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The time-dependent mechanical
properties of fibre reinforced polymers

- by -

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1. General Progress

In the six months from April to September 1968 the task of primary importance has been to design lateral extensometers and to improve the longitudinal extensometer and consequently enable reproducible measurements of the deformations undergone by creep specimens to be made.

Resulting from experience gained in testing using the existing tensile creep testing machine, slight alterations are being made to the five further machines now in production. So that specimens of unreinforced resin can be made, sheets of resin of constant thickness are required. A mould has been designed and manufactured in which these sheets of resin are produced.

To find the Young's modulus of the 'E'-glass fibres used for the reinforcement of the composite materials, a minor series of tests has been conducted using an Instron testing machine.

As the creep tests conducted during the past six months have used extensometers that have not been fully developed, the results obtained from them have been designated as preliminary. These results have been used to indicate the loading conditions required for the main series of creep tests.

The computation associated with the theoretical work reported in Progress Report No. 1 (March 1968) has been completed and the finished work with examples of its use has been recently published (August 1968). Consequently no theoretical work is contained in this report. The results from the preliminary creep tests on unreinforced resin and 100 second isochronous tests on the uniaxially reinforced system have been used to examine the accuracy of the theoretical predictions.

2. Apparatus Development

2.1 Longitudinal Extensometer

In the previous progress report (March 1968) the design of a longitudinal extensometer is described. This extensometer is clamped to the specimen at each end of the gauge length by means of two silver steel rods. Trials of this extensometer on I.C.I. Darvic unplasticised P.V.C. showed that at low stress levels the extensometer gave results for 100 second creep coinciding with the values quoted by Turner (December 1964). It should be mentioned here that a small 12 volt A.C. vibrator has been developed which is used to prevent any stick-slip of the transducers on all extensometers. At high stress levels however it was found that the extensions being recorded were larger than Turner's results. It was thought that this was probably due to premature yielding in the region of the extensometer clamps, indicating stress concentrations in the specimen in this region. To confirm that stress concentrations do in fact exist in this area, further 100 second creep tests were conducted on a rubber modified polystyrene. This material was chosen

because it is very prone to crazing which can easily be observed. The crazing mechanism is very stress dependent and consequently if the stress is chosen judiciously very little crazing is observed at the average stress in the specimen but at the increased stress in the region of a stress concentration the crazing is very marked. By loading a specimen with its extensometer attached at this stress level the stress concentrations can be readily observed around the grips of the extensometer.

To eliminate these stress concentrations it has been necessary to redesign the means of clamping the extensometer to the specimen. Figure 1 shows the details of one of these clamps. Stuck to either side of the specimen using Araldite glue are cylindrical aluminium pads, $\frac{1}{8}$ inch in diameter and .05 inch thick, located in position by a jig. The screws, by which the extensometer is clamped to these pads, have their ends machined to give a very fine point protruding from a flat surface. When the screws are tightened up the points pierce the aluminium pads and prevent slipping, and the hard shoulders, in contact with the surfaces of the pads, prevent any possible twisting of the extensometers. Figure 2 shows a general view of the extensometer and it can be seen to be of the direct reading type, using a Sogenique linear displacement differential capacitance transducer as its sensitive element. Further 100 second creep tests on rubber modified polystyrene using this type of extensometer failed to indicate any stress concentrations related to this method of clamping. The accuracy and reproducibility of this extensometer were checked by testing further samples of 'Darvic' P.V.C. in creep. The 100 second isochronous stress-strain curve obtained agreed with Turner's results to within 3% up to a stress of 4×10^8 dynes/cm² which is equivalent to a strain of 1.6% in this material. This accuracy is considered to be satisfactory, taking the variation from sample to sample into account.

2.2 Lateral Extensometer

A lateral extensometer is required to measure the lateral creep contraction associated with the longitudinal creep.

The first extensometer that was designed for this purpose is shown in Figure 3. In principle it is very simple as it consists of two probes in contact with the sides of the specimen with a linear displacement transducer measuring their relative movement. The spring incorporated in the transducer is adequately strong to keep the probes in contact with the specimen. The main problem associated with the use of this extensometer is that as the specimen creeps longitudinally it moves through the probes of the extensometer. Consequently if the specimen is not exactly parallel sided the transducer will record the change of width of the specimen, as well as its lateral contraction. Using the routing technique to produce samples the width is constant to within $\pm .002$ cm, but as the transducer measures with a sensitivity of .00001 cm. this represents a large error. Tests using this extensometer indicate that the errors involved are normally small but that large errors can occur. To enable this extensometer to be used with a guarantee of accuracy it is therefore necessary that the specimens should be ground in the

locality of extensometer probes to ensure a parallel section.

As this would be a time consuming process it was decided to try an alternative method first. The second extensometer is designed to measure the contraction over a fixed gauge length. Because the gauge length is small (of the order of $\frac{3}{4}$ inch) the pads stuck to the surface of the specimen must be small to keep end effects to a minimum. Figure 4 shows the extensometer designed to measure changes of this gauge length. This consists of two arms, each fixed to a pair of the brass pads and able to rotate freely about conical holes machined in the centre of each pad. These arms rest on rollers suspended between ball races to enable the arms to move without friction. The relative movement of the two arms is measured by a transducer. As the specimen creeps the arms rotate about the brass pads so as to remain in contact with the rollers. The slight geometrical error introduced by the two arms rotating about different centres has been calculated to be small enough to be ignored even at relatively large strains. This extensometer has been tested on 'Darvic' P.V.C. and consistent reproducible results have been obtained for the creep contraction ratio at 100 seconds. Very little data has been published for the creep contraction ratio of P.V.C. and what data is available is widely divergent but the value obtained lies within the range of values. Further tests using this extensometer but using specimens of less thickness have shown that some errors can still be incurred due to flexibility of the samples.

Attempts have been made to measure the creep contraction ratio of the specimen through its thickness by measuring the relative movements of the two faces. The variations of the thickness of the specimen are however too large to make this feasible without grinding the two surfaces in the locality of the probes.

2.3 Specimen production

In order to be able to produce samples of unreinforced resin it is necessary to make sheets of resin of constant thickness. Figure 5 shows the mould that has been designed to make these sheets in. It consists of two pieces of $\frac{3}{8}$ inch plate glass, 1 ft. square, bolted together around three sides of their circumference and held a constant distant apart by a polythene spacer around these same sides. To prevent any leakage from the mould, the polythene spacer is smeared with petroleum jelly before insertion. The glass plates are coated with a wax polish to act as a release agent for the resin. Around the edges of the glass plates are steel angle supports to prevent any bending. To facilitate the pouring of resin into the mould, the top edges of the glass plates are bevelled inwards. Resin is poured with the mould in the vertical plane and it has been found that the air bubbles in the resin have adequate time to rise to the free surface.

Many sheets of uniaxially reinforced resin have been produced using the filament winding machine. It has been found that the critical process for controlling the volume fraction of the glass of the composite is not the

variable rollers of the filament winding machine, as has been expected, but the large rubber rollers through which the composite is drawn to remove air bubbles. The minimum pressure that must be applied to the composite to remove all the air bubbles has been found to remove resin as well and a volume fraction for the glass of much less than 0.40 cannot be obtained in consequence. Using the equipment at present available it is difficult to raise the pressure exerted by the rollers high enough to make any significant increase in the percentage of glass present in the final composite. As a consequence, the volume fraction of glass present in all the composites manufactured to date has been of the order of 0.4.

2.4 Alterations to creep-rig

In the original design for the creep rig all the main bearings were specified as being roller bearings as these have a high static load capacity. These have no automatic method of location and it has been found to be inconvenient to need to centralise the system for each test. Consequently in the five new machines currently being produced, double ball races, having an adequate static load capacity and a suitable locating mechanism, will be used.

During the preliminary 100 second tests on the uniaxially reinforced system it was found that on some tests on the 0° system that loads were applied to the specimen up to the design limit of the machine. At these loads the specimen pulled out of the jaws. To prevent this, the inside faces of the jaws have been lined with fine grade carborundum paper allowing the specimen to be gripped adequately.

To facilitate the manufacturing procedure of the jaws the design has been altered slightly. Now the jaw faces are clamped to the specimen outside the machine and the specimen and jaws are loaded into the machine by passing a $\frac{1}{2}$ diameter pin through each pair of jaws. The only attendant problem with this design is the cutting of an accurate $\frac{1}{2}$ inch diameter hole on the centreline of a sample of glass reinforced resin. It is hoped to overcome this problem by trepanning using the $\frac{3}{16}$ inch hole, used to locate the router template, to centralise the $\frac{1}{2}$ inch hole.

3. Experimental Results

3.1 Young's Modulus of 'E'-glass fibres

The manufacturers of 'E'-glass fibres quote their Young's Modulus as being the same as that of the bulk glass from which they are drawn. It was decided to test the practicality of measuring the modulus of the fibres themselves, and if it should prove practical to check the value of modulus obtained with the values quoted by manufacturers.

The first problem encountered was to measure the diameter of individual fibres to an accuracy consistent with the required accuracy of the modulus. The 'E'-glass, as it is obtained from the manufacturers, is in the form of a

roving consisting of twelve strands of 204 fibres. As the diameter of the individual fibres is of the order of .001 cm a single fibre must be removed very carefully from the roving. This fibre was then stuck to a glass slide and viewed through a microscope with one thousand times magnification using transmitted light. The measuring graticule, used with the microscope was graduated into 1 mm. divisions. Consequently to obtain a 1% accuracy of measurement it was necessary to estimate to $\frac{1}{10}$ division. It being difficult to focus clearly on the centre plane of the fibre this estimation was not easy.

After the diameter of each fibre had been measured it was gripped in the Instron testing machine prior to loading. The grips used were rubber faced and designed specially for use with fibres. The load scale used for the tests was 0-10 gms and the cross head speeds chosen lay between 0.05 and 0.5 in/min. The extension of the fibre was measured as being equal to the cross head movement of the machine initially, but it was found that there was considerable contribution from the extension of the gripping system. This was allowed for in later tests by using fibres of lengths varying from one to twelve inches and thus being able to calculate the contribution of the grips.

The value of the Young's modulus obtained in this way for these 'E' - glass fibres was 6.55×10^{11} dynes/cm² with a scatter of $\pm 5\%$. This compares with the values of the modulus of the bulk glass quoted by the manufacturers varying from 6.90×10^{11} dynes/cm². Whether this difference is due to inaccuracies in the measuring system or to a change in the properties of the glass when it changes from its bulk form to fibres is not yet known.

3.2 Creep compliance of unreinforced resin

Several tensile creep tests have been made on samples of unreinforced Araldite MY 753 with HY 951 hardener to measure the creep compliance and the creep contraction ratio to enable some preliminary predictions to be made as to the behaviour of the uniaxially reinforced composite.

Two difficulties have been encountered during this series of experiments. The first has been that the lateral extensometers that have been in use have been in a state of development and the results obtained have not been completely reliable. The second has been that the sheets of resin have become warped during the curing process as the shelves in the oven used were not perfectly flat. These problems have now been rectified but the results shown here may contain inaccuracies.

Figure 6 shows the creep compliances of the two straightest samples that were tested. The first sample was tested for 5000 seconds, as this was only intended as a preliminary test, while the second sample was tested for 100,000 seconds which will be the normal duration of future creep tests. The variation between these curves is due to the first sample being less straight than the second. Other samples tested where the bend in the gauge length was more obvious gave considerably higher compliances as would be expected.

On the basis of some 100 second creep tests it was decided that the initial creep contraction ratio was 0.37 although this value could be in error. With no information to the contrary it was decided to assume that the Poisson's ratio of this resin behaved in accordance with Turner's (1966) approximation that the bulk modulus is independent of time.

The results of the preliminary creep test have been analysed using a computer program developed by the author while at the Civil Engineering Department of the University of Southampton. This program calculates the optimum finite retardation time spectrum of the resin from its creep results by a linear regression technique. In Figure 7 is the six element spectrum calculated to describe the three decades of creep curve analysed. The accuracy of this approximation is such that the sum of the square of the errors is $5 \times 10^{-29} \text{ (cm}^2/\text{dyne)}^2$ which represents an average error of 0.06%.

3.3 100 second creep compliance of the uniaxially reinforced resin

In order to enable a preliminary comparison between theoretical and experimental results to be made, a set of 100 second creep tests were conducted on specimens cut from a uniaxially reinforced sheet of resin at various angles to the fibre axis. It is hoped that while the values of the 100 second creep contraction ratio were measured using the first type of lateral extensometer described, and in consequence may not be very accurate, that they should indicate the correct trend.

Figure 8 shows the predicted variation with angle of the 100 second compliance of the uniaxially reinforced system used here compared with the experimentally obtained values. The prediction is based on the preliminary results of the creep compliance of the resin and the assumption that the volume fraction of the glass is 40%. The volume fraction of the individual specimens were measured by first weighing the gauge length of each specimen and then burning off the resin content in an oven at 680°C for 15 minutes. The weight of the glass left was then recorded and the volume fractions of the two phases calculated assuming that the specific gravity of the resin was 1.18 and the specific gravity of the 'E'-glass was 2.55. The volume fractions of the specimens varied between the limits of 37% and 42%.

There is good agreement between the experimental and predicted results with the predicted results being slightly high. This is due to the preliminary creep sample being slightly bent.

Figure 9 shows the predicted variation of the 100 second creep contraction ratio with angle for the uniaxially reinforced resin with a volume fraction of glass of 40%. The experimental results with which these predictions are compared show a similar but less noticeable trend.

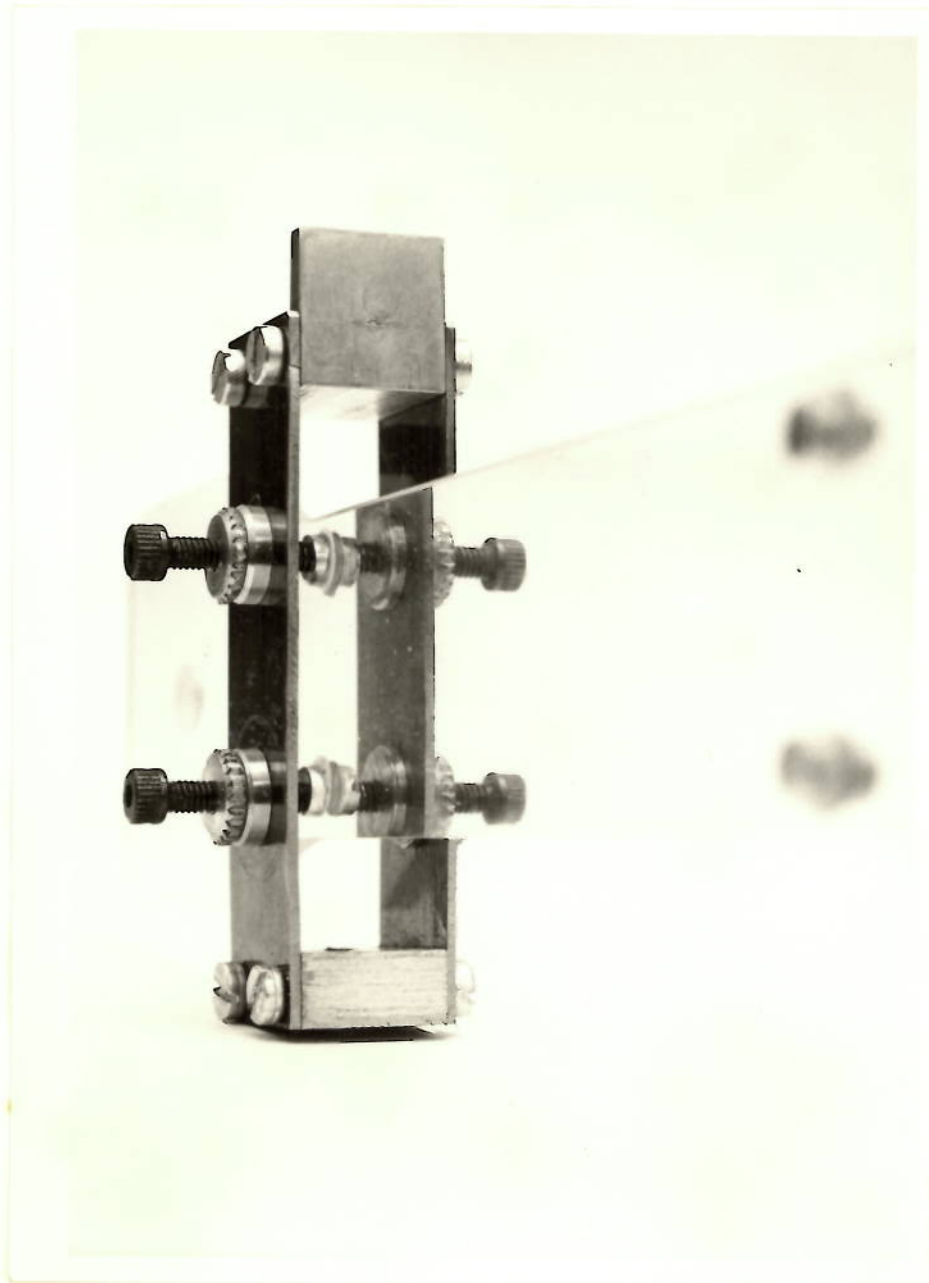


FIGURE 1 LONGITUDINAL EXTENSOMETER CLAMP

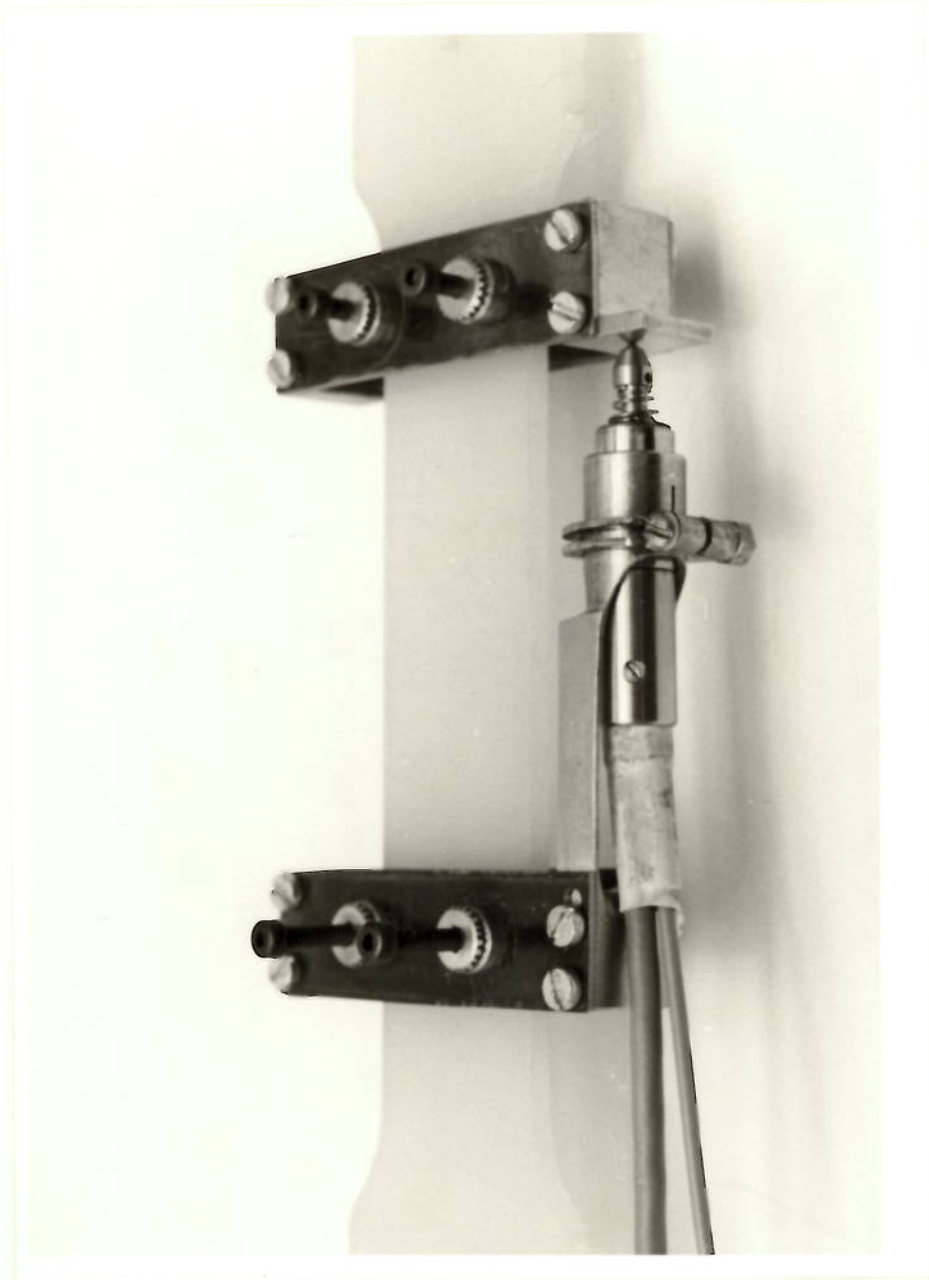


FIGURE 2 LONGITUDINAL EXTENSOMETER

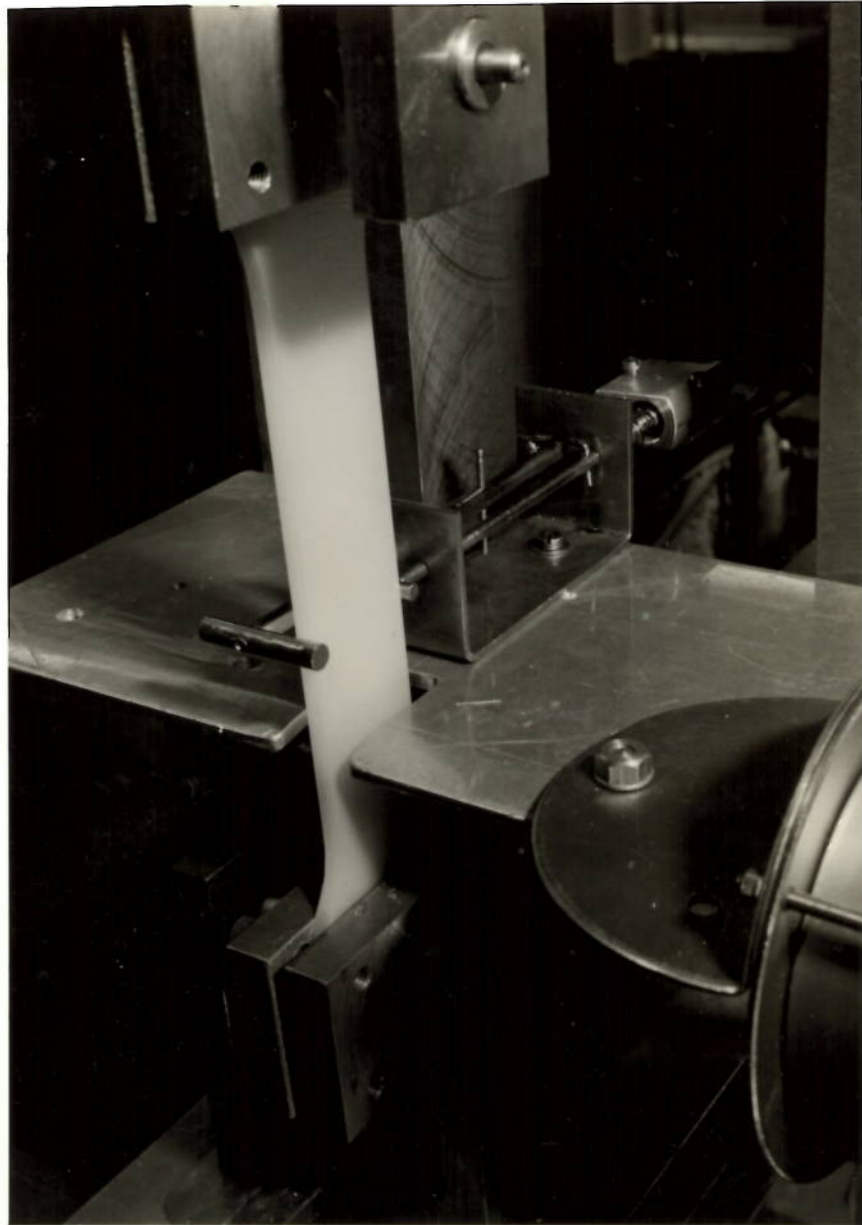


FIGURE 3 SURFACE MEASURING LATERAL EXTENSOMETER

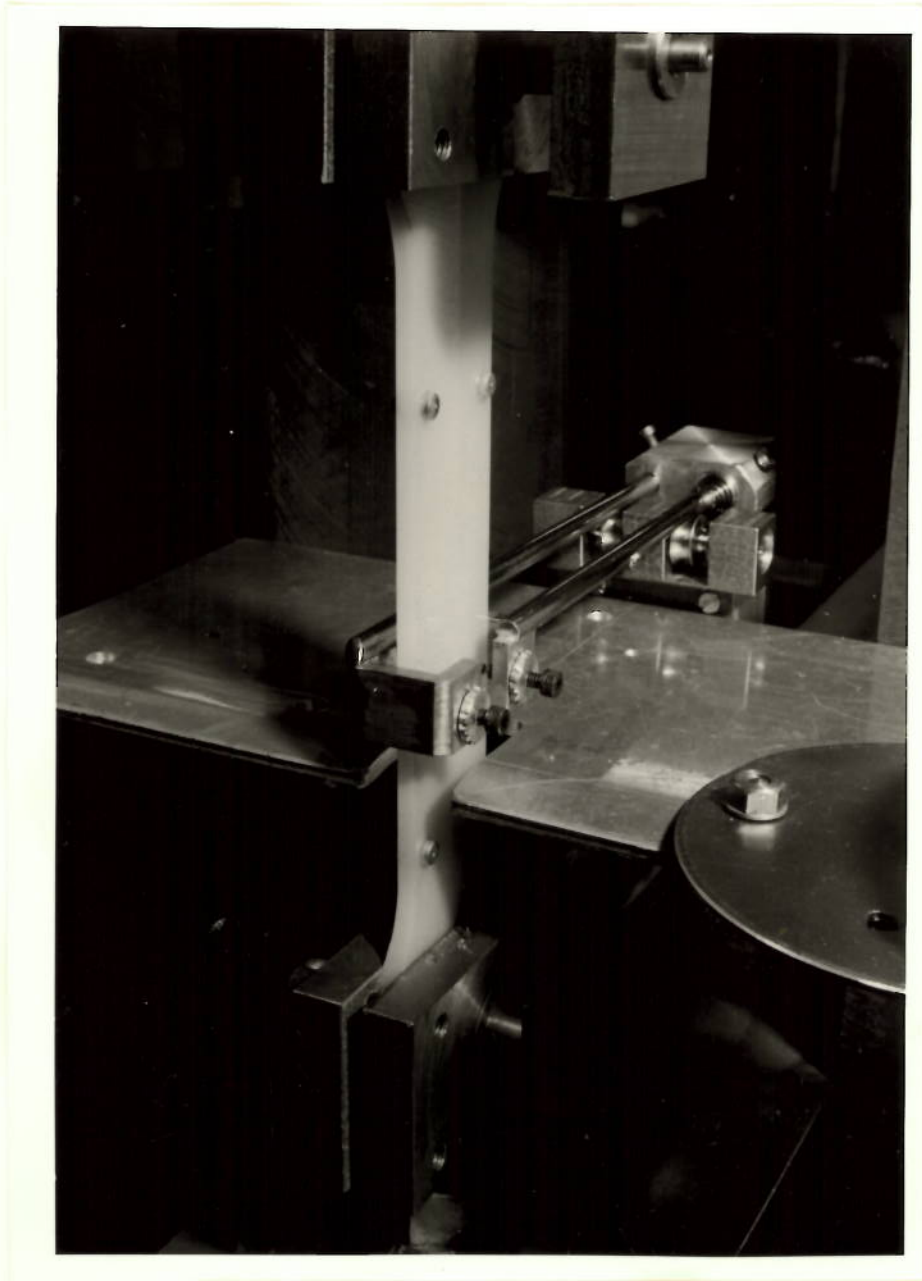


FIGURE 4 MODIFIED LATERAL EXTENSOMETER



FIGURE 5 MOULD FOR UNREINFORCED RESIN

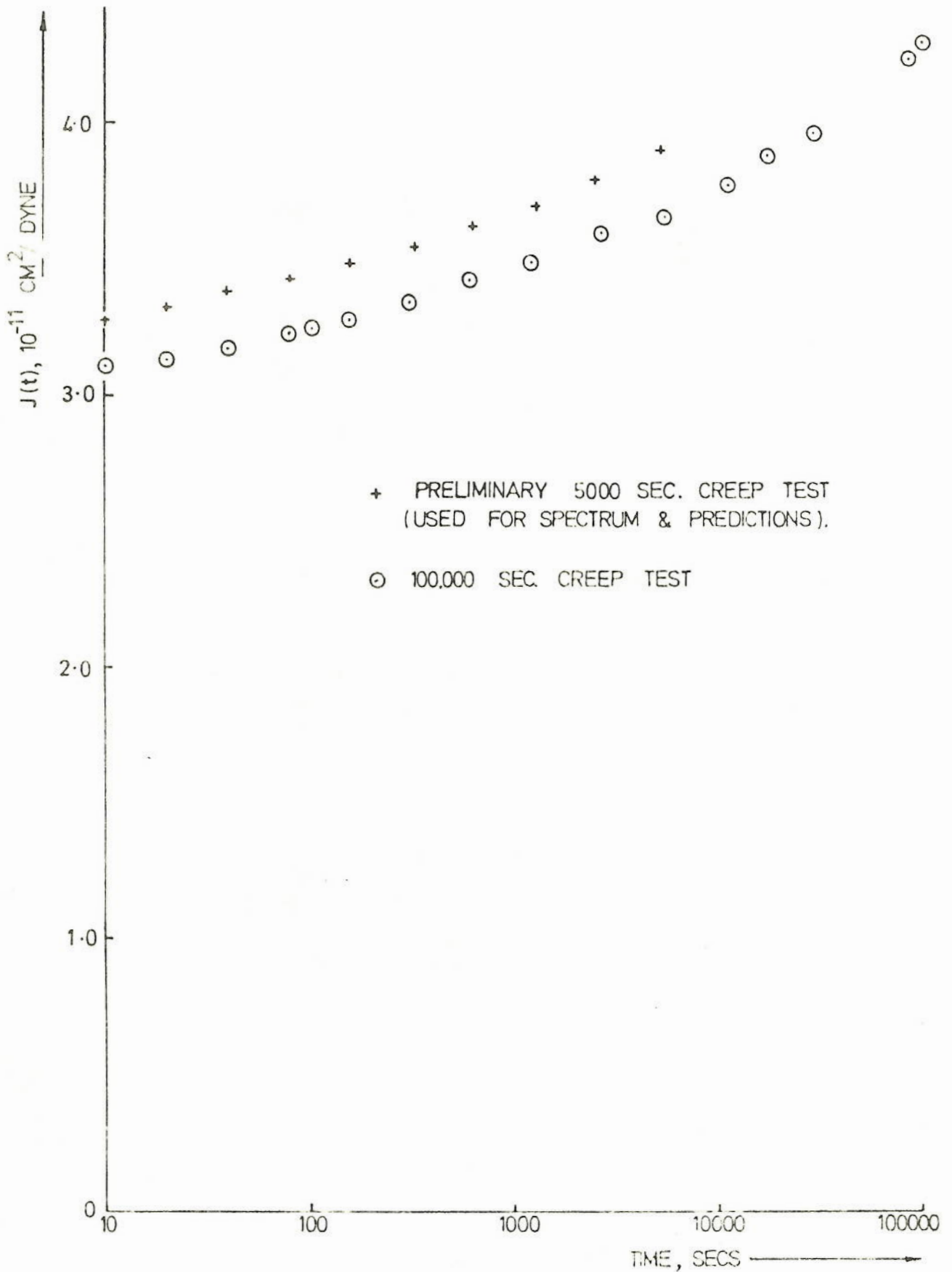


FIGURE 6. CREEP COMPLIANCE OF UNREINFORCED 100% MY 753

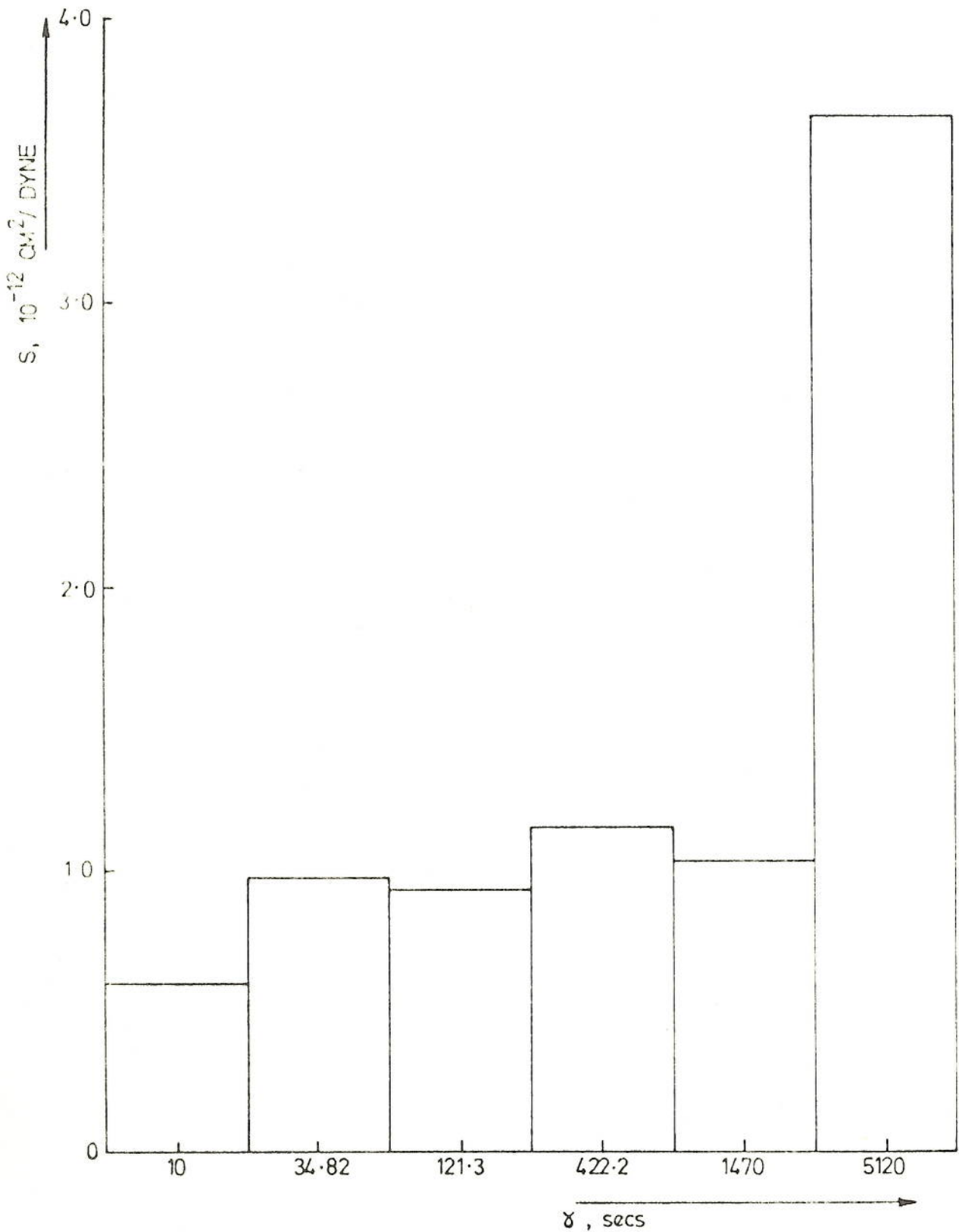


FIGURE 7. SIX ELEMENT FINITE RETARDATION TIME SPECTRUM OF 100% MY 753, BASED ON 5000 SEC. CREEP TEST.

$$S_0 = 3.199 \times 10^{-11}$$

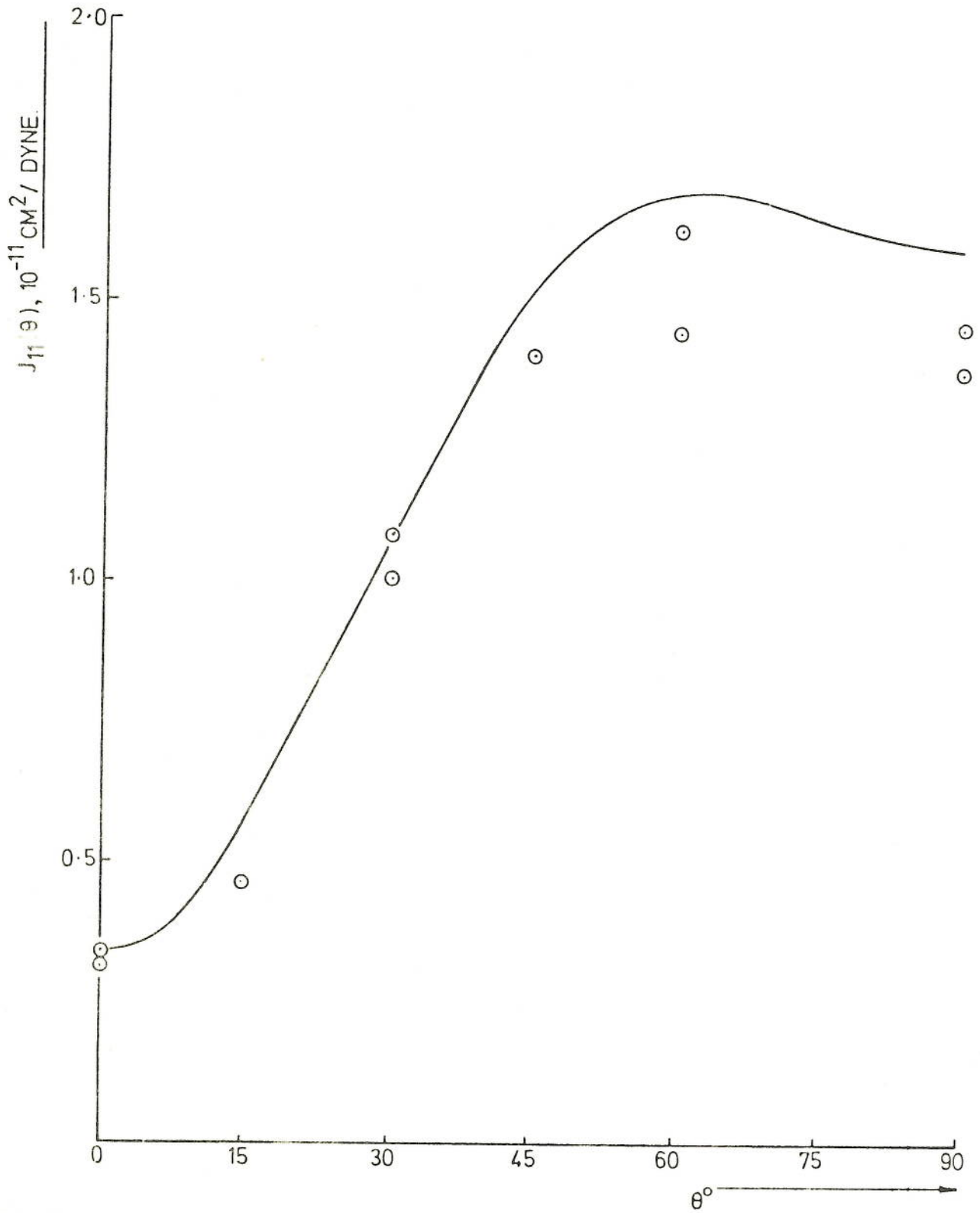


FIGURE 8. VARIATION OF THE 100 SEC. CREEP COMPLIANCE OF UNIAXIALLY REINFORCED MY 753 WITH ANGLE. $V_f = 0.40$.

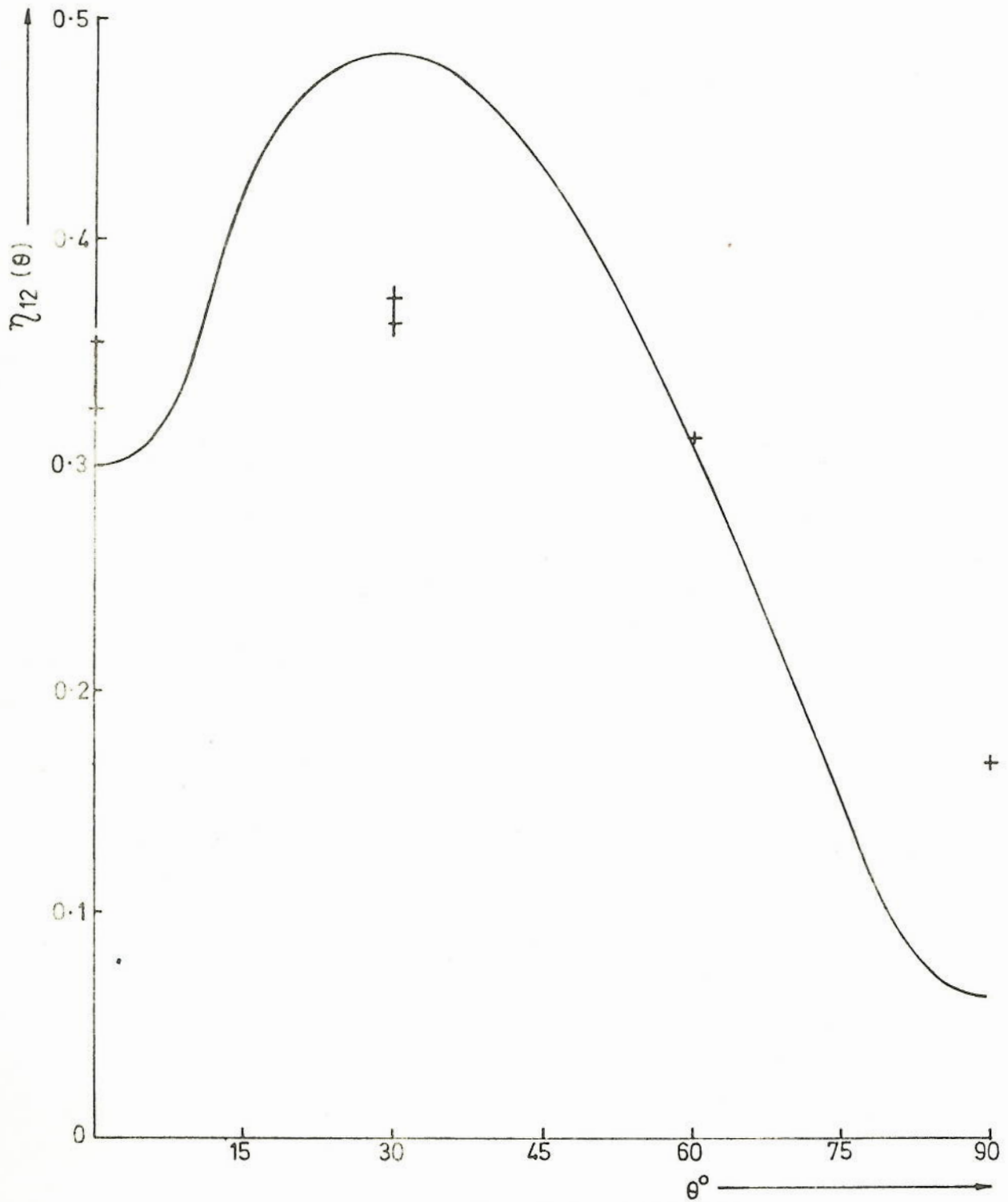


FIGURE 9 VARIATION OF THE 100 SEC. CREEP CONTRACTION RATIO OF UNIAXIALLY REINFORCED MY 753 WITH ANGLE. $V_f = 0.40$.