktop computer and a control panel

3.4 Tensile Data Manipulation

The tensile data was obtained automatically with the aid of the integrated desk top computer using the appropriate programmes. Data was then analysed using both kinetic and evolution equations.

Programmes
MATLAB

MATLAB, a high-performance language for technical computing was used for modelling. Other typical uses include math and computation, algorithm development, data acquisition, simulation, and prototyping, data analysis, exploration, and visualization Scientific and engineering graphics, application development, including graphical user interface building, integrates computation visualization, and programming in an easy-to-use environment where problems and solutions are expressed in familiar mathematical notation. MATLAB is an interactive system whose basic data element is an array that does not require dimensioning thus allowing one to solve many technical computing problems, especially those with matrix and vector formulations, in a fraction of the time it would take to write a program in a scalar no interactive language such as C or Fortran.

RCONTROL for Windows

RCONTROL for windows data analysis software is an extremely powerful extension to Lloyd instruments materials testing system and has been designed to meet the needs of quality assurance departments, production control and the research engineer.

The software has been designed to work under Microsoft Windows Version 3 XX and uses the capabilities of windows to enhance its functions. The software acquires, records, analyses, stores and prints test data with little skill or manual effort. The software has been designed to allow a variety of specialized testing procedures to be undertaken and is multi-stage control programme which can be configured by the user to meet a particular testing requirement. It can perform both standard load tests and advanced functions such as cycling, constant load and constant load rates. This makes it particularly suitable for use by research engineers. The test may be by a constant cross head speed or at a speed determined by the software to achieve the required testing condition.

The graph can comprise standard load/extension data or show more specialized information including load, extension or time. The software automatically calculates the stress, area under the curve, load at break etc. in seconds for most commonly required test results. It can also be tailored by entering formulae and logical expressions to determine the test profile and results that are provided in the test results.

3.5 Creep Tests

Several sets of creep tests were carried out under constant tensile force conditions in the temperature range 560°C to 640°C. This was carried out to determine dynamic recovery.

From the true strain versus time in seconds plot, the minimum creep rate was established for each plot. This, together with the true stress, was correlated with the tensile data for comparison.

3.5.1 *Effect of Creep on Softening*

A specified number of tensile yield stress measurements were also performed on interrupted creep specimens to assess any accelerated recovery effects due to the presence of an applied stress and to determine the softening effect of creep.

Four specimens aged at the same temperature of 603°C where tensile tested with two suffering creep. These tests were conducted at same load and temperature for varying times. The specified load was 79Mpa at a temperature of 603°C. Tensile specimens were attached during in each case to suffer the same temperature exposure but with zero loads.

Test specimen	Creep temperature ^{o}K	Holding time (Hour)	Applied load during creep
(Tensile specimen) A2h44T A2h68T	876	44 68	0MPa
(Creep specimen) A2h44C A2h68C	876	44 68	79MPa

Table 3.3: *Data for the determination of the effect of creep on softening*

The result from these tests were used to compare with 603^{o}C recovery results to see if softening behavior is different under creep from ageing under static conditions.

3.6 The Creep Machine

The Denison T48 creep machine (Figure 3.3) was used to carry out the creep tests. Specimens were stressed by weights, via a lever arm. A constant temperature was created around the specimen in a furnace. Thermocouples, a linear variable differential transducer (LVDT), an amplifier, an electric thermometer and a plotter were used to record and control the data during testing.

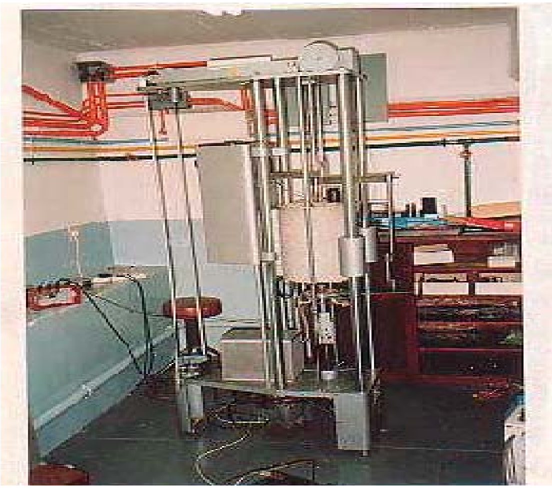

Figure 3.3: Experimental set-up for creep test showing the Denison T48 creep machine

3.6.1 The furnace

A three zone 17” furnace is used. The current through each zone was controlled from a control panel to achieve a constant temperature within the furnace.

The temperature control panel

The control panel has these furnace temperature control systems: heating, temperature monitoring and moderation. Three platinum resistance thermometers register the furnace temperature. The thermometers were inserted at three key points in the furnace; the top, bottom and the middle. The desired temperature is set digitally.

3.6.2 Height adjustment system

With the specimen creeping, the weight pan is displaced downwards. However, the lever-arm has to be kept horizontal. This is achieved by four micro- switches attached to the arm which in turn operates a height adjustment system. The height adjustment system consists of a geared motor which drives a screw thread. The motor is controlled from a panel at the bottom of the machine. By turning the screw thread, the height of the bottom tension bar is adjusted. As the weight pan nears the end of its course, the motor automatically drives the specimen down and the weight pan up. The motor is then switched off when the pan is at the top of its course.

3.6.3 Auxiliary equipment

Thermocouples

Three different lengths of thermocouples were inserted through the asbestos top and bottom seal of the furnace. This enabled the accurate finding of the temperature at the top, center and bottom zone of the furnace. The center zone of the furnace would align with the center of the specimen.

Transducer and amplifier

The linear variable differential transducer was mounted on a bracket parallel to the low alloy steel specimen. The output of the transducer went to the transducer amplifier. The transducer was, in turn, connected to the plotter.

The plotter

A two-channel plotter was used to record the displacement of the extensometer with time during the creep test via the transducer amplifier.

Acquisition of extension/time data

The extensometer (LVDT) (Photo 3.4.4) is attached to a bracket which in turn is attached to the upper part of the lever mechanism. This enables the LVDT to extend as the specimen increases in length. The extension of the LVDT is amplified and then sent to a plotter where it is recorded on a time vs extension set of axes. The amplifier is calibrated in such a way that 1.07 mm true extension of the specimen relates to 15mm on the plotter or plotted graph. The time axis read in 10 mm/hour i.e., the plotting paper is advanced 10mm every hour.

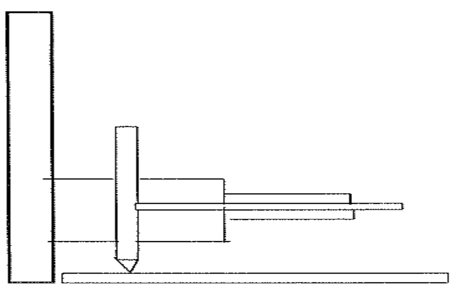

Figure 3.6: *Schematic view of the LVDT arrangement on the Denison machine*

3.6.4 Setting up the machine

- See that all switches are in the “OFF” position.
- Check all fuses in the control panels.
- A 24V DC supply must be connected to the height adjustment motor. Two fully charged 12V car batteries are ideal, and can be connected to the crocodile clamps at the end of the power cables. Note the polarity of the cables.
- Plug the machine into a 220V AC supply.

The Furnace

- Carefully pick up the furnace and secure it on the furnace holders, which are found on the thin vertical bars on either side of the machine. These are to be bolted onto the furnace.
- Connect furnace counterweight cables to the holders. These counter weights should exactly balance the weight of the furnace, facilitating easy slide up and down.
- Plug the furnace’s electrical cables into the socket provided at the back of the control panel

3.7 Aging Furnace

This comprise a standard piece of equipment that can accurately maintain temperature up to 900^{o}C.

The test specimen was kept in the furnace for a number of ageing tests, ranging from 560^{o}C to 640^{o}C for a period of two weeks. Tensile tests were then carried out on these samples, corresponding to stress rates of 1.5x 10^{-4} s^{-1}