# High Temperature Creep Measuring Apparatus

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**Abstract**
A high temperature creep measuring apparatus capable of operating over a wide range of temperatures, humidities and stress levels was developed. To assess its reliability, the apparatus was used to measure the creep behaviour of nylon6.6 tyre materials, under different temperature and humidity operating modes. At all operating conditions, cords exhibited instantaneous extensions on loading followed by steady creep over time with an eventual failure. The results obtained using the apparatus tallied well with results by other researchers, and this indicates that the apparatus can be used as a reliable measuring instrument.

**Key words:** creep, measuring apparatus, creep failure time, temperature, nylon6.6

## 1. INTRODUCTION

Creep measurement has been poorly understood due to lack of availability of creep measuring apparatus. A lot of work has been done on creep of textile materials, and each time an apparatus has to be designed for experimental work. For example Leaderman (1943) designed his own apparatus to help him measure the creep of nylon filaments. His results revealed that when the filaments were stressed and taken close to rupture at standard conditions of temperature and humidity, they recovered almost completely to their unstretched length soon after the stress was removed. Gillam (1969) used a different apparatus when he demonstrated that thermoplastic fibres such as nylon6.6, nylon6, polyester, etc show radical changes in mechanical performance when they are subjected to stress at high temperatures and temperature variations. Different researchers continued to use various types of measuring apparatus because an apparatus that could accommodate all different kinds of experimental work in creep, in textiles, had not and has not yet been found. A creep measuring apparatus was therefore designed and used to measure the creep of nylon6.6 tyre cord materials. The apparatus can use small and large weights and can measure creep from fine fibres to high tenacity yarns.

The creep measuring apparatus consists of an experimental chamber and a treatment chamber as shown in Figure 1. The treatment and experimental areas are well insulated to minimise the loss of heat to the outside. The inside dimensions of the experimental chamber are 40cm × 40cm × 45cm which permits testing of a 20cm specimen and allows the specimen to extend and creep until it ruptures. The apparatus is made of a 1 mm thick inside wall from stainless steel, which does not degrade due to temperature or humidity, and a 2.5 mm outside wall from ordinary steel painted to prevent rusting. Air circulates between the two walls 80 mm apart for insulation. In the treatment area, air is treated to suit the required conditions and circulated with the help of a fan. Experimental temperatures (up to 200°C) can be held either constant or controlled in cyclic modes by means of adjustable contact thermometers, which through a number of relays, ensure that the correct conditions are produced.

Cooling is achieved by using a refrigeration compressor. Humidity in the working chamber can be varied by using a water vapour injector. The supplies of heat, cold air, and water vapour are controlled through relays which open and close at appropriate controlled times. To enhance the method of control, energy regulators are used in heater and water vapour injector circuits, which reduce the possibility of unwanted fluctuations of temperature and humidity to a minimum [Fig 2].

Creep behaviour of yarn or cord specimens is measured with the help of a linear variable differential transducer (LVDT) situated under the treatment chamber so that it is not affected by temperature and humidity changes. The temperature and humidity are measured using a thermocouple and hygrometer respectively. Information relating to temperature, humidity and creep performance of the specimen at any given time during the experiment can be read, stored and displayed on a linked computer. The time at which the specimen ruptures is measured using a time counter. The time counter is connected to an electronic switch.. As soon as rupture occurs, the rod onto which the weight falls triggers 'off' the electronic switch (situated behind the chamber). When the switch is turned off, the counter stop (see Fig 1). The basic environmental chamber design of the creep measuring apparatus was adapted from the WEYCO climatic cabinet developed by Fisons(1969).

This paper reports on the development of a new high temperature creep measuring apparatus, and preliminary creep results obtained on nylon6.6 tyre materials, involving certain process and material variables.

## 2. EXPERIMENTAL VARIABLES: THEIR CONTROLS AND CALIBRATION [FIG 2]

Temperature and humidity are measured using a thermocouple and hygrometer respectively. Temperature up to 200° C can be held either constant or controlled in cyclic modes. Creep is measured using an LVDT. Data on temperature and creep performance can be stored and displayed on a linked computer. Humidity can be read on a humidity metre. The time at which the specimen ruptures is measured using a time counter, which connects to an electronic switch.

The control of temperature and humidity is achieved by means of adjustable contact thermometers through a number of relays. In addition to the relays, energy regulators are used to minimise the effect of electrical supply variations enabling the maintenance of constant temperatures. Once the main switch is on, the fan is immediately switched on to ensure that the chamber is not operated without air circulation.

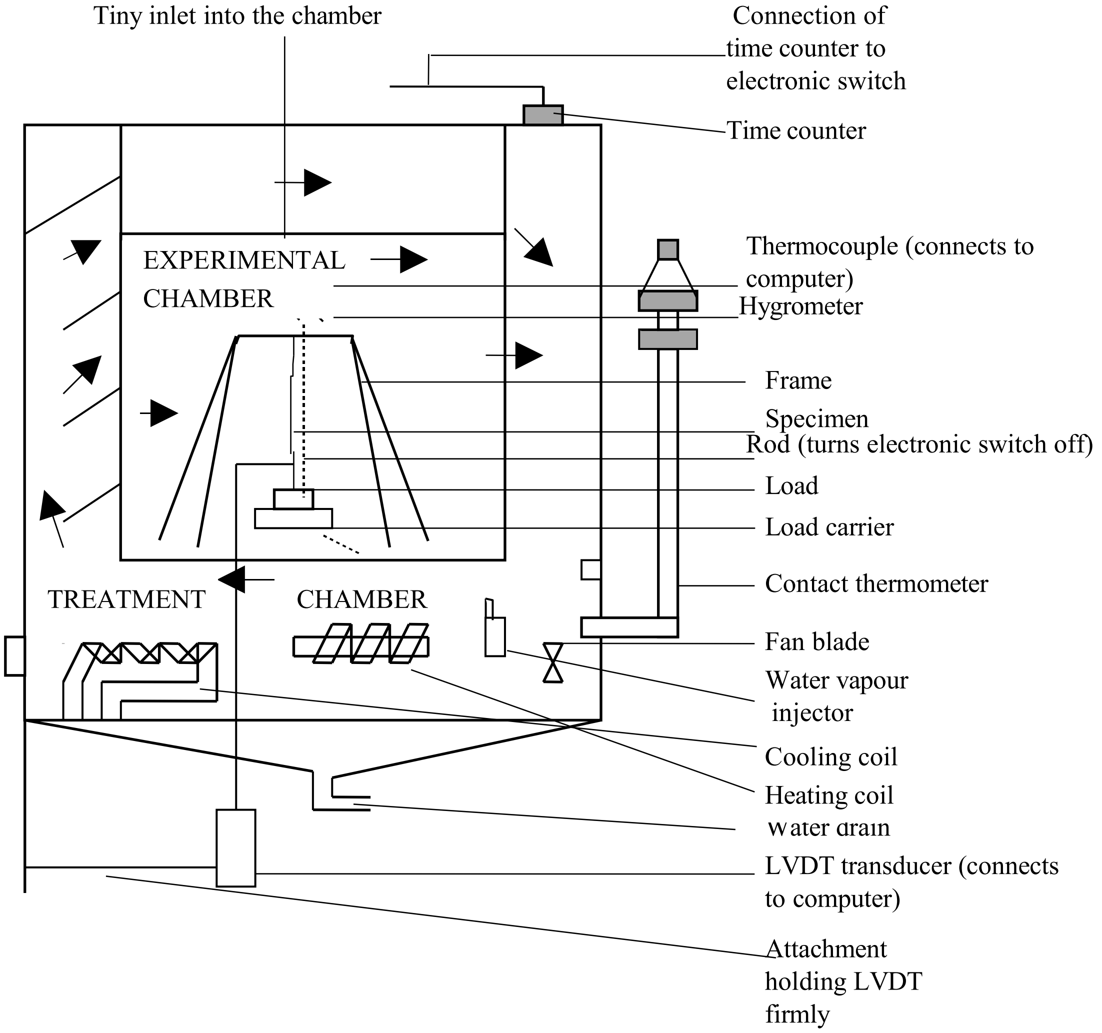



***Fig 1 Schematic diagram of the newly built creep measuring apparatus***

During cycling, the time switch turns 'on' and 'off'. On the 'on' position, relays 'A' and 'B' are energised, thereby breaking off the refrigeration circuit. In this instance the refrigeration can only be introduced through switch 'refrig A' when lowering of temperature is required. Energy is also supplied through the

'$H_A$' and '$M_A$' relays for heater and humidity respectively, passing through the contact thermometers which are calibrated accordingly. Through adjustable energy regulators, energy is passed to the heater, which controls the environment in the chamber.

On the 'off' position, the refrigeration circuit is made. Supplies to relays 'A' and 'B' are cut. Supplies are also cut for heater and humidity operating through relays '$H_A$' and '$M_A$' respectively, therefore stopping energy passing to the heater, resulting in the cooling off of the chamber, which can be accelerated by introducing refrigeration.

When the temperature is not being cycled, the time switch is not used. The energy is supplied from the main switch through the 'cycling/non-cycling' switch, which should be at the 'on' position (non-cycling position). In this situation relays 'A' and 'B' are continuously energised. The refrigeration circuit is cut off. The relays '$H_A$' and '$M_A$' are supplied with power through their respective switches. Non-cycling is a continuous process at a set temperature.

Relays '$H_B$' and '$M_B$' can be used during the cycling of temperature and humidity respectively. However these were made inactive during these experiments as the experiments were conducted at constant temperatures and constant humidity.

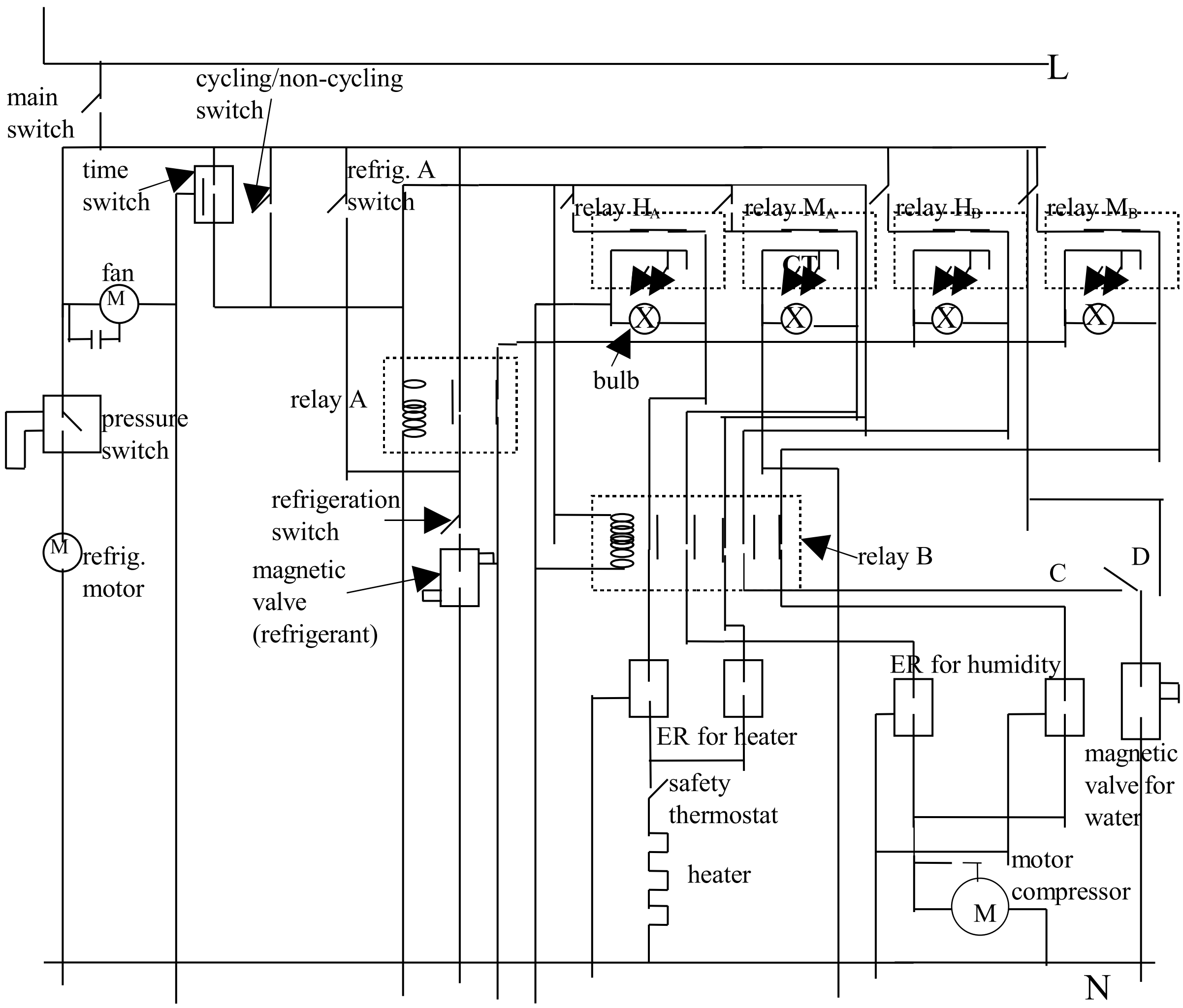
## 2.1 Temperature Calibration

A thermocouple type *k* was introduced into the experimental chamber through a small opening at its back. Care was taken to ensure that very little heat escapes through the opening. The thermocouple's correct value could be read on an indicator while its signal could be read on the computer. Having both values made calibration much easier. In fact, the relation between the two was

*Computer signal = 1.16 × thermocouple correct temperature.*

## 2.2 Humidity Measurement

The humidity can be read directly on the humidity indicator type HMI 41, which gives an actual humidity measurement. The indicator has a probe type HMP 45 install o-ring, connected to it, which, through the same opening as the thermocouple, can measure the humidity from the experimental chamber.




NB: ER are energy regulators for heater and humidity; C and D switch on main supplies to water valve

***Fig 2 Temperature and humidity control circuits***

The probe measures relative humidity between 0% and 99%. Both the indicator and the probe were obtained from Vaisala U.K., a company that specialises in temperature and humidity measuring equipment.

## 3. CREEP MEASUREMENT AND DATA ACQUISITION

Specimens used were 200mm long. At 200mm, the LVDT reading was set to be between 240-245, while at maximum extension of 250mm the LVDT reading was set to be between 10-15. Every 4.6 LVDT impulses represent 1mm creep of tyre cord. To enable continuous reading of the LVDT and thermocouple signals on the computer, a program written in the C language was used.

To ensure that it was a reliable tool, the apparatus was tested by using it to measure creep of nylon6.6 tyre materials.

### 3.1 Experimental materials

All experimental materials were either nylon6.6 filament yarns or cords made from them. The samples were obtained from Du Pont Nylon at their Doncaster manufacturing plant in the United Kingdom. The yarn was type 728 with a linear density of 140 tex. The yarns were converted into cords and subsequently dipped with resorcinol formaldehyde latex (RFL) dope. The physical properties of the samples of yarn, converted untreated cord (raw cord) and cord dipped in (RFL) referred to as dipped cord, were measured at standard atmospheric conditions of 65% relative humidity and 21°C temperature. All samples were conditioned at the same standard atmospheric conditions for at least 24 hours prior to measurements being taken. Tensile properties were measured using an INSTRON 4301 apparatus, while the number of turns of twist was counted using a standard twist tester. For each sample and every operating condition, ten specimens were tested and the mean values of results used for data presentation (Table 1).

### 3.2 Measurement Of Creep

Creep was measured under two thermal loading conditions: isothermal and non-isothermal (cyclic). Tests at isothermal conditions were done at controlled and uncontrolled humidity. This study reports only on tests done at isothermal conditions with both controlled and uncontrolled relative humidity.

***Table 1 Yarn samples and their properties***

| **Sample**<br>**Property** | ***Yarn (210 filaments)*** | ***Raw cord*** | ***Dipped cord*** |
|---|---|---|---|
| *Linear density(tex)* | 140 | 313 | 320 |
| *Twist/cm plied*<br>*single* | none | 3.2S*<br>3.1Z* | 3.4S*<br>2.8Z* |
| *Elongation at break (%)* | 15.5 | 21.5 | 21.6 |
| *Breaking force (N)* | 98.2 | 177.7 | 176.6 |
| *Tenacity (N/tex)* | 0.70 | 0.57 | 0.55 |

**S* and Z* show direction of twis**

Although tyre cords in a tyre are never exposed to humidity, tests on controlled humidity were carried out especially to assess the reliability of the apparatus. Test specimen were nylon6.6 yarn, raw cord and dipped cord. To ensure that testing times were reasonable to allow the completion of the study within the limited time, 60% stress level (60% of breaking load that was measured at standard atmospheric conditions) of each sample was chosen as initial strain load.

### *3.2.1 Isothermal Experiments At Uncontrolled Relative Humidity*.

Creep behaviour of various samples was measured at room temperature (around 20°C), 50°C, 75°C, 100°C, 130°C and 150°C. For each experimental temperature, ten specimens per sample were tested. The temperature of the working chamber was first raised to the required level and the specimens were then loaded while under stress. At 100°C and above, each sample loading took a little over half a minute resulting in a fall of the actual chamber temperature by no more than 5°C. It needed 2 to 3 minutes for the temperature to pick up to the required level. Below 100°C the fall of the chamber temperature for the above reason was no more than 3°C. The creep performance of the specimens as a function of time was recorded until they failed.

### *3.2.2. Isothermal Experiments At Controlled Relative Humidity*

Experiments at controlled relative humidity were conducted only at 35ºC, 50ºCand 70ºC, with humidity controlled at 25%, 60% and 90%. These temperatures
were chosen because it became apparent that relative humidity was difficult to maintain at a required level when temperatures were below 35ºC and above 70ºC. At 35ºC, relative humidity could not go as low as 25%. The temperature was too low to maintain the humidity at 25%. Instead the humidity gradually increased to values above 25. Above 70ºC it could not go as high as 90% due to high heat which kept on drying the chamber quickly. Like in experiments on controlled humidity, ten specimens per sample were conducted at each experimental temperature and humidity level.

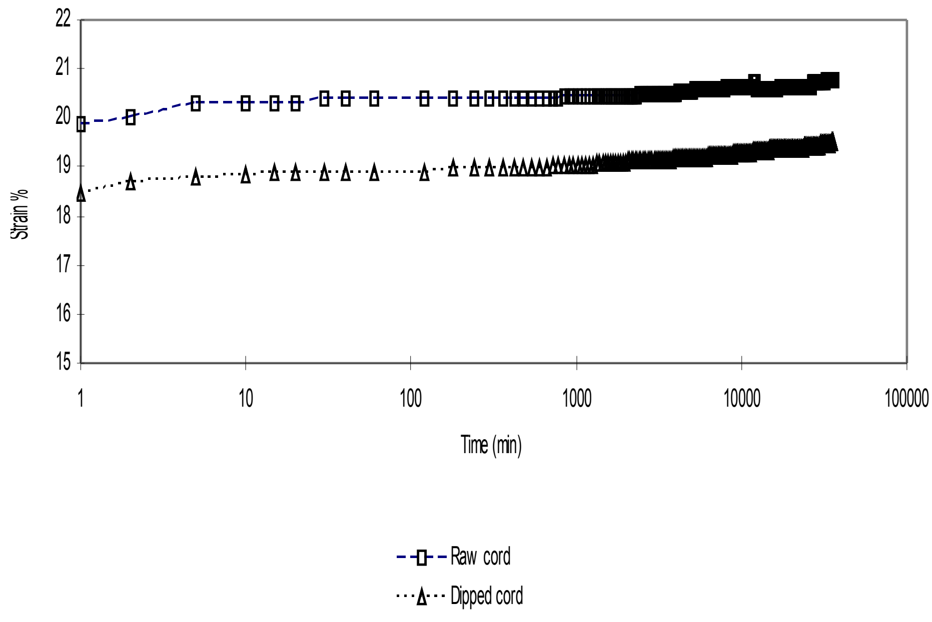



*Fig 3: Creep - time relationships of raw cord and dipped cord at room temperature*

## 4. RESULTS AND DISCUSSION

Preliminary results on isothermal experiments at uncontrolled relative humidity, conducted at room temperature, 50°C, 75°C, 100°C, 130° C, and 150°C on yarn, raw cord and dipped cord, have shown that at 60% stress level, all specimens, irrespective of their geometry, exhibit an instantaneous elongation on loading followed by steady creeping over time, with an eventual failure (Figures 3-5). According to Takeyama and Matsui (1971), tyre cords behave in a similar manner under real life loading conditions, although they carry a maximum load of only up to 20 % of their breaking load.

Results from the study show a creep trend similar to that observed by Wilding and Ward (1981 and 1984) when they investigated the creep and recovery of ultra-high-modulus of polyethylene. Like results from this study, Wilding and Ward's results show a steady and continuous increase in creep with time, which suddenly accelerated as the specimen nears rupture, even at a low temperature of 20ºC. Their creep rate, although higher than that observed in this study, also increased with increasing temperature, leading to quick rupture at high temperatures.

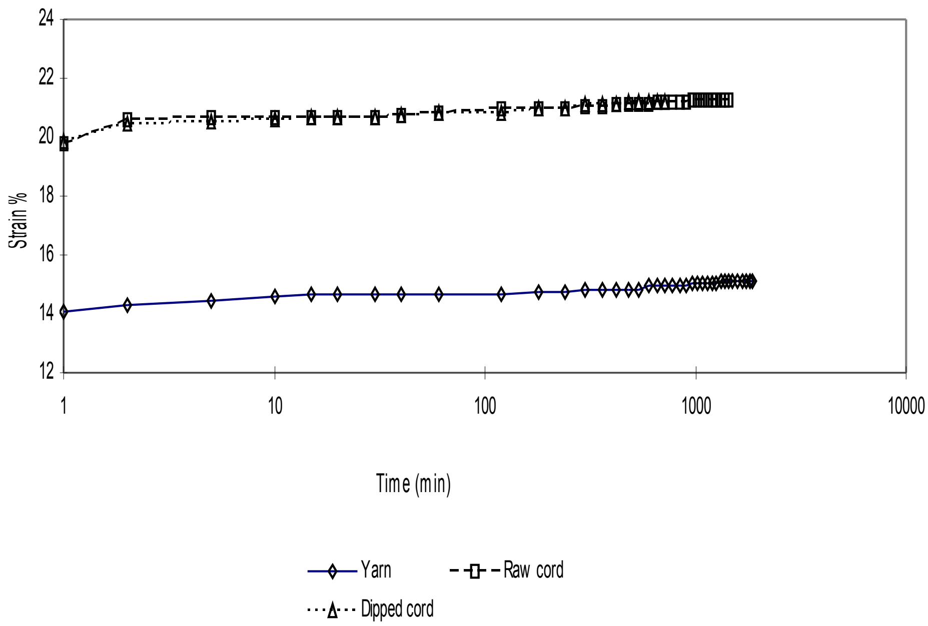



*Fig 4: Creep - time relationships of yarn, raw cord and dipped cord at 75°C*

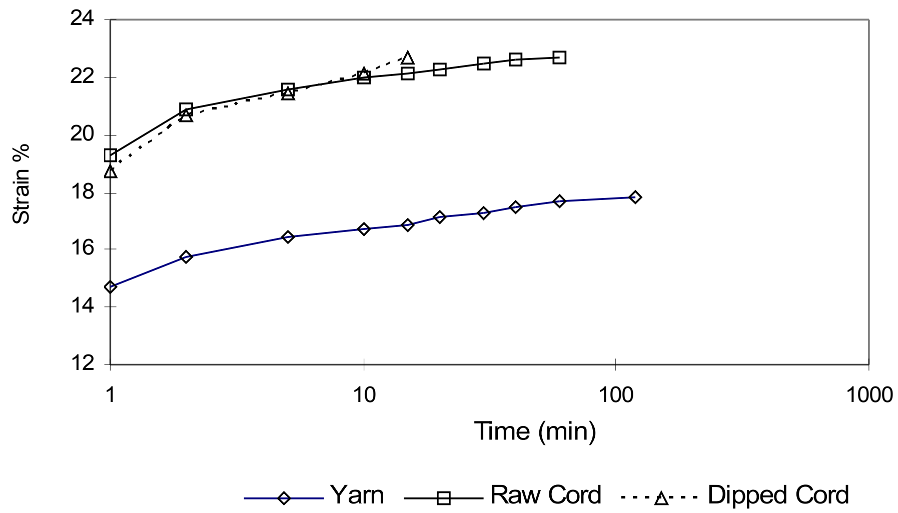



***Fig 5 Creep - time relationships of yarn, raw cord and dipped cord at 150°C***

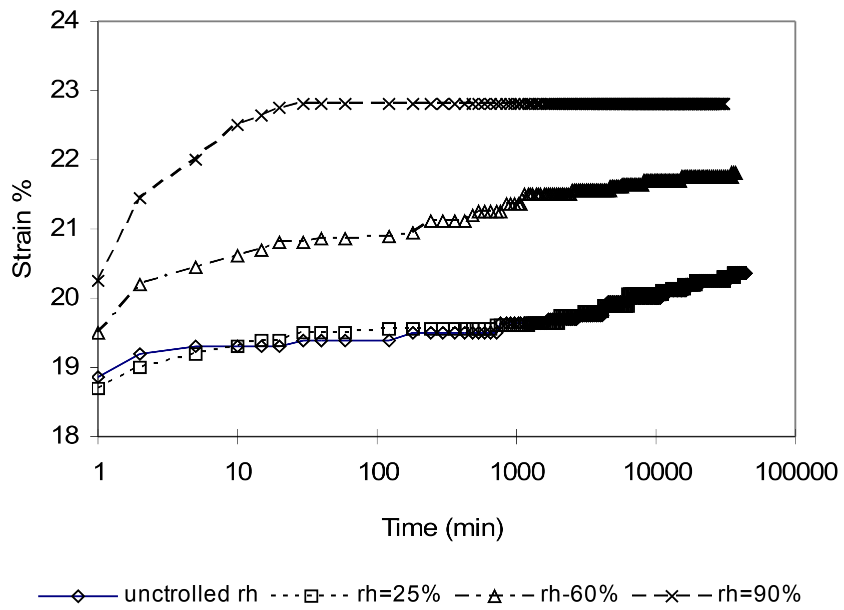



***Fig 6 Effect of humidity on creep of raw cord at 35 °C***

The results in Figures 3-5 show that increasing operating temperature significantly reduces creep failure time. This is due to accelerated creep rate. It is envisaged that high operating temperature and level of stress can activate a large number of processes in a semi crystalline polymer like nylon6.6. Literature has shown that at 100°C hydrogen bonds in nylon6.6 can be subjected to thermal vibrations and amorphous materials could be in a liquid like state of dynamic equilibrium (Mukhopadhyay, 1992). Application of uniaxial load to a nylon 6.6 yarn or cord at a reasonably high temperature (e.g.100°C) will, therefore, cause a flow in the amorphous regions activating rapid material extension, creating more weak points, initiating cracks and eventual failure.

A study by Nkiwane and Mukhopadhyay (1999) defines the creep curve of nylon6.6 tyre materials with an equation:

$$e = e_0 + creep$$

where $e_0$ is the instantaneous extension,

$$creep = \sigma_0 /E\ (1-e^{-t/\tau})$$

where $\sigma_0$ is initial stress, $E$ is elastic modulus, t creep is time, $\tau$ is retardation time.

When $t = 0$, $e = e_0$, and when $t = \infty$ $e_\infty \approx e_r = e_0 + \sigma_0 /E$, resulting in

$$\sigma_0 /E = e_r - e_0$$

where $e_r$ is the strain at rupture.

Strains $e_0$ and $\sigma_0 /E$, and retardation time $\tau$ are constant which means the total strain $e$ is dependent on time $t$.

Figures 3-5 show that both raw cord and dipped cord exhibit relatively high initial extensibility as compared to the yarn, which could be attributed to the highly twisted structure of the cord. However, the ultimate creep life of a cord is much shorter than a yarn. It is reasonable to assume that the filaments in a highly twisted and strained cord can promote high levels of frictional forces between themselves causing possible surface crack initiation (Nkiwane, 2001). As temperature increases, such cracks propagate very fast due to flow behaviour of polymeric chains resulting in faster failure.

For experiments at controlled relative humidity, at temperatures below and around nylon6.6's glass transition temperature (Tg) which is 45ºC, the creep on nylon tyre materials is not affected by the rh of 25% because little water is absorbed by the cord structure (Fig 6). Change in creep starts to show when the humidity is increased to 60% and then to 90%, because , then, the yarn and raw cord allow water to penetrate their structure ( Fig 7 and 8). A combination of increased temperature and high relative humidity leads to an increase in creep rate because the molecular cohesion of the nylon6.6 structure is weakened to a greater extent than when it is affected by temperature or humidity alone, thus causing a greater elongation of the cord structure.

The dipped cord did not show much change in creep life at increased rh and temperatures around and below its Tg because the RFL resin adhered to the cord surface, coating it and preventing water penetration into the cord structure, therefore resulting in no change in its molecular chain cohesion. At 70ºC the dipped cord behaved in the same manner as the yarn and raw cord under similar conditions, most creep taking place within shorter time as rh increased (Fig 8). This temperature was high enough to soften the RFL on the cord surface, allowing water molecules into the cord structure.

In general all results obtained using the suggested creep measuring apparatus are consistent with results carried out by some researchers (Leaderman 1943, Gillam 1969, Wilding et al, 1981 and 1984). However, results obtained in this study were further analysed in order to develop a deeper understanding of the creep of nylon6.6 tyre materials (Nkiwane et al 1999, Nkiwane 2001).

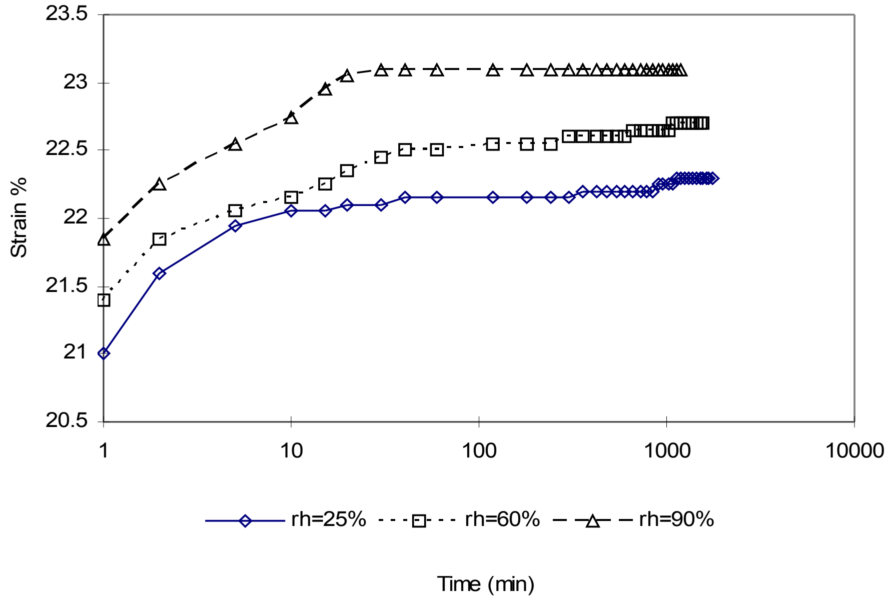



***Fig 7 Effect of humidity on creep of raw cord at 70 °C***

## 5. CONCLUSION

Results obtained using the new apparatus at constant temperatures were comparable to those done by other researchers, which suggests that the designed apparatus operated within acceptable standards. Nevertheless, the varied relative humidity remains an important area of study in order to minimise accidents that still occur due to tyre rupture. Hopefully this apparatus will go a long way in minimising the need to develop creep-measuring instruments not only for textile experiments, but for other fields as well.

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