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## ABSTRACT

This investigation was carried out to find the effect of gibberellic acid on fibre length and consequently the other gross and fine structural, and mechanical, properties of cotton fibres. In the course of investigations some general relationships were discovered which appear to apply to all cotton fibres whether treated or not.

Three cotton varieties were used: Samaru 26J and Rex, both Upland varieties, and Sea Island V.H.8 cotton. All three were grown in a greenhouse in Glasgow and Samaru 26J was also grown under normal field conditions in Nigeria.

Gibberellic acid treatment increased fibre mean length and decreased the coefficient of variation for Rex and Samaru 26J grown in Glasgow, but had no effect on Samaru 26J grown in Nigeria or on the Sea Island cotton. Its effect on the first two varieties is attributed to an increase in the length of the shorter fibres and an increase in the number of fibres in the longest fibre length groups. There was also an increase in the total number of fibres per seed in Rex.

Treatment with gibberellic acid also resulted in a decrease in fibre diameter, fibre weight and linear density, and maturity and fibre density. The number of convolutions per unit length and the average convolution angle were generally lower in the

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treated samples, this is attributed to the differences in maturity. The number of reversals per unit length was lower in the treated than in the control samples, this is attributed to the increase in the boll size.

Gibberellic acid treatment was found to regulate cellulose deposition. This resulted in fibres of different lengths having weight proportional to their length.

The effect of gibberellic acid treatment on crystallite and overall molecular orientation, comparing samples having equal lengths, was nil, which is not unexpected, since the spiral angle is highly correlated with fibre length.

The stress-strain curves were analyzed and the effect of the major weak places was removed to get the extrapolated "projected" stress-strain curves. Gibberellic acid treatment increased the projected extension to break, tensile strength, work of rupture, and stiffness of Sasaru 26J grown in Glasgow, Rex, and Sea Island, but these improvements were largely offset in practice because of the accompanying increase in major weak places, especially in Sea Island. On Sasaru 26J grown in Nigeria the effect on the projected mechanical properties was nil but the number of weak places increased and there was an overall deterioration.

Generally, the effect of gibberellic acid is dependent on variety and environment. It is more effective on short cottons

and under poor growing conditions than on long cottons and under optimum growing conditions.

The overall molecular orientation has been found to be dependent on both fibre length and fibre diameter. Therefore, a factor (radius/length) has been suggested to describe fibre dimensions. This factor gives high correlation with the overall molecular orientation.

The spiral angle of the successive cellulose layers of the secondary wall has been discussed. A hypothetical construction has been suggested in which the spiral angle decreases, level off, and finally may increase for the successive layers. This is supported by the results of molecular orientation, initial Young's modulus, and stiffness measurements on fibres having successive degrees of wall thickening.

It has been shown that the convolutions unfold as load is applied, thus they contribute substantially to fibre extensibility and work of rupture. This contribution varies from sample to sample. The remainder of fibre extension to break is suggested to be due to a partial unfolding of the spiral and to the extension of the amorphous regions located within the spiralling fibrils. The total theoretical extension to break resulting from fibre fine and gross structure is apparently much higher than the measured one.

THE INFLUENCE OF GIBBERELIC ACID  
ON GROWTH AND STRUCTURE  
OF COTTON FIBRES

by

MOHAMED EL-SAYED ABDEL-SALAM

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Gibberellic acid treatment was found to regulate cellulose deposition. This resulted in fibres of different lengths having weight proportional to their length.

The effect of gibberellic acid treatment on crystallite and overall molecular orientation, comparing samples having equal lengths, was nil, which is not unexpected, since the spiral angle is highly correlated with fibre length.

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It has been shown that the convolutions unfold as load is applied, thus they contribute substantially to fibre extensibility and work of rupture. This contribution varies from sample to sample. The remainder of fibre extension to break is suggested to be due to a partial unfolding of the spiral and to the extension of the amorphous regions located within the spiralling fibrils. The total theoretical extension to break resulting from fibre fine and gross structure is possibly much higher than the measured one.

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CHAPTER I.

INTRODUCTION AND LITERATURE SURVEY

## GENERAL INTRODUCTION

A fairly clear idea about the structure of the cotton fibre is of great importance to all those who are interested either in cotton breeding and raw cotton production, or in conversion of the raw fibres into finished end-products.

As early as half a century ago, Balls<sup>12-17</sup> pioneered the study of the structure and properties of cotton fibres, and since then numerous investigators have been pursuing this path to reveal the finest details of the cotton fibre structure, using several methods. The ordinary microscope and the polarizing microscope have been invaluable in revealing the gross and fine structure of the fibre, and recently the electronmicroscope has been found useful in investigating the surface characteristics and the fibrillar structure. X-ray diffraction techniques have been introduced in the last thirty years and have yielded invaluable information about the fine and molecular structure. Chemical methods have been used to study the fibre fine structure and mechanical tests have been carried out to gain information about the mechanical properties and thus to make deductions about fibre structure.

In all these investigations, cotton fibres have been investigated in their raw state, when, sometimes, the properties of fibres of different varieties or species, or fibres grown under different environmental conditions, have been compared; or they have been

investigated after being subjected to different chemical, physical, or mechanical treatments in order to modify the fibre structure. Hence information has been gained about the structure of the raw fibre and the changes in the structure caused by such treatments.

Recently, gibberellic acid, a plant hormone or growth regulator, has been found to accelerate the growth and increase the length of cells in plant structures. The cotton fibre is a single cell. If the length of the cell can be increased by appropriate treatment with gibberellic acid, other changes in the structure may follow and help in the understanding of the growth and structure of the cotton fibre.

#### GIBBERELLIC ACID

From 1926 onwards<sup>201</sup> a series of investigations were made in Japan into the biology of a fungus, *Gibberella fujikuroi*, which attacks rice, causing long, pale, spindly growth. Eventually in 1938 characteristic metabolic products called gibberellins were isolated from this fungus, and these were later shown to accelerate greatly the growth in length of seedling stems of a number of plant species when applied in lanolin paste.<sup>8</sup> These observations were later extended to a closely related product, gibberellic acid, which produces similar responses in the many species studied.<sup>86</sup> The most typical and striking plant response to treatment with gibberellins is stem elongation.<sup>201</sup> Despite some exceptions, cell

elongation in most cases was found to predominate as the result of application of gibberellins. However it is now apparent that gibberellins may markedly increase both the number and length of cells, depending on the nature of the plant material investigated, and the conditions under which the material is grown.<sup>166</sup> Another striking effect of the gibberellins is on certain dwarf plants, first reported by Brian and Hemming,<sup>35</sup> who found that a few micrograms of gibberellic acid applied to a leaflet would increase the growth rate of a dwarf pea plant to that of a tall variety.

In the last few years many workers have investigated the effect of gibberellic acid on the cotton plant for different objectives, using various doses, and different methods of application, and treating plants at different ages. Ergle<sup>66-68</sup> in an investigation of the effect of gibberellic acid on some characteristics of the cotton plant reported that the most pronounced effect of gibberellic acid was to increase main-stem length. This increase was directly related to the concentration of the gibberellic acid applied over a range of 1 - 100  $\mu$ g. doses. However, the highest rate reduced plant dry weight and the lowest one was ineffective. Clor,<sup>51</sup> and Bradford and Ewing<sup>34</sup> reported similar effect. Dransfield,<sup>64</sup> working upon a hirsutum variety in Nigeria, found that individual treatment of young bolls increased their retention but had no effect on yield or lint characteristics. Jackson<sup>117</sup> working upon a barbadense variety in the Sudan, found that spraying the plants

with gibberellic acid led to a marked increase in the rate of stem extension, like that found by Dransfield,<sup>64</sup> but it did not result in earlier flowering and also yield was generally reduced. Merritt<sup>139</sup> reported that research in the U.S.A. had demonstrated that three American varieties; Paymaster Stormrider, Acala 4.42, and Deltapine 15 would set more bolls when from 1 - 6 g. of gibberellic acid per acre was applied. The same varieties also have been induced to go on growing and producing bolls after the normal "cut out" time. Another fairly general effect of such applications is to increase fibre thickness. Furthermore the length of fibre has sometimes been increased by 0.04 to 0.25 inches. Walhood<sup>221,222</sup> demonstrated the dependence of the gibberellic acid effect on environmental conditions. Using two types of soil, heavy and light, he found that the relative increase in plant height was greater on the light soil and was also proportional to the number of applications of gibberellic acid, but the increase in yield following application of gibberellic acid was obtained only on plants growing on lighter soils, and this increase in yield was mainly due to the extension of the growing season. Treatment with gibberellic acid did not affect fibre properties.

## COTTON FIBRE GROWTH AND STRUCTURE

### Cotton Fibre Growth

The most complete account of the origin and growth of cotton fibres has been given in the comprehensive studies of Balls<sup>12-17</sup> who worked upon Egyptian cotton ( *G. barbadense* ), and whose findings were confirmed by Anderson and Kerr<sup>3</sup> who worked upon American Upland cotton ( *G. hirsutum* ). As a result of this work, fibre growth is known to occur in two fairly distinct stages; the first growth stage corresponds to the fibre elongation and the primary wall formation, and the second growth stage corresponds to the secondary wall formation.

It is now agreed that the cotton fibre cells arise from the epidermis of the cotton seed coat.<sup>1,69,93</sup> Each cotton fibre originates as an outgrowth from a single epidermal cell of the seed-coat. The first evidence of the formation of the cotton fibre is the appearance on the day of flowering of a slight swelling of the outer wall of these epidermal cells. The swellings elongate rapidly and on the day after flowering have already produced delicate tubular outgrowths, the young fibre cells. The diameter of the mature cotton fibre is reached soon after it originates, but elongation of the cell continues for a period of 15 - 20 days,<sup>1</sup> when it ceases abruptly. The period of elongation seems to be determined by species as well as environmental factors. The curve

for the growth rate during the period of cell elongation, is the typical sigmoid shape, the steepest part of which corresponds to the growth during the 8 - 18 days after flowering.<sup>1</sup> Balls,<sup>12,15</sup> and Barritt<sup>21</sup> believed that the primordia all appeared together on the day of flowering. Subsequent work, however, has proved that the differentiation of the fibres is a continuous process which may take 3 to 6 days,<sup>26</sup> a differential growth must be the basis for a physiological explanation of the fibre arrays that are obtained from single seeds.<sup>3,9,88,89,124,187,189,212</sup> Koshal and Ahmed,<sup>123</sup> and Berkley<sup>26</sup> found that the mean length for fibres at the chalazal end of the seed was greater than it was for fibres at the micropylar end. The fact that the fibres at the chalazal end are often several days older than those at the micropylar end may explain this difference in length.

At the end of the first growth stage, when the elongation ceases abruptly, another period of cellulose deposition, in successive layers on the inner surface of the primary wall, takes place and continues until a few days before the boll opens. Under certain conditions, the deposition of the secondary wall may be completed on, or about, the thirtieth day after flowering, or, in other circumstances, it may continue up to about the seventy-eighth day after flowering.<sup>78</sup> The fact that the rate of cellulose deposition falls off about the thirty-fifth day after flowering has been demonstrated by several authors.<sup>25,192,220</sup> Cellulose deposition

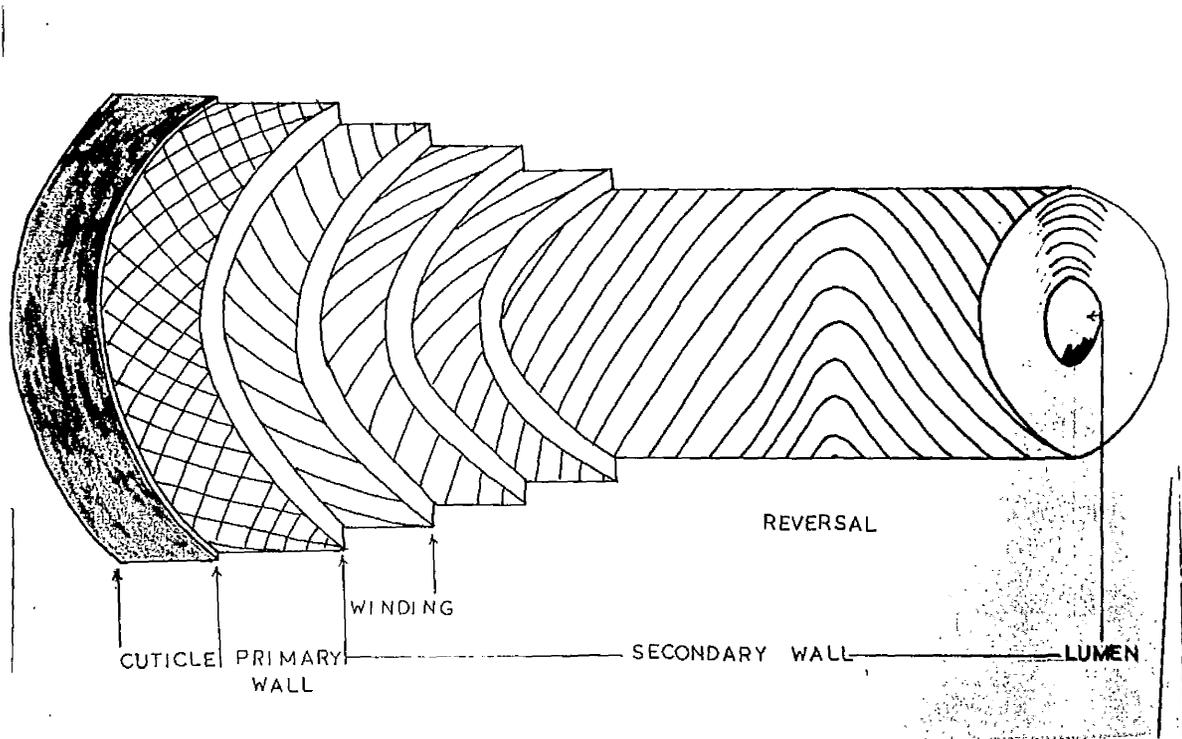


FIGURE 1

does not fill completely the cell cavity, this results in the presence of the lumen in almost all the fibres.

During the period of growth, the fibre retains its cylindrical shape and, because of the limited size of the boll and the crowded conditions inside it, the fibre usually does not grow in a straight line, but changes its direction back and forth many times. As soon as the boll opens, the fibre dries and as a result it loses its circular cross-section and convolutes upon itself many times.

### Cotton Fibre Structure

A typical mature cotton fibre consists of a primary wall (covered with the cuticle), a secondary wall, and finally a lumen (Fig. 1).

#### (a) The primary wall and the cuticle

In the cotton fibre, the primary wall, together with the cuticle, is the only covering for the protoplasm during the first period of cell elongation and development. The cuticle is very thin, considerably less than  $0.25 \mu$  thick.<sup>165</sup> It has been defined as a semi-elastic, retaining membrane which conforms to all changes in the size of the fibre, but does not expand past the predetermined maximum set when the fibre was growing and in a state of turgor.<sup>161</sup> The cuticle is extremely tightly moulded to the primary wall, and although it has a rough surface<sup>63,207</sup> it remains unbroken, except over the grossest fibre faults. The cuticle consists of wax,<sup>3,125,161</sup> a large proportion of pectic material,<sup>3,149</sup> and some incrusting

mineral matter.

The primary wall may be defined as the limiting membrane of a plant cell.<sup>119,171</sup> The fact that it is capable of extension in length and breadth, and allows an increase in the size of the cell,<sup>11</sup> distinguishes it from the secondary wall, which is neither largely extensible nor capable of increasing in area.

It is now agreed that the primary wall generally consists of pectic compounds together with some cellulose,<sup>3,4,11,82,171,172</sup> and that the latter may fail to give the typical cellulose reactions until certain wax-like substances have been removed.

Balls<sup>14</sup> was the first to discover the presence of two opposing systems of fine spirally-wound threads of cellulose in the walls of 10-day-old fibres. The spiral threads were found to make an angle of approximately  $70^{\circ}$  with the long axis of the fibre. Optical microscopic,<sup>207</sup> electronmicroscopic,<sup>153</sup> and X-ray<sup>25</sup> studies have revealed a third and transverse system. Anderson and Kerr<sup>3</sup> concluded that the cellulose micelles in the primary wall are grouped into delicate anastomosing threads which have at least two systems of orientation: (1) a flat right-hand spiral, (2) a flat left-hand spiral, and possibly also (3) a transverse system. All the three systems seem to be uniform over the entire surface of the fibre cell. No evidence has been found that the direction of the spirals changes so as to produce reversals as are common in the secondary wall.

The technological significance of the primary wall lies in the fact that it is, with the cuticle, the first morphological component of the cotton fibre to come in physical contact with the processing environment. Although the primary wall comprises only a small part of the mature cotton fibre, it nevertheless greatly influences dyeing, finishing, wettability, reactivity, attack by micro-organisms, and other fibre properties.<sup>42,197-199,207,208</sup>

(b) The secondary wall

The secondary wall is the deposit of cellulose which, for the most part, is laid down after the cell has reached its mature size, finally stabilizing the size and shape of the cell. The greater part of the secondary wall is deposited, at the expense of the cell lumen,<sup>121</sup> between approximately the eighteenth and the thirty-fifth day after flowering.<sup>25,112</sup> The secondary wall consists of many tiny threads, called "fibrils", laid side by side to form the ring-shaped lamellae. The fibrils constituting each lamella do not lie parallel to the central axis of the fibre but follow a helical course, screw fashion, around the lumen. Under the microscope, striations arising from this criss-cross organization of fibrils have been observed by many investigators,<sup>12,54,62,71</sup> particularly when the fibre is swollen. The earlier suggestion<sup>12</sup> that the fibrils of a lamella may abruptly change direction and adopt the reverse spiral path has been confirmed.<sup>3</sup>

(1) The growth rings

The examination of fibres swollen in cuprammonium hydroxide reveals a layer pattern in the secondary wall. In the longitudinal view these layers look like strips running parallel to the long axis of the fibre, in cross-sectional view they look like concentric circles or rings. It is curious that although these rings are large enough to be within the range of visibility, they are not seen in untreated sections.<sup>62</sup> Balls<sup>13,15</sup> believed that the secondary wall thickening is a stepwise process and that the successive layers of cellulose in a fibre cross-section are daily growth rings, which represent the rapid synthesis and deposition of cellulose during <sup>?</sup>sunlight hours, and that the number of lamellae or growth rings corresponds with the number of days during which the secondary wall has been laid down. Kerr<sup>120</sup> stated that the diurnal temperature fluctuations are probably responsible in large part for growth-ring formation.

There seems to be wide variation in the number and width of the lamellae reported by different authors.<sup>3,13,22,112,120</sup>

(2) The secondary wall differentiation

Anderson and Kerr<sup>3</sup> reported that, in studying the structure of the cotton fibre, differentiation of the secondary wall into two or three layers on the basis of cellulose orientation, similar to that found in wood fibres,<sup>119</sup> did not seem to be possible.

All parts of the secondary wall show a pronounced spiral structure, and for this reason layering cannot be distinguished in cross-sections by variations in birefringence. However, following Hock and co-workers,<sup>112</sup> Rollins,<sup>183</sup> Berkley,<sup>26</sup> Bigler,<sup>32</sup> Levine,<sup>125</sup> and Herzog and Berkley,<sup>111</sup> it is possible to consider the structure of the secondary wall as consisting of three parts: (1) an outer layer, (2) central layers, and (3) the innermost layer.

(1) The outer layer

Hock and co-workers<sup>112</sup> found that when depectinized fibres were swollen in cuprammonium hydroxide, a thin outer layer was present which possessed a helical structure with reversals along the length of the fibre. When this outer layer possessed an ( S ) spiral, the main part of the secondary wall possessed a ( Z ) spiral and vice versa. Hock and co-workers<sup>112</sup> identified this outer layer as the outermost part of the secondary wall and called it the "winding". Anderson and Kerr<sup>3</sup> had previously presented evidence that the outer-most layer of the secondary wall may be distinguished at times from the succeeding layers during cell wall development. They also found that it appeared suddenly about the sixteenth day after flowering; it completely covered the inner surface of the primary wall, and consisted of anastomosing strands which wound in a steep spiral and made an angle of 20 to 30° with the long axis of the fibre. Many reversals were seen in the spiral direction.

(2) The central layers

When of average thickness, these constitute about 90 % of the weight of the fibre. The fibrils composing the central layers are finer than those of the winding.<sup>112,183</sup>

Orr and collaborators<sup>160</sup> reported that while all layers of the secondary wall, except that of the winding, spiral in the same direction, the angle of spirality for the different layers is unknown. There are two possibilities; the first is that all these layers spiral at equal angles, and the second is that these layers have equal spiral pitch so that they spiral at different angles with the inner layers being of steeper angles to the fibre axis. They concluded that neither of these two assumptions gives an adequate explanation of fibre strength, and that some compromise between these two hypothetical constructions would lead to a stronger fibre than either alone. Berkley,<sup>25</sup> who studied cellulose orientation during cell wall development by means of the X-ray diffraction method, reported that the secondary wall pattern, changed gradually with cellulose deposition for the first few days, showing a more or less progressive improvement in the orientation of the successive layers of cellulose in relation to the fibre axis. However, little or no improvement in the orientation, as shown by the X-ray pattern, was observed after about the sixth day from the beginning of cellulose deposition in the secondary wall.

### (3) The innermost layer

There is some evidence that the innermost layer, next to the cell lumen, of the secondary wall may differ from the main part of the secondary wall in the steepness of its spiral orientation to the fibre axis.<sup>111,125</sup>

#### The submicroscopic or fine structure of the secondary wall

Our present knowledge regarding the submicroscopic or fine structure of cellulose has been contributed by a large number of workers over a period of many years. Detailed surveys of most of this work have been published by several authors.<sup>99,196</sup>

The evidence obtained<sup>80,97a</sup> by chemical methods established that cellulose is made up very largely of D-glucopyranose units linked together through  $1\beta-4$  linkages. The crystallinity of cellulose was first confirmed by Nishikawa and Ono<sup>150,151</sup> who showed that the X-ray diagram consisted of definite diffraction rings, and the first unit cell dimensions were calculated by Polanyi.<sup>168</sup> A series of investigations by Meyer and co-workers<sup>128,140,141</sup> led to the postulation of a monoclinic unit cell with axes:  $a = 8.35 \text{ \AA}$ ,  $b = 10.3 \text{ \AA}$  (fibre period),  $c = 7.9 \text{ \AA}$ , and  $\beta = 84^\circ$ . Further information regarding the nature of forces which hold the lattice structure together has been reviewed by Mark.<sup>129</sup> This unit cell has been subjected to many criticisms,<sup>227</sup> and recently Liang and Marchessault,<sup>126</sup> from infra-red evidence, have modified this unit cell and suggested a different hydrogen bonding system.

In a cellulosic fibre the three dimensional unit cell arrangement is repeated in all directions to build up a crystalline region. The dimensions of these crystalline regions or crystallites can be calculated by the examination of the breadth of the X-ray reflections,<sup>39</sup> or by the study of the distribution of X-rays diffracted at very small angles.<sup>101,110,130a</sup> The percentage crystallinity of cellulose has been the subject of many investigations. Hermans,<sup>100</sup> from X-ray evidence, has reported that 70 % of cellulose in cotton fibre is crystalline while the remainder is amorphous. Several theories have been put forth to explain the relation between the crystalline and noncrystalline states. A comprehensive survey has been given by Hearle.<sup>96</sup>

The existence of a well organized fibrillar fine structure has been thoroughly established. The size of these fibrils has not been established with any certainty. Various estimates<sup>10,22</sup> of diameter ranging from a probable value of  $1\mu$  down to  $0.25\mu$  have been discussed in some detail. They are extremely long in comparison with their widths. Frey-Wyssling<sup>83</sup> has proposed that the microfibrils<sup>(150-250A)</sup> which can be detected in native cellulose with the electronmicroscope and can be further disintegrated by several means, are aggregates of crystalline elementary fibrils (or micelle strands)<sup>(70-90A)</sup> connected together with paracrystalline cellulose. Hearle<sup>94,95</sup> assumed that the fibrils constitute the crystalline proportion of cellulose. These crystalline fibrils are embedded in (and are molecularly continuous with) a matrix of noncrystalline regions. The orientation of the molecules is along the fibrils.

## The physical methods of investigating the orientation

Several methods have been devised for measuring the orientation of cellulose chains and crystallites in cotton fibres. Outstanding of these methods are the X-ray and optical methods.

### (a) The polarized light method

The optical methods of measuring orientation use the phenomena of dichroism or polarized fluorescence described by Morey,<sup>142</sup> or the refractive index in plane polarized light described by Preston.<sup>170</sup> The latter is the most widely used method.

The polymer molecules in both the amorphous and crystalline regions of a fibre usually show a tendency to orient with their long axes *arranged about* helically <sup>about</sup> the fibre axis but show no preferential orientation of either of the other two perpendicular axes. The effect of this lack of order in the lateral direction leads to an averaging of the optical properties perpendicular to the fibre axis and thus the specimen behaves optically like a uniaxial crystal.<sup>73</sup> therefore the refractive indices for light polarized parallel and perpendicular to the fibre axis will in general be different. The difference between the higher and the lower refractive indices, with light vibrating parallel and perpendicular to the fibre axis respectively, which is the birefringence, can, by comparing it with the birefringence of an ideal fibre, be taken as a measure of its orientation.

The birefringence can be measured in two ways, the first

one is by measuring the difference in optical path between polarized light vibrating parallel and perpendicular to the fibre axis. This is usually done by interposing in the light path a compensator<sup>91</sup> of the Babinet or Berek type. This method measures the average birefringence through the fibre, but has a severe limitation for use with cotton fibres in that the thickness of the fibre must be known. Obviously, this can only be measured with any accuracy if the fibre has a regular cross-section and is not convoluted like a cotton fibre. However, Orr<sup>160</sup> was able to use a Berek retardation compensator for birefringence measurements on cotton fibres. The diameter of a fibre in the direction of retardation was obtained with a slide equipped with means for turning the fibre  $90^{\circ}$  on its axis. Only mature fibres are measured accurately by this method.

The second method of measuring the birefringence is to determine the two refractive indices separately. This can be done by immersing the fibre in a liquid of approximately the same refractive index as the one it is desired to measure and then changing the refractive index of the immersion liquid either by altering its temperature, or by changing the wavelength of the light used until the edge of the fibre is no longer visible. The method is known as the Becke line method<sup>23</sup> because of the bright fringe or line of light that is observed near the edge of the fibre when the refractive indices are unequal. When the microscope objective is raised the bright fringe moves towards the medium of higher refractive

index, thus indicating whether the refractive index of the immersion liquid has to be increased or decreased to match that of the fibre. This method is usually assumed to measure the refractive index near the surface of the fibre.

Because it is easy to alter the composition of the liquid, many workers choose this method rather than altering the temperature or the wavelength of the light. It is usual to prepare a series of liquids providing a step-like increase in refractive index. In order to cover enough refractive index range, immersion liquids usually consist of a mixture of two stable and non-volatile liquids that do not affect the fibre. For cellulose, Hermans<sup>97</sup> recommended mixtures of butyl stearate (  $n=1.445$  ) and tricresyl phosphate (  $n=1.558$  ). Faust<sup>73</sup> has found liquid paraffin (  $n=1.481$  ) and 1-bromonaphthalene (  $n=1.659$  ) suitable for nylon. Meredith<sup>134</sup> used these two liquids for cotton: they are miscible, inert to each other and to the fibre, non-volatile and stable.<sup>79</sup> Meredith<sup>134</sup> has stated that the mixture of these two liquids has a high temperature coefficient of about  $-0.0004$  per  $^{\circ}\text{C}$ . so that it is important to control and measure their temperature during the determination of the refractive index. Hermans<sup>99</sup> pointed out that the liquid and fibre should be conditioned in the same atmosphere so as to minimize any re-distribution of moisture between them.

Faust<sup>73</sup> has recently discussed the application of the Becke line method to fibres and concluded that the accuracy of the index

measurement is a function of the fibre shape and dimensions and of the degree of defocussing of the microscope. Under optimum conditions a path difference of  $\lambda/100$  can be detected, and the index of a  $10\ \mu$  diameter fibre can therefore be determined to better than  $\pm 0.0005$ .

Becke line measurements on cotton<sup>81,134</sup> have always given refractive indices consistent with the structure of the secondary wall and not that of the outer primary wall. Different explanations have been offered for this behaviour.<sup>90,173</sup> The difficulties are resolved as soon as it is realized that the index of the primary wall, which is only about  $0.2\ \mu$  thick, will be found only if the defocussing is about  $0.2\ \mu$ .<sup>72</sup>

The orientation can be calculated from the birefringence measurements in two ways; the orientation factor and the average angle of orientation proposed by Hermans,<sup>99</sup> and the orientation angle method used by Meredith.<sup>134</sup>

Meredith,<sup>134</sup> reported, from work on 26 varieties ranging from the finest to the coarsest cottons, that the higher refractive index varied from 1.573 for short coarse cottons to 1.581 for long fine cottons, whilst the lower refractive index was approximately constant at 1.531. The corresponding angles of the spiral fibrils were calculated to range from  $27^\circ$  for fine Sea Island cottons to  $35^\circ$  for coarse Indian cottons.

(b) The X-ray diffraction method

Of the various experimental methods which have been employed for measuring orientation, the X-ray method has been the most widely used and is in many ways the least controversial. It distinguishes most clearly between the orientation of the amorphous and crystalline regions since it gives the orientation of the crystalline regions and most important, it is the only one which is capable of giving a distribution of orientations.

Clark<sup>49</sup> described qualitatively the variation in crystallite orientation which occurs in cotton fibres, and later Sisson and Clark<sup>190</sup> developed an empirical X-ray method for quantitative comparison of orientation in cotton fibres. They obtained the diffraction patterns on plates, and, using a microdensitometer, derived intensity distribution curves from the fibre patterns by measuring the optical density around the ( 002 ) diffraction arc. This method was based upon the assumption that the distribution of intensity round the ( 002 ) diffraction ring was proportional to the distribution of the crystallites around the pencil of the X-rays. Later work by Tsien<sup>214</sup> verified the selection of the ( 002 ) plane as that most practicable for the purpose, although theoretically not entirely appropriate. Sisson<sup>193</sup> gave a detailed description of the X-ray method of comparing crystallite orientation in cellulose fibres, and suggested three ways of calculating a measure of

orientation of which the most sensitive was the angle of 40 per cent. maximum density. This was the method used by Berkley and Woodyards<sup>24,29</sup> for raw cotton and they have discussed the corrections which have to be applied in order to get an accurate estimate of orientation. Meredith<sup>135</sup> developed a method which employed a calibration strip on each X-ray film, thus allowing transmitted intensity readings to be converted directly into the corresponding X-ray intensities, independent of the opacity of the film or of the particular characteristics of the microdensitometer used. This method takes more time but requires less rigid standardization of experimental conditions and gives angles which are highly correlated with those given by Berkley and Woodyard.<sup>24</sup> Meredith<sup>135</sup> also used a method which gave the average angle of inclination to the fibre axis of the chain molecules in the crystalline regions; this method has been used by Hermans and co-workers<sup>99</sup> mainly for viscose rayon, and takes into account the distribution of X-ray intensity around the ( 002 ) arc instead of depending on the intensity at an arbitrary point on the arc. Meredith<sup>135</sup> found that this average orientation angle did not correlate so well with the 40 per cent. X-ray angle, due to differences in the shape of the X-ray intensity distribution curves for the ( 002 ) arcs of different cottons. Creely and co-workers<sup>59,185</sup> devised a technique for the quantitative determination of the degree of the crystallite

orientation of cellulose in cotton and related cellulosic fibres, using the X-ray diffraction spectrometer with potentiometer recording and a rotating specimen mount. They used, as a measure of orientation, the angular displacement from the point of maximum intensity on the ( 002 ) diffraction arc to the point where the intensity is 50 per cent. of the maximum. This 50 per cent. X-ray angle was found to correlate well with the 40 per cent. X-ray angle used by Berkley and Woodyard.<sup>24</sup>

Meredith<sup>136</sup> reported that the variation in the average convolution angle within a variety affected the orientation as measured by the X-rays but had little influence on the strength and that this observation may explain some of the lack of correlation between X-ray orientation and tensile strength. He also stated that, if the average convolution angle was subtracted from the average orientation angle measured by X-rays, the difference was roughly constant. Since these two angles are not directly comparable, this procedure is not strictly valid, but it suggests that the spiral angle of the crystallites in all cottons in the original unconvoluted fibre may be the same. This has been confirmed by Betrabet and co-workers.<sup>30</sup>

Sisson<sup>194,195</sup> in an extensive study on the crystallite orientation in cellulosic fibres and membranes, and of the relationship between this parameter and tensile strength, suggested

a method which took into account, qualitatively, the spiral structure of the cotton cellulose chains around the fibre axis and the distribution of crystallites within the spiraling strands in explaining the shape of the diffraction arcs. He has shown that the ( 002 ) arcs displayed by fibres with a spiral structure can be explained by assuming two equal crystallite distributions separated by twice the spiral angle. DeLuca and Orr<sup>60,61</sup> made use of this method and developed a method in which this distribution of crystallites was assumed to be Gaussian and the experimental arc would be regenerated theoretically and analyzed to give average and projected values of crystallite orientation and spiral angles. They found that the crystallite orientation and spiral angles of several native cottons that represent a wide range of physical properties and ( 002 ) diffraction arc sizes, did not vary greatly.

Some possibilities and limitations of the X-ray method for estimating the strength of raw cotton have been discussed by Conrad and Berkley.<sup>56</sup> The advantages claimed for the X-ray method are speed, general applicability to all staple lengths and all strengths, pattern little affected by atmospheric conditions, and test specimen preserved, whilst the disadvantages include less precision, a calibration by imperfect methods and the possibility of no modification of the X-ray diagram as a result of chemical degradation or attack by micro-organisms. Meredith<sup>136</sup> reported a correlation ranging between 0.77 and 0.84 between tensile strength

of single fibres and of flat bundles of fibres and the orientation of the crystallites as measured by X-ray. Several investigators have also reported high degrees of correlation.<sup>25,27,181,202,203</sup>

### The structural reversals

The spiral fibrils, composing the first and subsequent layers of the secondary wall show characteristic reversals. A structural reversal occurs at those points where the direction of rotation of the cellulose fibrils about the fibre axis changes from a left-hand spiral to a right-hand spiral, or vice versa. Anderson<sup>3</sup> has stated that two general types of reversals may be observed: (a) the commonest type of reversal is one in which the spiral strands simply change their direction by bending around in the form of an arc, and (b) the second type is one in which one set of spiral strands ends and a second system of spiral strands running in the opposite direction begins. The ends of the threadlike strands of the two systems overlap at the place of the reversal. Various hypotheses for the cause of reversals in cotton fibres have been proposed. After devoting much attention to them, Balls and Hancock<sup>17</sup> came to the conclusion that the two important factors in determining the presence of reversals were the length of the adult cell, and the duration of the growth in length. Berkley,<sup>26</sup> and Wakeham<sup>218</sup> have proposed the crowded conditions in the cotton boll as the cause for reversals. As the fibre grows in length it does not grow

in a straight line, instead, it is crimped by folding back and forth in the boll. Certain of these bends are gradual, others are very sharp. At the point of bend the outer wall is longer than the inner wall in relation to the bend. The cellulose in the secondary wall is deposited while the fibre is bent or crimped. The spiral fibrils appear to be interrupted by the sharp bends in the fibre, causing reversals. Wakeham<sup>218</sup> has stated that if the crowded condition in the boll is a real cause for reversals, then the reversal frequency should be related to the boll size. This has been confirmed, since he found that fibres from large bolls possessed less reversals per unit length than fibres from small bolls.

Wakeham and Spicer<sup>217</sup> have reported, from evidence obtained from moisture and hydrolysis effect on the strength of cotton fibres at and between reversals, that the cellulose in the regions of reversals is more highly crystalline than the cellulose between the reversals. Wakeham and co-workers<sup>219</sup> found that X-ray diffraction patterns taken for single fibres at and between reversals showed that the cellulose is more highly oriented at the reversals than that in the remainder of the fibre. The same interpretation has been reached by Orr<sup>160</sup> from optical evidence.

The number of reversals per unit length varies within wide limits.<sup>3,14</sup> Although reversals may be found along the entire length of an individual fibre, the frequency of reversals along the fibre length differs.<sup>218</sup>

### The convolutions

It is well known that normally thickened cotton fibres twist when dry, the twists, or convolutions, appear when the cell loses "water of construction". The process is irreversible and plasmolysis is not sufficient to bring it about.<sup>3,14,20,112,153</sup>

The original suggestion put forward by Balls,<sup>14</sup> that the pattern and structure of the convolutions is determined by the internal spiral structure of the wall, and that the changes in the direction of the convolutions are correlated with the reversal points in the spiral fibrils of the wall, has been confirmed and elaborated by subsequent research. Hock and co-workers<sup>112</sup> have correlated the pattern of the convolutions and the changes in their direction with the spiral structure and reversal points of one particular part of the secondary wall, the first layer or the winding. Denham<sup>62</sup> has suggested spiral inequalities among the fibrils to be the cause of fibre convolutions. According to Osborne,<sup>161</sup> the mechanism of formation of convolutions is probably to be found in the drying out of the cell walls, the structure of the fibre being such that greater shrinkage occurs in the direction perpendicular to the fibrils than in the direction parallel with them; and since the fibrils are spirally arranged, collapse and shrinkage of the wall causes them to form mutual angles with each other and the fibre axis, thus producing a twisting movement. Clegg and Harland<sup>46</sup> have found that the number of convolutions in any portion of a single cotton

fibre depends upon the ratio of ribbon width to wall thickness and that the highest mean number of convolutions is shown when the ratio has the values between 3.4 and 3.6. Very thin-walled fibres do not convolute until swollen in strong caustic soda solution; similarly very thick-walled fibres convolute little if at all since they do not collapse and the greatest number of convolutions, therefore, occurs in fibres with walls of intermediate thickness.<sup>26</sup>

The number of convolutions in different cotton varieties varies widely. Bowman<sup>33</sup> has given estimates of average number of convolutions per inch for different cottons; Sea Island possesses the highest number of convolutions, about 300 convolutions per inch while the Indian cottons possess the lowest number, about 150 convolutions per inch. Other workers<sup>1,61</sup> have found even wider ranges.

#### FIBRE LENGTH

Fibre length is the most obvious feature of a cotton fibre and has always attracted considerable attention because staple length is associated with not only spinning performance but also yarn properties and characteristics, e.g. spinning limit, yarn appearance, evenness, and strength. In the early days of increasing demand of a rapidly growing cotton industry, fibre length was the paramount factor in evaluating different cottons, and improvement concentrated on this property. However it has gradually been realised that the other fibre properties, such as fibre strength, fineness, and

maturity are also important.

In any one sample of cotton,<sup>127</sup> or in any one variety, there is, however, a large variation in the length of individual fibres. This variation arises mainly from differences encountered between fibres from the same seed, and is increased by additional differences between seeds in the same boll, between bolls from the same plant, and between plants.

Webb and Richardson<sup>223</sup> reported from an analysis of 766 cottons that larger coefficients of variation in length were associated with a general deterioration in yarn properties. Tallant and co-workers<sup>204-206</sup> found that a 1 % increase in fibres shorter than 3/8 inch caused a strength loss in yarns of somewhat more than 1 % , and concluded that increases in short fibres content resulted in: 1. decreased yarn strength, elongation, appearance grade, and evenness, 2. increased difficulty in spinning at medium yarn numbers and low twists, and 3. increased twist in the roving for constant hardness. Kohler<sup>122</sup> has indicated that the "length of slippage" is approximately 8 mm. or slightly more, i.e. fibres 8 mm or less are likely to slip rather than break when a yarn ruptures.

#### FIBRE DIAMETER

In the green boll, while the fibres are growing they are cylindrical, but as they dry, they shrink and the cylinder tends to collapse into a flat ribbon. Balls<sup>12</sup> stated that the diameter

of the uncollapsed fibre was reached by the fibre during the early few days of fibre initiation and that this property was controlled by genetical factors. Clegg<sup>48</sup> pointed out that, for a given type of cotton, the original diameter of the living fibre varied within relatively narrow limits but during maturation cell walls varied considerably in degree of thickening and this gave rise to the extreme variability in both size and shape often observed in cotton fibres.

As a result of the dependence of both the shape of the ribbon and the cross-section on the degree of wall thickening and the shrinkage which accompanies fibre drying, the diameter of the fibre can be measured only from the uncollapsed fibres. Moore,<sup>143</sup> and others<sup>50</sup> have found a high correlation between the diameter of the mercerized fibres and that of uncollapsed fibres, but in the mercerized state the diameter is equal to only 64 % of that of the uncollapsed fibre and this is due to the shrinkage which accompanies fibre mercerization. Yoseif<sup>229</sup> measured fibre diameter from fibres swollen in 18 % sodium hydroxide solution. He reported a correlation of 0.99 between fibre diameter in the swollen state and that of the uncollapsed fibres. However, fibre diameter as measured by this method is still 8 % smaller than that of the uncollapsed fibres.

Christidis<sup>44</sup> reported that fibre diameter, as measured

from uncollapsed fibres, was the most important factor in classifying cottons into fine and coarse. Yoseif<sup>228</sup> demonstrated the importance of fibre diameter which he used to calculate fibre density and this he found to be highly correlated with yarn strength.

#### FIBRE LINEAR DENSITY

The linear density is the weight per unit length and it provides a useful general way of describing the fineness or coarseness of cotton fibres. The linear density of cotton fibres is governed by variety, and within varieties it is affected by fibre diameter and the actual amount of cellulose present.

The linear density can be measured by several methods. The early method of the gravimetric fineness test, and one which is still commonly used, involves cutting known lengths from the middles of parallelized fibres, counting out a suitable number of those lengths, and weighing them.<sup>12,38</sup> This method is vulnerable to some criticisms because of the considerable variation along the fibre length.<sup>15,113,162,212</sup> The second gravimetric method is by weighing the whole fibres of known length and gives a mean value of the linear density along the whole fibre.<sup>6</sup> The difference between these two methods has been discussed by several authors.<sup>145,147</sup>

The linear density of the different fibre length groups of a cotton sample was found by Iyengar and Turner<sup>115,116</sup> to increase as the length of the fibre decreased and to be highest

for the shortest length group. Nanjundayya and Ahmed<sup>146</sup> confirmed this finding and added that the variability in linear density among fibre length groups was different in different types of cotton. They found that the rate of increase of linear density with length was greater with the shorter than with the longer cottons and in two long cottons they found very nearly constant linear density for different lengths. Shah<sup>188</sup> reported that shorter fibres of a cotton sample were coarser than the longer fibres, and Chytiris<sup>45</sup> confirmed that this was true for the same seed, strain or variety. He also found that long and fine cottons were much more uniform in their fibre weight distribution than the shorter and coarser ones but that the natural fibre fineness distribution was modified and even changed through ginning, mixing, and other processes that broke some of the longer and finer fibres. In contrast to these findings, Fiori<sup>75</sup> found that the short fibres were finer than the long ones, and Sands and co-workers,<sup>184</sup> from work on 42 varieties, confirmed his findings. Morlier<sup>144</sup> measured the weight fineness of the centre sections only of fibres found that it varied with length and with variety; in general it increased with fibre length to a maximum value at the length slightly shorter than the modal length group, and then decreased. Pillay<sup>167</sup> found that the linear density of extreme length groups was generally lower than those of the middle groups, the longest length group being the finest.

The explanation of these contradictory findings seems to lie in differences of technique<sup>41,76</sup> or random variations in sampling and testing, but there may also have been real changes over the decades in the linear-density-length distribution as suggested by Fiori.<sup>76</sup>

The fineness of fibres determines to a large extent the processing difficulties which are likely to be encountered, the spinning performance and the strength of the yarn, the nep count and the appearance of the finished material. Fiori and Brown<sup>74</sup> found that, with other variables remaining constant, cotton with low linear density would produce substantially greater strength and greater number of neps than would a cotton with a high linear density. Whilst Kapida<sup>118</sup> concluded that fibre strength was an important factor of yarn strength, Turner<sup>213</sup> found that fibre length, first, and fineness, next, contributed more than fibre strength. Balls<sup>15</sup> postulated that the effect of length was largely exaggerated and that long-fibred cottons would be spun into fine yarn number not because of their length but because of their fineness. Other investigators<sup>18,19,29,122,148,182,215</sup> have presented the significant fibre properties in a different order of importance.

TYPICAL STRESS STRAIN CURVE  
OF COTTON FIBRES

STRESS

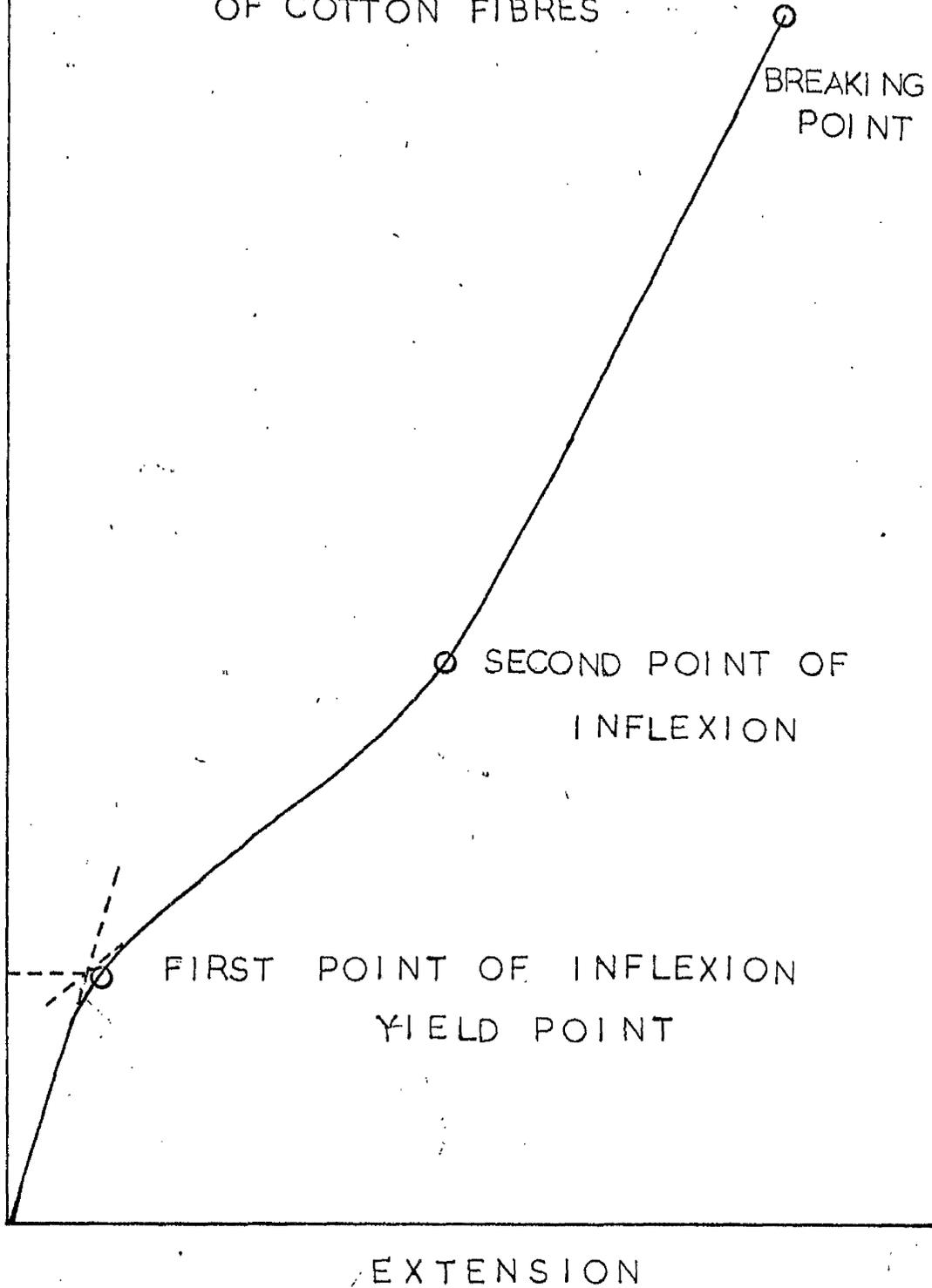


FIGURE 2.

## COTTON FIBRE MECHANICAL PROPERTIES

The resistance of a single cotton fibre to extension is the ultimate factor in the tensile behaviour of all cotton textiles, and is also of interest in the study of fibre structure.

The stress-strain curves give a greater amount of information on the behaviour of fibres under load than the results of a simpler tensile test for breaking load and extension, but are more laborious to obtain. Most of the stress-strain curves (Fig. 2) show an initial part where the stress is proportional to the strain and for this part of the curve it has been possible to measure the "initial Young's modulus" which is the ratio of stress to strain. This modulus has been found to be highly correlated with molecular orientation.<sup>133</sup> Certain fibres show a sudden increase in extension for a small increase in stress after this initial part. The place where this occurs is sometimes called the yield point although the effect usually takes place over a region of extension. The significance of the yield point is that if a fibre is stretched beyond this point it will not show complete immediate recovery, although it may creep back slowly to its original length. A second point of inflexion\* often occurs as the point of rupture is approached, more particularly if there are no local weak places and therefore rupture does not occur early. The curve typically swings upwards in this final stage, increasing increments of

\*The term "point of inflexion" is commonly used for this section of the curve<sup>127</sup> although it does not agree with the mathematical definition.

stress have to be applied to produce equal increments of extension.<sup>127</sup> The amount of work required to rupture the fibre, which is a measure of the fibre ability to absorb energy, i.e. to withstand a sudden shock, can be calculated from the stress-strain curve. This is represented by the area enclosed by the stress-strain curve and the strain axis,<sup>134</sup> and would be half the product of the breaking load and the breaking extension if Hooke's law was obeyed. The stress-strain curves for cotton depart considerably from a linear relation but the idea of work of rupture as one half the product of load and extension at break can be retained by introducing a "work factor" which is the ratio of the actual work of rupture to the product of load and extension at break: for a material obeying Hooke's law right up to the breaking point, the work factor is 0.5.

The various factors determining the strength and other mechanical properties of cotton and other textile fibres may be divided into two broad categories: firstly the structure of the fibre, both fine and gross, and secondly variations in test conditions, including those associated with the rate of loading, the length of specimen subject to test, and the nature of testing atmosphere.

Mann and Peirce<sup>131</sup> pointed out that the breaking load of a cotton fibre depended not only on the strength of the fibre but

also on the time allowed for the fibre to stretch to the breaking point. They found that the breaking load increased with the rate of loading, reached a maximum, and then became independent of loading rate. The most general expression of the effect is a linear relation between the breaking load and the logarithm of the time of break, which can be directly related to other effects of elastic imperfection.

Moisture has a singular effect on the strength and extension of cotton fibres. A number of investigations have been undertaken by various workers on the effect of relative humidity on the strength and extension of cotton textiles; on single fibres,<sup>36,37,114,130</sup> bundles,<sup>40,169</sup> and yarns.<sup>84,92,138,164,211</sup> Brown<sup>36</sup> reported that the average fibre elongation at the breaking point rose continuously by increasing relative humidity to the maximum value at 100 % relative humidity. The breaking load increased as the relative humidity was raised to 60 per cent., at which it reached a constant value and became independent of humidity. It has been claimed<sup>152</sup> that a further strength increase of 20 % is obtained if the fibres are immersed in water. The higher strength and greater extension of fibres containing moisture is attributed to a more uniform distribution of loads over the cross-section.<sup>36,200</sup>

The effect of molecular chain length on fibre strength have been deduced almost entirely from the results of heterogeneous

degradation studies, and as such may not give a valid picture of the relationship involved.<sup>57,156,109</sup>

The proportionately higher breaking strength of fine cottons have been ascribed to a skin effect which becomes less pronounced as wall thickness increases.<sup>46,52</sup> The phenomenon was described by Mann<sup>131</sup> as follows "It is generally known that thin filaments are proportionately stronger than thick ones, the surface layers than the internal layers. The strength of the cotton fibre may be regarded as due to two elements, an outer relatively more elastic and constant with a varying amount of internal thickening of more imperfect elasticity."

The orientation or the degree of alignment of the chain molecules and the fibrils with respect to fibre axis is regarded as the most important structural factor in determining cotton fibre strength and extension to break.<sup>25,28,29,134,193,202,203</sup> Orr<sup>159,160</sup> stated that the cellulosic fibres usually have either good strength or elongation, but to be outstanding in both is unusual. The reason appears to be that high strength can be achieved only by very high alignment of the cellulose chain molecules, and the elongation is then limited principally to extensibility of the primary bonds of the chains and to the lateral hydrogen bonds between chains. Such a structure occurs naturally in ramie, which has high tenacity and low elongation.

Several investigators have reported the existence of high correlations between orientation, as measured either optically or by X-ray methods, and single fibres and bundle strengths. Orr<sup>159,160</sup> reported that untwisting of the spiral structure had been observed as load was applied to the cotton fibre, and that the reversals were a vital structural feature which affects the untwisting. Orr<sup>160</sup> concluded that a reversing spirality represented an idealized structure for obtaining optimum strength, elongation, and elastic recovery from the straight-chain molecules of cellulose. The extension due to untwisting of the spiral could be calculated from the equation:

$$\text{Fractional extension} = (1 / \cos \theta) - 1$$

where  $\theta$  is the average angle of inclination of the spirals with respect to fibre axis. Rebenfeld reported the existence of a highly significant correlations between the spiral angle and the extensibility of cotton fibres, as measured by single fibre elastic modulus and elongation to break. These findings confirmed the work of Hertel and Craven<sup>102</sup> on bundle extensibility properties. Rebenfeld<sup>175</sup> concluded that the extensibility properties of cotton fibres depended on the spiral angle, the larger the spiral angle, the greater fibre extension to break.

The effect of convolutions on fibre mechanical properties has been investigated from different standpoints. Their effect

on the clinging power of single cotton fibres was examined by Sen and Ahmed<sup>186</sup> who found that the extent to which clinging power might be expected to increase with the number of convolutions per unit length was largely offset by the irregularity of spacing of the convolutions. Clegg<sup>46</sup> stated, "It was noticed that with fibres of the type having what may be described as first-class convolutions, the breaking load was high, although the cell wall was not thick. When the strain was applied, the fibre first appeared to uncoil. The comparatively high breaking load of this type of fibre appears in some way to be connected with the quality or consistency of celluloses. On the other hand, it was found that when the hair was very thick-walled, and consequently not well convoluted, the breaking load was sometimes comparatively low." Other investigators<sup>30</sup> have reported correlation between the average convolution angle and single fibre and bundle strength, and between the average convolution angle and fibre extension to break.

Several authors have reported that the ultimate fibre breaking strength at finite gauge lengths is not wholly a function of the spiral angle or chain molecular length but rather is dependent upon the occurrence of weak points along the fibre length. The structural reversals,<sup>217,218</sup> the growth rings and the nonuniformity of the fibre fine structure,<sup>65</sup> and the fibre structural or morphological abnormalities,<sup>47</sup> have been given as

possible causes for fibre weakness.

The manner in which cotton fibre mechanical properties are altered by chemical treatments through altering some structural features has been the subject of many investigations.<sup>132,159,160,177-180</sup> It is generally agreed that the change in cotton fibre extensibility caused by mercerization is a function of the degree of fibrillar orientation of the different cottons, while the increase in the fibre breaking stress of most of the cottons examined is not a function of fibrillar orientation but is caused by repair of weak points along the fibre length.

The transmission of mechanical properties of single cotton fibres, notably strength and extension to break, has been a matter of great interest for many authors.<sup>157,216,224</sup> It is generally agreed that the breaking strength of single fibres is not fully transmitted to more complex textile structures and that the degree of transmission of single fibre strength is not constant for all cottons, but is dependent on the breaking tenacity of single fibres. The transmission of single fibre elongation to break to more complex textile structures has also been found to vary for different cottons and to be inversely proportional to the single fibre breaking elongation.

CHAPTER II.

EXPERIMENTAL METHODS

## GROWING THE PLANTS AND THEIR TREATMENT WITH GIBBERELLIC ACID

Two experiments were carried out in the green house, on the roof of the Royal College of Science and Technology, Glasgow. A hot bed was prepared and the seeds were sown in small "jiffy type" pots, which are made of 75 % peat and 25 % wood pulp, through which the plant roots can grow: these were filled with "John Innes No. 2" compost. A heating system was used with soil heaters and a thermostat to keep the temperature in the soil and air around the young plants around 90°F. One 80-watt, fluorescent lamp was fixed above the hot bed to give about four hours lighting daily to compensate for the lack in natural light in the morning and evening. Water was given to the plants when needed. After four weeks, the plants in the small pots were transferred into permanent 10 inch pots which were filled with eleven pounds of compost each. Twenty four of these pots were used. A heating system of two soil heaters and four air heaters, connected with thermostats, was used to keep the temperature around 80°F. Four 125-watt, fluorescent lamps were used to give about four hours lighting daily in the early morning and the early evening to compensate for the lack in natural light in the early spring. These lamps were abandoned after two months. Water and fertilizers were given to the plants when needed.

The two experiments carried out in the green house were as follows.

(1) April to September 1962: a Nigerian cotton, Samaru 26J(G.hirsutum)

was grown. Twenty three plants were grown, six were left as control and the remainder were divided into three groups ( 5, 6, 6 ) which were treated with three successive concentrations of gibberellic acid ( 25, 100, and 200 p.p.m. )

(2) April to September 1963: two cottons were grown, an American Upland cotton, Rex ( *G. hirsutum* ), and Sea Island V.H.8 ( *G. barbadense* ). Twelve plants were grown of each variety, six plants were left as control and the other six were treated with gibberellic acid ( 200 p.p.m. )

A third experiment was carried out in Nigeria by Dr. Dransfield, The Institute of Agricultural Research, Zaria, Nigeria, from July to November 1963. About fifty plants of Samaru 26J were grown in a test plot under the normal field conditions: half of these were treated with gibberellic acid ( 250 p.p.m. ) and the rest left untreated.

The gibberellic acid solution was prepared according to the information supplied by the manufacturers. About 0.02g. of gibberellic acid powder was weighed exactly and dissolved in 1 cc. alcohol, water was added to obtain the stock solution with concentration 200 p.p.m., the other two concentrations were prepared by diluting 10 cc. of the stock solution for each. The treatment was carried out by dropping 0.2 ml. of the solution

into the calyx cup of the young boll using an Agla micrometer syringe. For Samaru 26J grown in Glasgow, 0.2 ml. of water was applied to the control plants instead of gibberellic acid. The treatment was carried out on the third day after flowering. The treatment of Samaru 26J grown in Nigeria was carried out on the second day after flowering, 0.4 ml. of gibberellic acid solution was applied.

Gibberellic acid in aqueous solutions is stable for 24 hours after which it deteriorates. Therefore a fresh solution was prepared each day: preparing the solution and treating the plants was usually completed within 2 - 3 hours. The treatment of the plants in Glasgow continued for every new flower for a period of about three weeks, after which the number of new flowers became very small so that the treatment was stopped. In Nigeria treatments were carried out over a period of a fortnight during which the flowering was greatest.

The bolls were collected at three successive stages: 30, 40, and 50 ( normally opened ) days after flowering for Samaru 26J grown in Glasgow, and 30, 40, and 60 ( normally opened ) days after flowering for Rex. The young bolls, 30 and 40 days old, were left in the green house after collection for opening to be completed. For Samaru 26J grown in Nigeria and Sea Island, only normally opened bolls were collected. All the samples were

ginned by hand.

#### FIBRE LENGTH

The instrument used to measure fibre length was a "Shirley" comb sorter. This consists of a rack on which rest nine lower combs and eight top combs, each set is accurately spaced  $1/4$  inch (6.3 mm.) apart except the first two bottom combs, which are  $3/16$  inch (4.7 mm.) apart. The fibres are laid across the combs with one end of each fibre aligned with the first comb and then the lower combs are dropped and the top combs lifted successively so that fibres of different lengths may be removed. By using the grip supplied it is possible to remove groups of fibres whose average lengths differ by chosen increments. These groups are then weighed and the data evaluated in such a manner as to give an array or diagram of fibre length distribution with pertinent measurements.

A  $75 \pm 0.4$  mg. representative sample was used, dealt with in the manner described by several authors,<sup>87,127</sup> The fibres were pulled out in small tufts, each 0.5 mm. shorter than the previous one, and these were then combed, straightened, and laid down on a black velvet pad. These tufts were arranged into groups by measuring the length of each small tuft and grouping together all fibres that fell within a range of 3 mm. Grouping together

the fibres that fell within a range of only 1 mm. was found to give a very slightly different mean length value. All groups were then weighed on a sensitive balance to the nearest 0.05 mg. and the weights recorded. The mean length, the standard deviation, and the coefficient of variation were calculated from the following equations:

$$\text{The mean length} = \frac{\sum (WL)}{\sum W}$$

where  $W$  is the weight of each length group,  $L$  is the mid point of the length group.

$$\text{The standard deviation} = \sqrt{\frac{\sum (W \times L^2)}{\sum W} - \left\{ \frac{\sum (WL)}{\sum W} \right\}^2}$$

$$\text{The coefficient of variation} = \frac{\text{standard deviation}}{\text{mean length}} \times 100 \%$$

#### FIBRE DIAMETER

Fifty fibres from each test sample were mounted on glass slides, in five groups, each of ten fibres, on a glass slide, several drops of 18% sodium hydroxide solution were placed on the fibres of the first group so as to flood and cover the central 13-15 mm., and left for 5 minutes for swelling to be completed. A cover slip was placed on the fibres and the extra sodium hydroxide solution was removed. The diameter was measured in three places in the central 11 mm. of each fibre using a magnification of 300X. The procedure was repeated for the other four groups.

## FIBRE LINEAR DENSITY

The gravimetric whole fibre method was used for measuring the linear density. The " Shirley " comb sorter was used to sort out the components of each sample into the different length groups. Fifty fibres of each length group were used, each fibre was withdrawn first, straightened on the velvet pad and its length was measured to be sure that it represented the mid point of the length group  $\pm 0.5$  mm. The fibres were then weighed in ten groups each of five fibres on a " Shirley " microcantilever balance. From the data obtained, the weight per fibre and the linear density were calculated.

## FIBRE MATURITY

Three methods were used in measuring fibre maturity.

### (1) The sodium hydroxide swelling method

Each test specimen consisted of about 100 fibres mounted on three slides, 30 - 35 on each, laid parallel and evenly separated with their centres in alignment. After placing a cover slip over the fibres several drops of 18 % sodium hydroxide solution were added so as to flood the fibres. The fibres were left for 5 minutes for swelling to be completed. Under the microscope, with magnification of 300X, the fibres were classified into three groups.

a. "Normal fibres (N)": those which, after swelling, appeared

rod-like with no continuous lumen. Because of the full degree of swelling and this rod-like form, Normal fibres showed no well-defined convolutions.

b. "Dead fibres (D)": those in which, after swelling, the wall thickness was one fifth or less of the maximum ribbon width. The maximum ribbon width was taken as the width of the widest portion of the fibre in the microscope field of view, usually midway between two convolutions. Swollen Dead fibres varied from flat ribbons, with no convolutions and little or no secondary wall, to highly convoluted forms with a greater wall development.

c. "Thin-walled fibres": those which did not fall into either the "Normal" or the "Dead" group.

The number of fibres belonging to each of these three groups was counted, and the procedure was repeated for the other two slides. The percentages of "Normal", "Dead", and "Thin-walled" fibres were calculated.

## (2) The polarized light method

This method effectively gives the thickness of the fibre from observations of the retardation of polarized light. The test specimens were as described in the first method. The fibres were immersed in liquid paraffin instead of sodium hydroxide solution. A first order quartz plate was inserted in the microscope between the crossed polarizer and analyzer, and the mechanical stage

was adjusted so that the fibres were at an angle of  $45^{\circ}$  with both the polarizer and the analyzer and the fibre axis lay parallel to the slow vibration direction of the quartz plate. The fibres were examined at 150X magnification and classified into two groups as follows.

(1) Fibres which appeared purple or indigo throughout their entire length in the field of the microscope and turned orange on rotation of the stage through  $90^{\circ}$ , and fibres which appeared deep blue or alternatively blue and purple and turned orange-yellow upon rotation of the stage, were classed as "Immature."

(2) a. Fibres which appeared blue-green or alternatively blue and yellow and turned yellow-white on rotation of the stage, and b. fibres which appeared yellow or yellow-green throughout their entire length and showed practically no change of colour on rotation of the stage, were classed as "Mature" fibres.

In some experiments, the polarized light method was used to classify the fibres into three groups by dividing the "Mature" fibres into two groups: the group (a) was regarded as "Mature" and the group (b) as "Very mature."

(3) The fibre density method

Since only a mean value for the density of a sample of fibres could be obtained thus, this method gave only a mean value of maturity, but the results were useful in comparing

different samples.

From the information obtained on fibre linear density and fibre diameter, fibre density was calculated from the following formula:

$$\text{Fibre density} = \frac{\text{Fibre weight per cm.}}{(\text{fibre radius})^2}$$

Fibre density indicates the degree of filling of the cell tube, it increases as the degree of the filling of the tube increases or in other words the degree of wall thickening.

#### THE NUMBER OF CONVOLUTIONS AND THE CONVOLUTION ANGLE

The determination of the number of convolutions per unit length was made on the central 11 mm. region of the fibres. Fifty fibres of each test sample were used. The fibres, 10 on each glass slide, were mounted straight, horizontal, and parallel to each other, taking care that the central region of all the fibres should lie more or less in the same area. A cover slip 11 X 22 mm. was placed on the central region of the fibres, a drop of liquid paraffin was placed at the edge of the cover slip and was left to spread. Using a magnification of 300X, the number of convolutions was counted for each fibre along its central 11 mm. region, and the mean ribbon width was determined from measurements made at three different places for each fibre. Also the mean true length of a convolution was determined by measuring the lengths of the convoluted portions of each fibre at three individual convolutions.

The average convolution angle and the actual convolution angle were calculated from the following formulae.

(1) The average convolution angle ( $\phi$ ),

$$\tan \phi = \frac{\pi}{2} \times \frac{D}{C}$$

where  $D$  is the average ribbon width, and  $C$  is the average convolution pitch which is the length of the specimen ( 11 mm. ) divided by the number of convolutions.

(2) The actual convolution angle ( $\phi'$ ),

$$\tan \phi' = \frac{\pi}{2} \times \frac{D}{C'}$$

where  $D$  is the average ribbon width, and  $C'$  is the true convolution length.

#### THE NUMBER OF REVERSALS

The number of reversals per unit length was counted on the same fibres used in the determination of the number of convolutions. A first order quartz plate was inserted in the microscope between the crossed polarizer and analyzer with its slow vibration direction making an angle of  $45^\circ$  to the plane of polarization. The fibres were placed parallel to the plane of polarization so that the (S) and (Z) spirals appeared in different colours and the number of reversals could be counted. This was checked by removing the quartz plate so that the reversals appeared as dark bands.

## ORIENTATION

Two methods were used to measure the molecular and the crystallite orientation.

### (1) THE REFRACTIVE INDICES AND THE ORIENTATION ANGLE

The refractive indices of the fibres in plane polarized light were measured by the Becke line method. This method has been described in detail by Meredith.<sup>133</sup> Briefly, a series of liquids with refractive indices increasing in steps of 0.001, from 1.527 to 1.533 and from 1.560 to 1.582, was prepared by mixing different volumes of 1-monobromonaphthalene ( $n_D=1.658$  at  $20^\circ\text{C}.$ ) and liquid paraffin ( $n_D=1.480$  at  $20^\circ\text{C}.$ ) For a mixture of two liquids the refractive index of the mixture is

$$n = \frac{(n_1 v_1 + n_2 v_2)}{(v_1 + v_2)}$$

where  $n_1$  and  $n_2$  are the refractive indices and  $v_1$  and  $v_2$  are the volumes of the liquids 1 and 2. The refractive indices of the liquid mixtures for the sodium D line were measured at  $20^\circ\text{C}.$  with an Abbe<sup>4</sup> refractometer, calibrated with several liquids of known refractive index. The fibres were immersed in a series of these liquids of increasing refractive index until a match was obtained, i.e., the fibre became invisible. The Becke line was used at each stage to detect any mis-match.

To measure the mean values of the higher and lower refractive indices of a sample, a series of glass slides was prepared, five

fibres of known and equal length were mounted parallel to each other and just straight. The fibres were fixed at each end by a drop of Gum Arabic which took some time to dry and allowed time for the fibres to be straightened without tension. A cover slip, 11 X 22 mm., was placed on the central region of the fibres, a drop of a liquid of approximately the same refractive index as the fibre was placed at the edge of the cover slip and was left to spread and cover the fibres, the fibres were re-straightened without tension and examined under the microscope with their long axes parallel to the direction of vibration of the plane polarized light. From the observed degree of matching of the outer edge of the fibres a second liquid was chosen with higher or lower refractive index as required. When the liquid of the nearest refractive index to that of the fibre was defined, this liquid was re-used on another two slides, each bearing five fibres, to measure the exact and final mean value of the higher refractive index. This procedure was repeated to measure the lower refractive index using another series of fresh slides. The refractive indices of the fibres were measured always on straight, unconvoluted parts of the fibre which were free from reversals. It would appear that errors might arise because the two refractive indices were not measured on the same fibres: however it was found that no such error arose because the lower refractive index was almost constant for all fibres.

All the measurements were made in an air-conditioned room at  $65 \pm 2$  % R.H. and  $20 \pm 1^{\circ}\text{C}$ . The temperature was watched closely and the measurements were carried out only when the room temperature had been fairly constant for 2 hours. Corrections for small temperature fluctuations were made when needed, using Meredith's<sup>133</sup> value for the temperature coefficient.

The Orientation angle ( $\theta$ ) of the chain molecules was calculated from the formula;<sup>173</sup>

$$\cos^2 \theta = \frac{(n_y)^2 (n_{11} - n_a) (n_{11} + n_a)}{(n_{11})^2 (n_y - n_a) (n_y + n_a)}$$

where  $n_{11}$  is the higher refractive index measured for the test specimen,  $n_y$  and  $n_a$  are the higher and lower refractive indices respectively measured for ramie fibres.

Preston used the values  $n_a = 1.528$  and  $n_y = 1.596$ , measured for flax and ramie fibres in which the crystallites are orientated almost parallel to the fibre axis. Meredith<sup>133</sup> used the values  $n_a = 1.531$  and  $n_y = 1.595$  measured for flax fibres. In this work, the two refractive indices were measured for ramie fibres and found to be  $n_a = 1.530$  and  $n_y = 1.594$ .

As  $n_1$  was found constant for all the samples under investigation, the orientation angle was found sufficient to express or describe the overall molecular orientation.

## (2) THE X-RAY DIFFRACTION METHOD

### (a) Preparation of samples

The object was to obtain a parallel bundle of fibres, of equal length and of length equal to the mean length of the test sample, and of fairly constant thickness from one test sample to another. The procedure followed for preparing the bundle of fibres was the same as that used in measuring fibre length; a 75 milligrams fibre specimen was used and sorted on the "Shirley" comb sorter, all fibres longer than the mean length + 0.5 mm. were pulled out and rejected. Fibres in the range mean length  $\pm$  0.5 mm. were pulled out of the sorter, combed, and mounted on the specimen holder.

Specimen holders made from stainless-steel or brass were used in mounting fibre bundles of about 0.8 - 1.0 mm. thickness. The specimen holder consisted of two jaws, one fixed and the other one moveable by means of a long screw-shaft. Each jaw had a groove of 1 mm. width and 2 mm. depth in its centre, in which the end of the fibre bundle was placed, a clamp with length and width equal to that of the jaws and with a tongue in its centre with the same size as that of the groove, was placed on the jaw to fix the fibre bundle. This clamp could be tightened by means of two small screws.

The free end of the fibre bundle, after being pulled out of the sorter by means of a grip, was clamped in the fixed jaw. The fibres were then re-combed and straightened, and clamped in

the moveable jaw. The sample with its fibres parallel, was stretched gently to remove the crimp by moving the moveable jaw outwards.

(b) Sample exposure

The X-ray photographs were taken using Ni-filtered  $\text{CuK}\alpha$  radiation of wave-length 1.54 Å., from a Philips sealed-off tube run at 40 Kv. and 20 mA. The X-ray beam was collimated by a lead glass capillary tube 5 cm. long and having an internal diameter of 0.3 mm. encased in brass. A metal holder was fixed on the outer end of the collimator casing and was used to hold the specimen holder in a definite position in which the fibre bundle was up against the end of the collimator and the X-ray beam was passing through the centre of the fibre bundle at right angle to its axis. The X-ray film, a quarter plate Ilford Industrial B film, was held in a cassette mounted on rails running parallel to the collimator. The front of the cassette was covered with thin aluminium foil ( 40  $\mu$  thick ), and at the centre of the foil a lead stop was stuck with Durofix, in order to absorb the primary un-diffracted X-ray beam. The cassette was placed parallel to the bundle axis and perpendicular to the X-ray beam at 4 cm. distance between the fibre bundle and the flat film, and was adjusted so that the lead stop in the centre was in line with the main X-ray beam issuing from the collimator.

The time of exposure varied according to fibre maturity:

young, immature fibres needed longer exposures. It ranged between 60 and 120 minutes to get a diffraction photograph with suitable density ( maximum density on 002 arc of 0.5 to 0.7 ).

The films were developed according to the manufacturer's recommendations. Each film was developed for 5 minutes at 65<sup>o</sup>F., washed for 30 seconds, fixed for 6 minutes, washed again for 30 minutes, and then rinsed and dried.

(c) Intensity measurements

A "Joyce Loebel Mark I Microdensitometer," with a graduated rotating stage was used for recording the intensity distribution around the (002) diffraction arc of the X-ray photograph.

The X-ray photograph was fixed in the centre of the rotating stage. Starting from the centre of the photograph, scanning was carried out radially across the (002) arc and approximately at its centre which is the point of maximum blackening. From this point one half of the arc was scanned radially, first at three successive azimuthal intervals of 3<sup>o</sup> each, and then at intervals of 6<sup>o</sup> until the arc vanished. Then the other half of the arc was scanned in the same manner starting again from the first starting point. Thus this starting point was scanned twice: the result of the two scans was found to coincide throughout this work which can be taken as a proof of the consistency of the apparatus. The scanning was carried out always from the inner side of the

arc, just beyond the (  $10\bar{1}$  ) arc, and across the ( 002 ) arc with the machine running at constant speed. These scans were recorded automatically on graph paper. The peak height of each radial scan was measured by a ruler and taken as a measurement of the degree of blackening at this definite point on the ( 002 ) arc. These measurements were plotted against the angle of scanning and the intensity distribution around the ( 002 ) arc was constructed. This procedure was repeated to obtain the angular intensity distribution around the second ( 002 ) arc of the X-ray photograph. At least two photographs from different fibre bundles were scanned for each test sample, and the results from individual photographs differed by  $1^{\circ}$  or less.

The calibration of the films was made according to the method described by Meredith,<sup>135</sup> but using separate films other than those used in obtaining the fibre diffraction photographs. A rotating sector sensitometer giving a linear series of 18 exposures times was used. A number of Industrial B films from the same boxes as were used for the fibre photographs were exposed to Cu  $K\alpha$  at 40 Kv. and 10 mA. for long enough ( about 10 seconds ) to give a maximum density equal to the maximum in the fibre photographs (  $0.7 D$  ). These films were processed under the same conditions as the fibre photographs and measured on the Microdensitometer. The results obtained showed that the

X-ray intensity/photographic density relationship was linear within the range of density used.

(d) Calculation of Orientation

After having obtained the angular intensity distribution of each of the two ( 002 ) arcs of the X-ray photograph, the crystallite orientation was measured in two ways; assuming always that photographic density was proportional to X-ray intensity.

1. The 40 per cent. X-ray angle ( absorption ) was measured as the azimuthal angle between the point of maximum blackening and the point on the arc where the blackening was 40 per cent. of the maximum.
2. The 50 per cent. X-ray angle ( absorption ) was measured as the azimuthal angle between the point of maximum blackening and the point on the arc where the blackening was 50 per cent. of the maximum.

## THE MECHANICAL PROPERTIES

The "Cambridge" Extensometer was used to determine the load extension curves of single fibres.

### (a) Testing procedure

To mount a fibre with a test length of 1 cm., it was laid along the centre of an aperture of 1 cm. length ( and 0.5 cm. width ) in a special card mount, and secured at each edge by a small drop of Durofix. The fibre was mounted straight with just sufficient tension to remove any kinks. The ends of the card were fixed into the jaws of the Cambridge Extensometer, the thin sides of the mount were then cut to leave the fibre free, and then, starting with the fibre just taut, it was extended at constant rate until it ruptured. The constant rate of extension used was 4 mm. / minute so that the fibre broke within 10 - 20 seconds. About 50 fibres from each test sample were tested. The mass per unit length was determined for the 50 fibres weighed in mass. The testing was carried out in an air-conditioned room at  $65 \pm 2$  % R.H., and temperature at  $20 \pm 1^{\circ}\text{C}$ .

### (b) Measurements on the stress-strain curves

The load-extension curves were recorded on graph paper. The load and extension at break were measured for each curve and the

mean values found for each sample. The yield point for each curve was located according to Coplan's<sup>58</sup> construction in which he defined the yield point as that occurring at the stress given by the intersection of the tangent at the origin with the tangent having the least slope. The yield stress and the yield strain were measured for each curve and the mean values found for each sample. The initial Young's modulus was calculated as the ratio of yield stress to yield strain.

Typical stress-strain curves for cotton<sup>127</sup> show two ill-defined points of inflexion. The first occurs at a low value of extension when there is an increased rate of yielding as the stress is increased further. A second point of inflexion often occurs as the point of rupture is approached, more particularly if rupture does not occur early because of local weak place. The curve typically swings upwards in this final stage, increasing increments of stress having to be applied to produce equal increments of extension.

To determine the typical stress-strain curve for each sample, the curves of the sample were sorted out into two groups, the first one comprised the curves in which breaking took place at or near the second inflexion point apparently because of a major local weak place: this group was designated as prematurely-broken fibres. The second group comprised the curves which showed a final upward

swing after the second inflexion point. These were designated as normally-broken fibres. The measurements were carried out as follows:

1. The stress and the strain at the first inflexion point were taken as being equal to the yield stress and yield strain and these were measured for all the curves in the sample.
2. The stress and the strain at the breaking point in the first group of curves, the prematurely-broken fibres, were measured. The stress and the strain at the second inflexion point of the second group, the normally-broken fibres, were measured. These two sets of measurements were added together and the mean values of stress and strain were calculated and supposed to represent the stress and the strain values at the second inflexion point.
3. The stress and the strain increments between the second point of inflexion and the breaking point for the second group of curves, i.e. for the normally-broken fibres, were measured and the mean values of stress and strain were calculated in two ways.
  - (a) By summing all these stress and strain increments and dividing by the total number of curves, for both prematurely- and normally-broken fibres, and then adding these mean values of stress and strain to those at the second point of inflexion. The total mean values obtained thus should be equal to the mean values of recorded breaking stress and strain of the sample and this alternative

method of calculation supplied a useful check on the accuracy of the calculation already made.

(b) By dividing the total values of these stress and strain increments by the actual number of curves from which these values were obtained, which was the number of normally-broken fibres, and then adding these mean values of stress and strain to that at the second point of inflexion. The total mean values thus obtained were higher than the recorded mean values of stress and strain at break. These values were supposed to be the real ones if there had been no major local weak points, and were designated as the projected mean values of stress and strain at break.

Having obtained the mean values of stress and strain at four points, i.e. the yield point, the second point of inflexion, the actual breaking point, and the projected breaking point, the typical stress-strain curve for each sample was constructed. From this stress-strain curve; the work of rupture was measured by means of an "ALLBRIGHT" planimeter, and the "work factor" calculated. Stiffness was calculated as the breaking stress per unit breaking strain. These characteristics were measured for both the actual and extrapolated stress-strain curves.

### The role of convolutions in fibre extensibility

#### The apparatus

A simple apparatus was designed to stretch single cotton

fibres under the microscope. The main features of the apparatus were two jaws which could be moved outwards or inwards simultaneously at the same speed by means of a single screw with a right and left hand threads. The relative motion of the jaws was indicated by a scale on the knob by which the screw was turned. Between the jaws was a frame 1.2 cm. wide with an aperture of 1.0 cm. width covered by a thin glass cover slip. The whole apparatus was mounted on a base which could be clamped to the microscope stage in such a position that a fibre lying across the aperture was beneath the objective.

The fibre was mounted on a special card mount in the same manner as was previously described for testing on the "Cambridge" Extensometer. The two jaws were brought to the edges of the frame and the ends of the card mount were placed fairly loosely into the jaws. The fibre was examined under the microscope, using a magnification of 300X: it was first centred, and then the ends of the card were finally fixed into jaws and the thin sides of the mount were then cut to leave the fibre free to extend. By turning the screw, the fibre was extended from both ends at the same time which kept the same portion of the fibre in the field of the microscope throughout.

The mechanism of the unfolding of the convolutions was watched, the point of extension at which the convolutions completely

disappeared and the point of extension at which the fibre broke were determined for 50 fibres of one test sample.

The percentage of extension due to the unfolding of the convolutions ( $E_c$ ) was calculated from the following formula;

$$E_c = (\sec \phi' - 1) \times \text{C.P.} \%$$

where  $\phi'$  is the actual convolution angle, C.P. is the percentage convoluted portion of the fibre which is,

$$\text{C.P.} = \frac{\text{the average convolution length (mm.)} \times \text{the number of convolutions per mm.} \times 100}{\text{the length of the fibre}}$$

#### Correction of the stress-strain curves for the effect of convolutions

The stress-strain curve of each sample was corrected for the effect of the convolutions on fibre extensibility on the assumption that no stress had been applied in unfolding the convolutions since it was not possible in this stage of work to determine or calculate the stress actually used in unfolding the convolutions.

The percentage extension due to the unfolding of the convolutions which was calculated for each sample, was subtracted from the percentage extension at the first and second points of inflexion in proportion to the actual extensions at these points. The stress-strain curves were then replotted and, the initial Young's modulus, the work of rupture, the work factor, and the stiffness were found.

## MERCERIZATION PROCEDURE

To mercerize at constant length, the fibre bundles were prepared in the same manner as for the X-ray exposure, mounted on stainless-steel holders, stretched just to remove crimp, and mercerized by the following procedure, which was carried out at 20°C throughout.

10 minutes in 18 % sodium hydroxide solution,

10 " washing in running water,

5 " in 1 % acetic acid,

10 " washing in running water,

followed by drying.

## RESULTS AND DISCUSSION

The following symbols are used in the text:

Descriptive:

G.A.	Gibberellic acid
M	Fibres from normally opened bolls
Samaru 26J (G)	Samaru 26J variety grown in Glasgow
Samaru 26J (N)	Samaru 26J variety grown in Nigeria
S., I.	Samaru 26J (G) - control
S., II.	Samaru 26J (G)- treated with gibberellic acid ( 25 p.p.m. )
S., III.	Samaru 26J (G) - treated with gibberellic acid ( 100 p.p.m. )
S., IV.	Samaru 26J (G) - treated with gibberellic acid ( 200 p.p.m. )
S., V.	Samaru 26J (N) - control
S., VI.	Samaru 26J (N) - treated with gibberellic acid ( 250 p.p.m. )
R., I.	Rex variety - control
R., II.	Rex variety - treated with gibberellic acid ( 200 p.p.m. )
S.I., I.	Sea Island variety - control
S.I., II.	Sea Island variety - treated with gibberellic acid ( 200 p.p.m. )

Measured quantities:

G	The average convolution pitch
C'	The average true convolution length
D	Fibre ribbon width
$E_m$	The measured extension to break
$E_p$	The extrapolated extension to break
$E_c$	The percentage extension due to unfolding the convolutions
$E_f$	$= E_p - E_c$
l	Fibre length
n	The number of spiral turns along the fibre length
$\frac{l}{n}$	The spiral pitch
r	Fibre radius
$T_m$	The measured tensile strength
$T_p$	The extrapolated tensile strength
$S_m$	The measured stiffness $(\frac{T_m}{E_m})$
$S_{ma}$	Calculated stiffness $(\frac{T_m}{E_f})$
$S_p$	Extrapolated stiffness $(\frac{T_p}{E_p})$
$S_{pa}$	Calculated extrapolated stiffness $(\frac{T_p}{E_f})$

$WR_m$	The measured work of rupture ( the area enclosed between the measured stress-strain curve and the strain axis )
$WR_p$	The extrapolated work of rupture ( the area enclosed between the extrapolated stress-strain curve and the strain axis )
$WR_{pa}$	The work of rupture due to fibre fine structure ( the area enclosed between the re-plotted stress-strain curve and the strain axis )
$WF_m$	The work factor of the measured stress-strain curve $( \frac{WR_m}{E_m X T_m} )$
$WF_p$	The work factor of the extrapolated stress-strain curve $( \frac{WR_p}{E_p X T_p} )$
$WF_{pa}$	The work factor of the re-plotted stress-strain curve $( \frac{WR_{pa}}{E_f X T_p} )$
$\phi$	The average convolution angle
$\phi'$	The actual convolution angle
$\theta$	The average orientation angle
$\gamma$	The 40 per cent. X-ray angle
$\psi$	The 50 per cent. X-ray angle
$\Phi$	The 40 per cent. X-ray angle after subtracting the average convolution angle $( \gamma - \phi )$

$Y_m$ 

The measured initial Young's modulus

 $Y_c$ The calculated initial Young's modulus ( after  
the removal of the effect of convolutions )

CHAPTER III.

COTTON FIBRE GROWTH AND GROSS STRUCTURE

TABLE 1 . Fibre length distribution.

Length group (mm.)	Percentage by weight			
	S., I.	S., IV.	S., V.	S., VI.
3	2.0	1.0	0.5	0.8
6	1.5	0.5	0.5	0.5
9	1.5	0.5	0.5	1.0
12	2.0	1.0	2.0	2.2
15	3.0	1.5	3.5	2.5
18	4.0	2.0	7.0	5.0
21	4.0	3.5	10.5	7.5
24	16.5	19.0	14.0	12.0
27	19.5	26.0	18.0	20.0
30	21.0	20.0	18.5	21.0
33	18.0	12.0	15.5	17.5
36	7.0	9.0	9.5	10.0
39	-	4.0	-	-

TABLE 1A . Fibre length distribution.

Length group (mm.)	Percentage by weight			
	S.I., I.	S.I., II.	R., I.	R., II.
3	1.5	2.0	1.3	1.0
6	1.0	1.5	1.0	0.5
9	2.5	2.0	1.2	1.0
12	2.5	2.5	2.0	0.5
15	3.5	3.5	2.5	2.0
18	4.0	3.5	3.5	2.0
21	4.0	4.5	7.5	5.0
24	5.0	5.0	12.0	7.0
27	5.0	5.0	24.0	17.0
30	6.0	5.5	24.0	26.5
33	6.0	6.5	14.5	26.5
36	7.5	7.0	6.5	11.0
39	8.5	8.0		
42	11.5	10.5		
45	11.5	11.5		
48	8.5	10.0		
51	5.5	6.0		
54	3.5	3.0		
57	2.5	2.5		

The following are the coefficients of variation calculated for the sample ( R., II. - 29 mm. long ), as an example of the variability found in measuring the different characteristics.

Characteristic	Coefficient of Var. %	Number of readings
Fibre Weight ( and linear density )		
Sample 30 days old	28	10
Sample 40 days old	15	10
Sample 60 days old	18	10
Fibre diameter	7	50
Ribbon width	7	50
Number of Convolutions / mm.	8	50
Number of reversals / mm.	12	50

TABLE 2 . Gross structural properties of fibres of the  
different length groups.

Sample	Fibre length (mm.)	Diameter ( $\mu$ )	Ribbon width ( $\mu$ )	Conv. per mm.	Av.conv. angle ( $\phi^{\circ}$ )	Convol. portion %	Act.conv. angle ( $\phi^{\circ}$ )
S., I.	36	23.2	22.0	4.18	8.25	27.2	28.0
	34	22.2	21.5	5.19	9.90	-	-
	32	25.2	20.4	4.53	8.25	-	-
	30	25.1	20.6	4.28	7.90	-	-
	28	25.8	21.6	4.82	9.30	31.3	27.6
	26	25.9	21.0	4.00	6.70	-	-
	24	26.6	21.2	5.54	10.50	-	-
	22	25.9	21.5	5.94	11.35	-	-
	20	26.9	21.9	4.75	9.30	30.7	27.8
S., IV.	38	21.9	20.0	4.06	7.30	-	-
	36	22.9	20.3	4.12	7.45	26.8	26.1
	34	23.2	20.4	4.20	7.65	-	-
	32	24.6	21.5	4.08	7.85	-	-
	30	23.8	21.2	4.00	7.60	-	-
	28	24.8	20.9	4.03	7.55	26.2	26.8
	26	25.4	21.6	4.38	8.45	-	-
	24	25.1	23.1	3.76	7.75	-	-
	22	26.2	21.4	4.26	8.15	-	-

TABLE 2 . ( continued )

Sample length (mm.)	Fibre Diameter ( $\mu$ )	Ribbon width ( $\mu$ )	Conv. per mm.	Av.conv. angle ( $\phi^\circ$ )	Convol. portion %	Act.conv. angle ( $\phi'$ )
S., IV. 20	26.4	22.1	3.37	6.70	21.9	28.1
S., V. 28	25.9	19.2	4.43	6.80	28.4	25.2
S., VI. 28	25.9	19.6	3.48	5.50	21.0	27.1
S.I., I. 35	18.9	17.1	2.87	4.40	16.1	25.7
S.I., II. 35	18.8	17.0	2.70	4.10	16.7	23.3
R., I. 35	24.3	21.0	6.40	11.95	40.3	27.7
32	24.4	21.9	4.80	9.40	28.8	29.9
29	25.4	21.5	5.60	10.75	29.7	32.0
27	26.4	22.4	4.90	9.75	28.9	30.8
23	26.4	23.4	3.70	7.70	22.6	31.1
20	26.9	23.1	3.50	7.25	20.2	32.1
R., II. 35	23.6	20.7	5.30	9.80	35.0	26.3
32	24.7	20.9	4.80	9.00	30.4	27.5
29	25.6	21.1	5.70	10.60	33.9	28.9
26	25.2	21.7	4.50	8.75	26.7	30.7
23	25.7	23.0	4.00	8.15	24.1	30.7
20	26.0	21.8	3.30	6.50	19.5	30.2

TABLE 3 . Fibre weight for the different length groups.

Sample	Fibre length (mm.)	Fibre weight ( $10^{-8}$ g. )		
		30 days old	40 days old	'Mature' fibres (M)
S., I.	36	171	297	356
	34	167	278	321
	32	156	275	333
	30	155	264	381
	28	155	259	392
	26	157	257	372
	24	164	257	371
	22	166	266	361
	20	145	237	338
S., IV.	38	170	241	384
	36	151	248	353
	34	152	208	337
	32	145	224	322
	30	144	222	306
	28	165	215	317
	26	163	213	295
	24	155	230	272
	22	139	193	264
20	134	147	219	

TABLE 3 . (continued)

	Fibre length (mm.)	Fibre weight ( $10^{-8}$ g. )		
		30 days old	40 days old	'Mature' fibres (M)
S., V.	28	-	-	432
S., VI.	28	-	-	452
S.I., I.	35	-	-	326
S.I., II.	35	-	-	331
R., I.	35	335	346	491
	32	314	342	478
	29	315	358	508
	27	307	334	500
	23	272	330	516
	20	207	280	498
R., II.	35	280	414	518
	32	267	402	482
	29	257	370	470
	26	260	384	452
	23	200	366	398
	20	158	316	378

TABLE 4 . Fibre linear density for the different length groups

Sample	Fibre length (mm.)	Fibre linear density ( $10^{-8}$ g. )		
		30 days old	40 days old	'Mature' fibres ( $\mu$ )
S., I.	36	48	82	99
	34	49	82	94
	32	49	86	104
	30	52	88	127
	28	55	93	140
	26	61	99	143
	24	68	107	155
	22	76	121	164
	20	73	118	169
S., IV.	38	47	63	101
	36	42	69	98
	34	45	61	99
	32	45	70	100
	30	48	74	102
	28	59	77	113
	26	63	82	114
	24	65	96	114
	22	63	87	120
20	66	73	110	

TABLE 4 . ( continued )

Sample	Fibre length (mm.)	Fibre linear density ( $10^{-8}$ g. )		
		30 days old	40 days old	'Mature' fibres (M)
S., V.	28	-	-	154
S., VI.	28	-	-	161
S.I., I.	35	-	-	93
S.I., II.	35	-	-	95
R., I.	35	96	99	140
	32	98	107	149
	29	108	123	175
	27	114	124	185
	23	118	143	225
	20	104	140	249
R., II.	35	80	118	148
	32	83	126	151
	29	89	128	162
	26	100	148	174
	23	87	159	173
	20	79	158	189

TABLE 5 . Fibre density for the different length groups.

Sample	Fibre length (mm.)	Fibre density ( g cm <sup>-3</sup> )		
		30 days old	40 days old	'Mature' fibres (M)
S., I.	36	0.352	0.610	0.733
	34	0.400	0.665	0.768
	32	0.308	0.543	0.658
	30	0.328	0.560	0.809
	28	0.334	0.558	0.843
	26	0.360	0.590	0.851
	24	0.389	0.610	0.880
	22	0.452	0.723	0.982
	20	0.400	0.654	0.934
S., IV.	38	0.393	0.533	0.850
	36	0.323	0.530	0.755
	34	0.330	0.454	0.735
	32	0.300	0.464	0.662
	30	0.339	0.522	0.718
	28	0.383	0.500	0.735
	26	0.384	0.500	0.700
	24	0.410	0.600	0.718
	22	0.368	0.510	0.700
20	0.380	0.422	0.631	

TABLE 5 . ( continued )

Sample	Fibre length (mm.)	Fibre density ( g cm <sup>-3</sup> )		
		30 days old	40 days old	'Mature' fibres (M)
S., V.	28	-	-	0.918
S., VI.	28	-	-	0.960
S.I., I.	35	-	-	1.041
S.I., II.	35	-	-	1.074
R., I.	35	0.653	0.673	0.952
	32	0.658	0.718	1.000
	29	0.671	0.764	1.087
	27	0.655	0.712	1.063
	23	0.678	0.822	1.293
	20	0.575	0.774	1.377
R., II.	35	0.576	0.849	1.065
	32	0.546	0.829	0.993
	29	0.543	0.780	0.988
	26	0.628	0.931	1.094
	23	0.527	0.964	1.048
	20	0.467	0.935	1.118

## FIBRE LENGTH AND FIBRE LENGTH DISTRIBUTION

Table ( 6. ) summarises the results obtained for the control samples and samples treated with gibberellic acid. The mean length and the coefficient of variation are mean values of 4 - 6 test specimens of each sample. The upper quartile length and the effective length have been derived from the mean fibre length/per cent. weight-diagram.<sup>127</sup>

TABLE 6.

Variety	G.A. concent. p.p.m.	Fibre mean length mm.	Coef. of variation %	Upper quartile mm.	Effective length mm.
Samaru 26J (G)					
(S., I.)	000	26.8	24.2	31.5	31.8
(S., II.)	025	28.2	20.1	-	-
(S., III.)	100	28.4	19.3	-	-
(S., IV.)	200	28.5	16.6	31.7	31.8
Samaru 26J (H)					
(S., V.)	000	26.7	23.2	31.5	31.5
(S., VI.)	250	27.2	23.1	31.6	32.0
Rex					
(R., I.)	000	26.8	24.3	30.8	31.0
(R., II.)	200	28.9	21.3	33.0	33.0
Sea Island					
(S.I., I.)	000	35.4	35.9	45.1	46.5
(S.I., II.)	200	35.1	39.0	45.5	47.0

The mean fibre length is a sensitive index which may be used to detect changes in the length characteristics, while the upper quartile length and the effective length are measures typifying the length of the longer fibres in a given sample. The coefficient of variation is a measure of fibre-length irregularity and is independent of the general length of the sample.

From table ( 6 ) it can be seen that the treatment with gibberellic acid has resulted in the following:

1. an increase in the mean length of Samaru 26J (G) ( S., III., S., III., and S., IV. ) of about 5.3 - 6.2 %, and of Rex ( R., II. ) of 7.3 % : both are significant at 5% level,
2. a decrease in the coefficient of variation of Samaru 26J (G) ( S., II., S., III., and S., IV. ) of 14 - 31 %, and of Rex ( R., II. ) of 12.3 % : both are significant at 5 % level,
3. a negligible increase in the mean length of Samaru 26J (N) ( S., VI. ), and a negligible decrease in the mean length of Sea Island ( S.I., II.), these differences are not significant,
4. a negligible decrease in the coefficient of variation of Samaru 26J (N) ( S., VI. ), and an increase in the coefficient of variation of Sea Island ( S.I., II. ), these differences are not significant,
5. (a) no difference in the upper quartile length and the effective length between the control samples and the samples treated with gibberellic acid for Samaru 26J (G), Samaru 26J (N), and Sea Island.

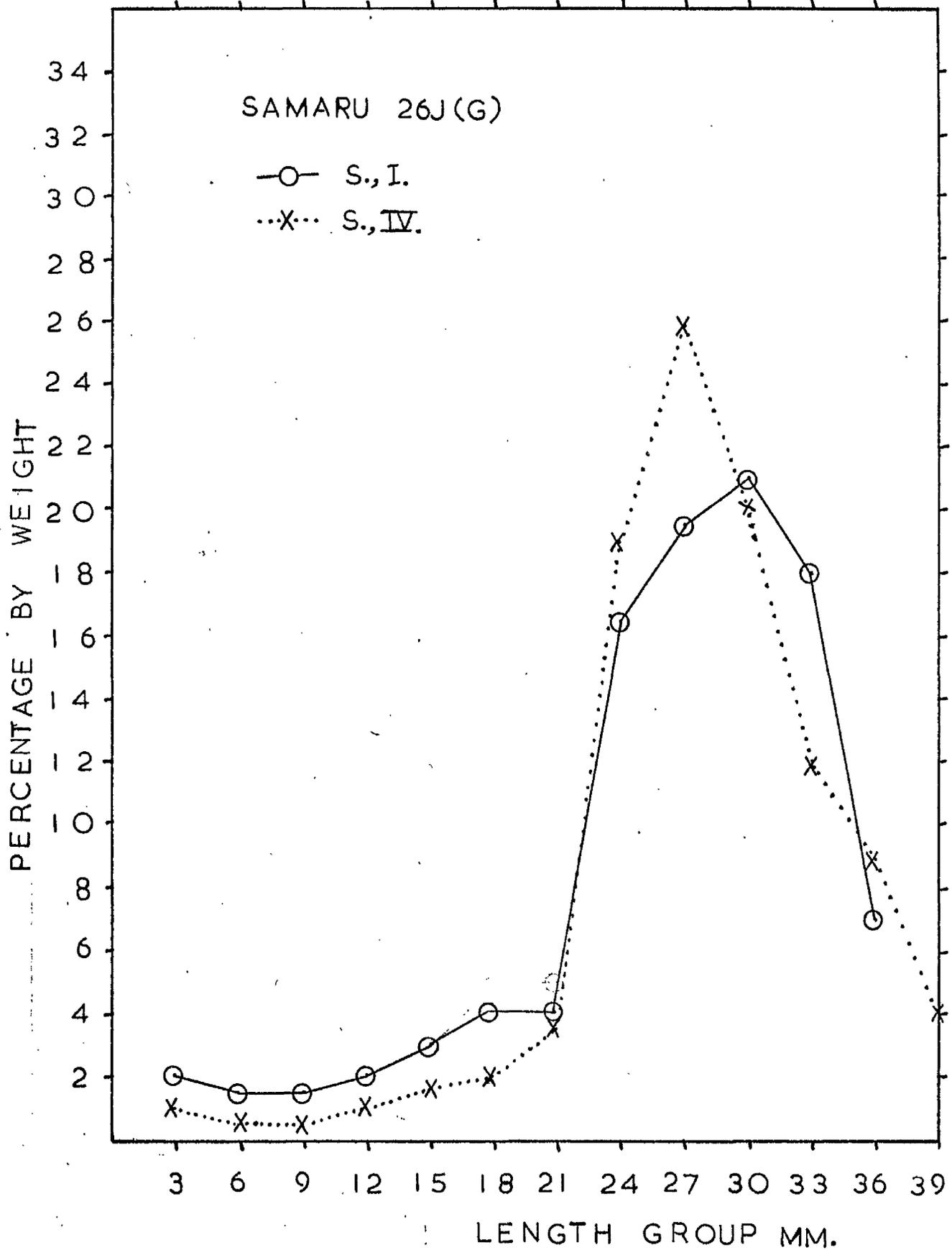


Fig. 3 Fibre length distribution

(b) higher values of upper quartile length and effective length for the treated sample in the Rex variety,

6. approximately the same increase in the mean length and a gradual decrease in the coefficient of variation resulting from the three successive concentrations of gibberellic acid used for Samaru 26J (G).

Comparing the length distribution in the control sample of Samaru 26J (G) ( S., I. ) with that of the sample treated with 200 p.p.m. ( S., IV. ), Figure 3, there is a remarkable decrease in the size of the short length groups and a marked increase in the size of the length groups near the mean length of the sample, in the treated sample. There is also the appearance of a new length group in the treated sample longer than the longest length group in the control sample. The decrease in the size of the shorter length groups and the increase for the longer length groups can be explained in part by the pattern of distribution of the weight per fibre of the different length groups and partly by a shift in the number of fibres in each length group: this will be discussed in detail for the Rex variety. The appearance of the new longest length group in the treated sample can be explained only by the possibility of shifting of some fibres of the three longer groups into this new group since the total size of the longer groups of the control sample ( 30, 33, and 36 mm. )

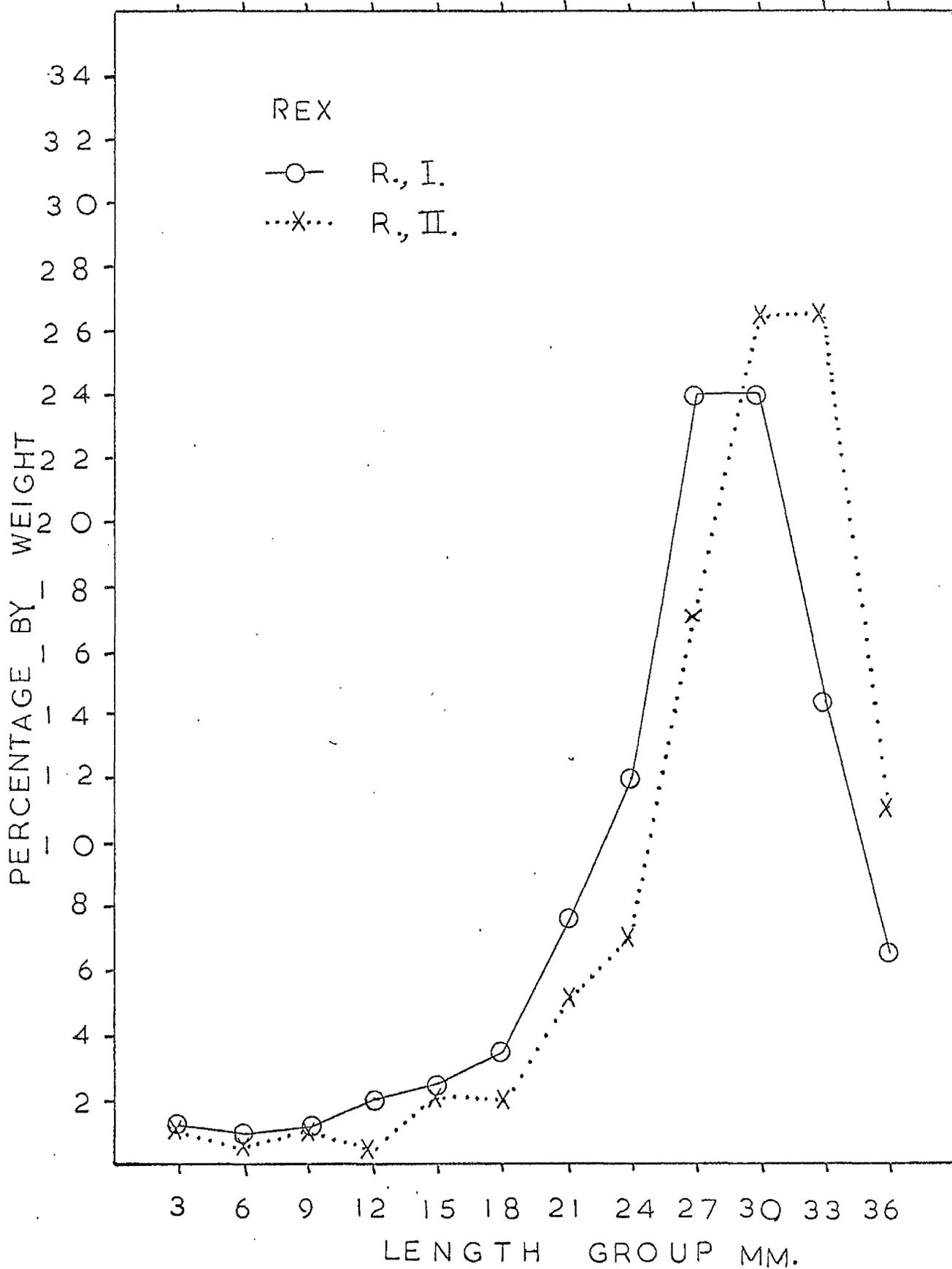


Fig. 4 Fibre length distribution

is approximately equal to that of the same groups plus the new group ( 30, 33, 36, and 39 mm. ) of the treated sample.

Comparing the length distribution in the control sample of Rex ( R., I. ) with that in the treated sample ( R., II. ), Figure 4, it is seen that a general decrease in percentage by weight of all the length groups in the treated sample compared with the control sample, except for the longer three length groups where there is a marked increase; but no additional length group appears. This difference in the percentage by weight of the different length groups could be due to either (1) a difference in the weight per fibre in the different length groups if the number of fibres in each of these length groups is constant for the control and treated samples, or (2) a decrease in the number of fibres in the short length groups and an increase in the number of fibres in the three longer length groups of the treated sample if the weight per fibre in both the control and the treated samples is constant for all the length groups. However, both these factors may participate at the same time. The weight per fibre is approximately constant for all the length groups in the control sample, but decreases with the decrease in fibre length in the treated sample ( see Figure 10 ). This difference may explain part of the difference in percentage by weight of the different length groups, but is not great enough to be taken as

the major cause. To examine this point, the weight per fibre of the different length groups has been used to calculate the number distribution of fibres among the different length groups and the two distributions compared ( Table 7 ), for the control and the treated samples of Rex. In order to facilitate calculation, the number distribution has been calculated only for the length groups longer than 19 mm., and for length groups shorter than 19 mm. it has been assumed that the weight and number distributions are identical, being 7.7 and 5.6 per cent. in the control and the treated samples respectively.

TABLE 7.

Length group mm.	( R., I. ) Percentage		( R., II. ) Percentage	
	by weight	by number	by weight	by number
34 - 36	9.1	9.3	16.5	14.9
31 - 33	18.8	19.7	31.5	30.7
28 - 30	29.3	28.8	27.0	26.9
25 - 27	18.5	18.0	9.6	10.0
22 - 24	7.5	7.3	5.7	6.8
19 - 21	9.1	9.2	4.1	5.1
Total	92.3	92.3	94.4	94.4

From table 7 , it is apparent that for the control sample ( R., I. ), the difference between the weight and the number distributions is negligible. While in the treated sample ( R., II. ) the number percentage is greater than the weight percentage for the short length groups and smaller for the three longer length groups. The difference in the pattern of the number distribution between the control and the treated samples is comparable to that of the weight distribution, but it is smaller in magnitude. This means that part of the difference in the mean length of the control and the treated samples, and part of the difference in the weight distributions is due to the difference in weight per fibre of the different length groups affected by gibberellic acid treatment. The number distribution in the treated sample suggests that the increase in the percentage by weight of the longer length groups is mainly due to an increase in the number of fibres in these groups, while the decrease in the percentage by weight of the shorter length groups is mainly due to a loss of an unknown number of fibres. This may lead to the conclusion that gibberellic acid has transferred the short fibres, or at least part of them, into the longer length groups by increasing their length, while it has no effect on the long length groups because if it had had the same effect as on the short fibres a new length group longer than the longest length group in the control sample

would have appeared.

The total number of fibres in all the different length groups ( longer than 19 mm. ) has been calculated from the knowledge of the weight per fibre and the total weight of fibres ( longer than 19 mm. ) per seed. The mean value of fibre weight for these length groups is  $501 \times 10^{-8}$  g. and  $472 \times 10^{-8}$  g. for the control and the treated samples respectively. The mean value of the total weight of these length groups per seed is 0.0732 g. and 0.0816 g. for the control and the treated samples respectively. The ratio of the number of fibres ( longer than 19 mm. ) per seed in the treated sample to that in the control sample is 118 : 100 which means that the number of fibres in these length groups in the treated sample is 18 % higher than in the control sample. Thus the decrease in the percentage by weight of the length groups shorter than 19 mm. in the treated sample ( 7.7 and 5.6 % in the control and the treated samples respectively ) cannot be explained wholly by the loss of some of their fibres which are shifted to the longer length groups. The only explanation can be the emergence, under the influence of gibberellic acid, of additional fibres from epidermal cells, which normally do not grow to produce fibres. Some of these additional fibres may appear in the longer length groups thus making up the additional 18 % and others replace, to some extent, those which have shifted

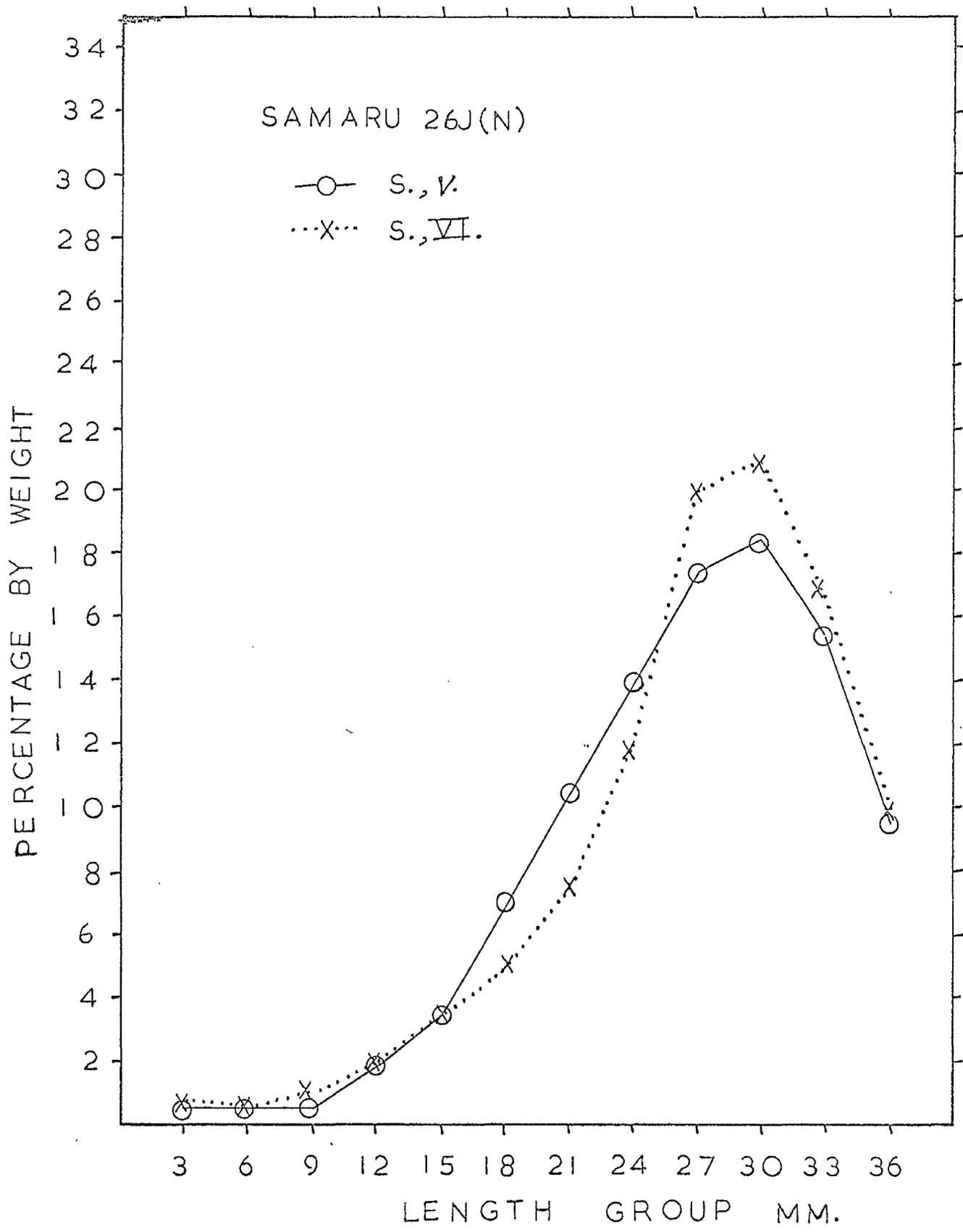


Fig. 5 Fibre length distribution

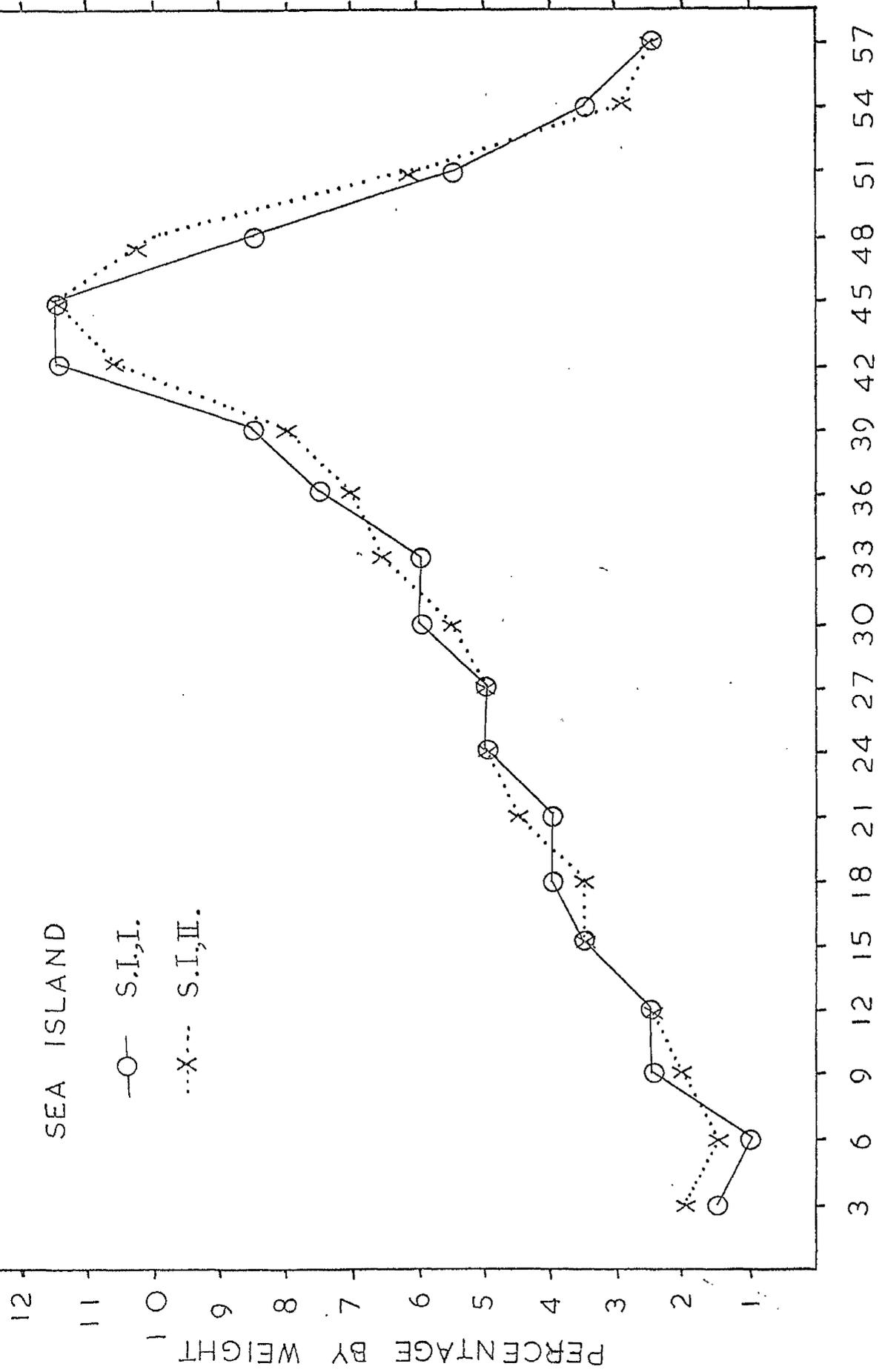


Fig. 6 Fibre length distribution

to longer lengths.

Comparing the length distribution in the control sample of Samaru 26J (N) ( S., V. ) with that of the treated sample ( S., VI. ), Figure 5 , it is found that percentage by weight of the four shortest groups is approximately equal for the two samples, in the next three groups the percentage by weight is smaller in the treated sample, and in the four longest length groups the percentage by weight is greater in the treated sample. The difference in the length distribution between the control and the treated samples is, to some extent, similar, but smaller in magnitude than that in the Rex variety.

For Sea Island, Figure 6 , the length distribution for the treated sample ( S.I., II. ) is nearly the same as that for the control sample ( S.I., I. )

From the previous investigation of fibre length and fibre length distribution it can be assumed that the effect of gibberellic acid is to accelerate the growth in length of the short fibres pushing them to longer lengths and in the mean-time increasing the total number of fibres per seed presumably by stimulating more epidermal cells on the seed coat to grow and produce fibres. This increase in the length of the short fibres and the emergence of additional fibres, coupled with the differences brought about

by gibberellic acid treatment on the weight per fibre of the different length groups, are responsible for the modification of the mean length and the length distribution within a sample. This effect of gibberellic acid on short fibres and the increased number of fibre cells is in agreement with reported effect of gibberellic acid on several plants.<sup>166</sup>

The magnitude of the effect of gibberellic acid on cotton fibre length and length distribution is dependent on two factors; these are the cotton variety and the growing conditions or environment. Gibberellic acid may be more effective on short cottons than on long cottons, and in poor growing conditions than in optimum growing conditions. The role of variety is apparent from the positive effect of gibberellic acid on the Rex variety and the negative effect on Sea Island, when the two cottons were grown side by side in the same environment which of course was not the optimum for either. The role of environment is apparent from the positive effect on Samaru 26J (G) grown in the poor environment of Glasgow and the small or zero effect on Samaru 26J (N) grown under its normal environment.

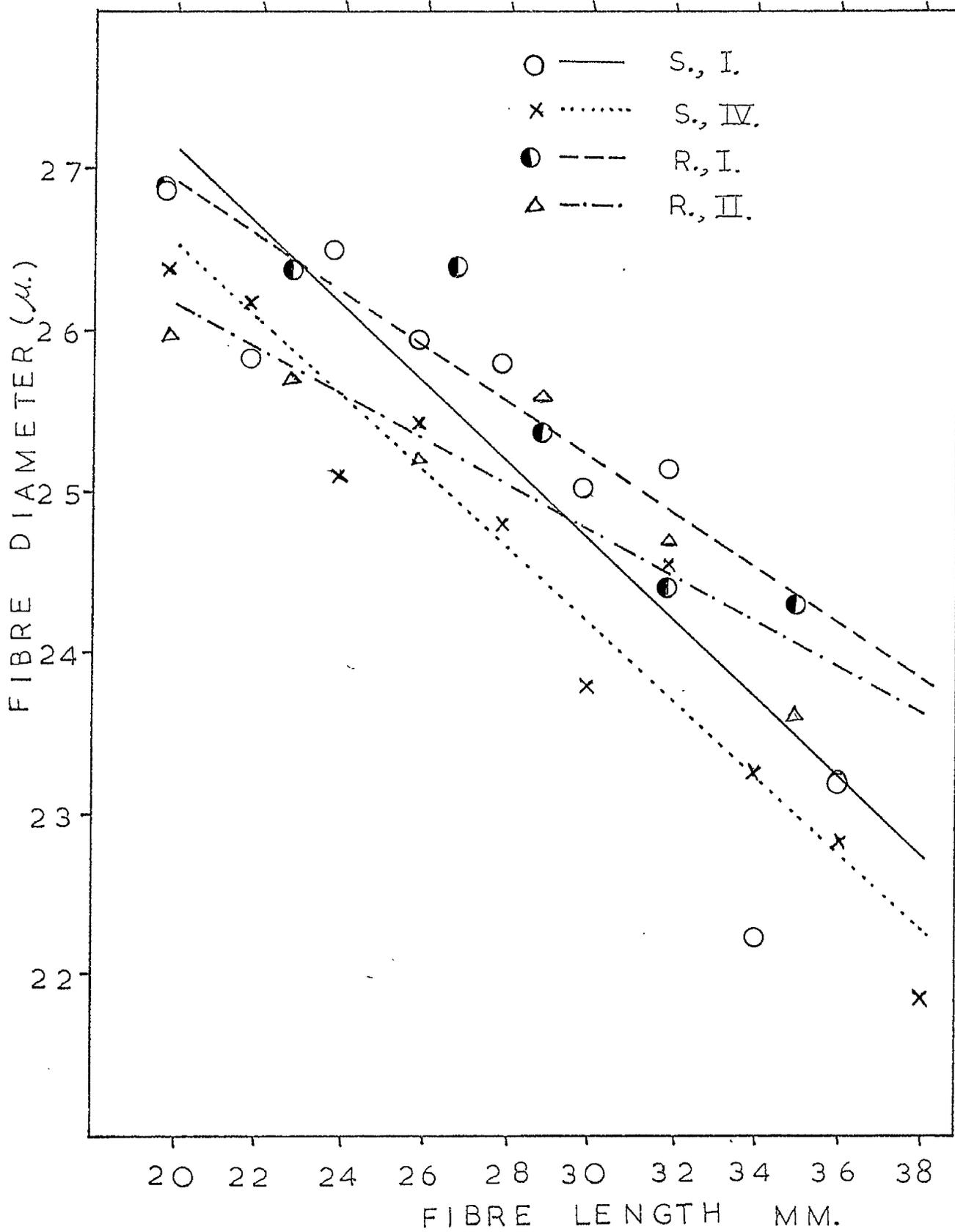


Fig. 7 Fibre diameter for the different fibre length groups

## FIBRE DIAMETER

The importance of fibre diameter has always been overshadowed by fibre length because of the difficulty in measuring fibre diameter and because, generally speaking, there is a negative correlation between fibre diameter and length. This property has been investigated in this work in order to find the relation between fibre length and fibre diameter within a sample, as well as to find the effect of gibberellic acid on this property. The investigation has not been carried out to cover a wide range of different cottons because this is outside the scope of this investigation, however, it does deserve a careful investigation to reveal the role of fibre diameter in fibre fine structure and mechanical properties ( these will be discussed later.)

Detailed results are given in Table 2 on page 71 and are summarized in Figure 7. From Figure 7, it is clear that a close relationship exists between fibre length and fibre diameter within each sample. Within a variety or a sample, long fibres have smaller diameter, and as fibre length decreases fibre diameter increases steadily. The correlation factors between fibre length and fibre diameter within each sample are:

for	( S., I. )	$r = 0.868$
	( S., IV. )	$r = 0.962$
	( R., I. )	$r = 0.852$
	( R., II. )	$r = 0.894$

This relationship does not hold between varieties: in figure 7 the regression lines for Samaru 26J (G) and Rex varieties are separate, fibres of equal length but belonging to different varieties may have different diameters ( see table 8 )

TABLE 8.

Variety	Sample	Fibre diameter for fibres
		35 mm. long ( $\mu$ )
Samaru 26J (G)	S., I.	23.5
	S., IV.	23.0
Rex	R., I.	24.4
	R., II.	24.3
Sea Island	S.I., I.	18.9
	S.I., II.	18.8

Treatment with gibberellic acid has resulted in a small decrease in fibre diameter ( Figure 7 ). It is well known that the full fibre diameter is reached by the fibre during the early days of fibre initiation and is controlled by genetical factors. Therefore it is not expected that gibberellic acid would have had any effect whatsoever on fibre diameter, since the treatment took place on the third day after flowering, i.e. after the young

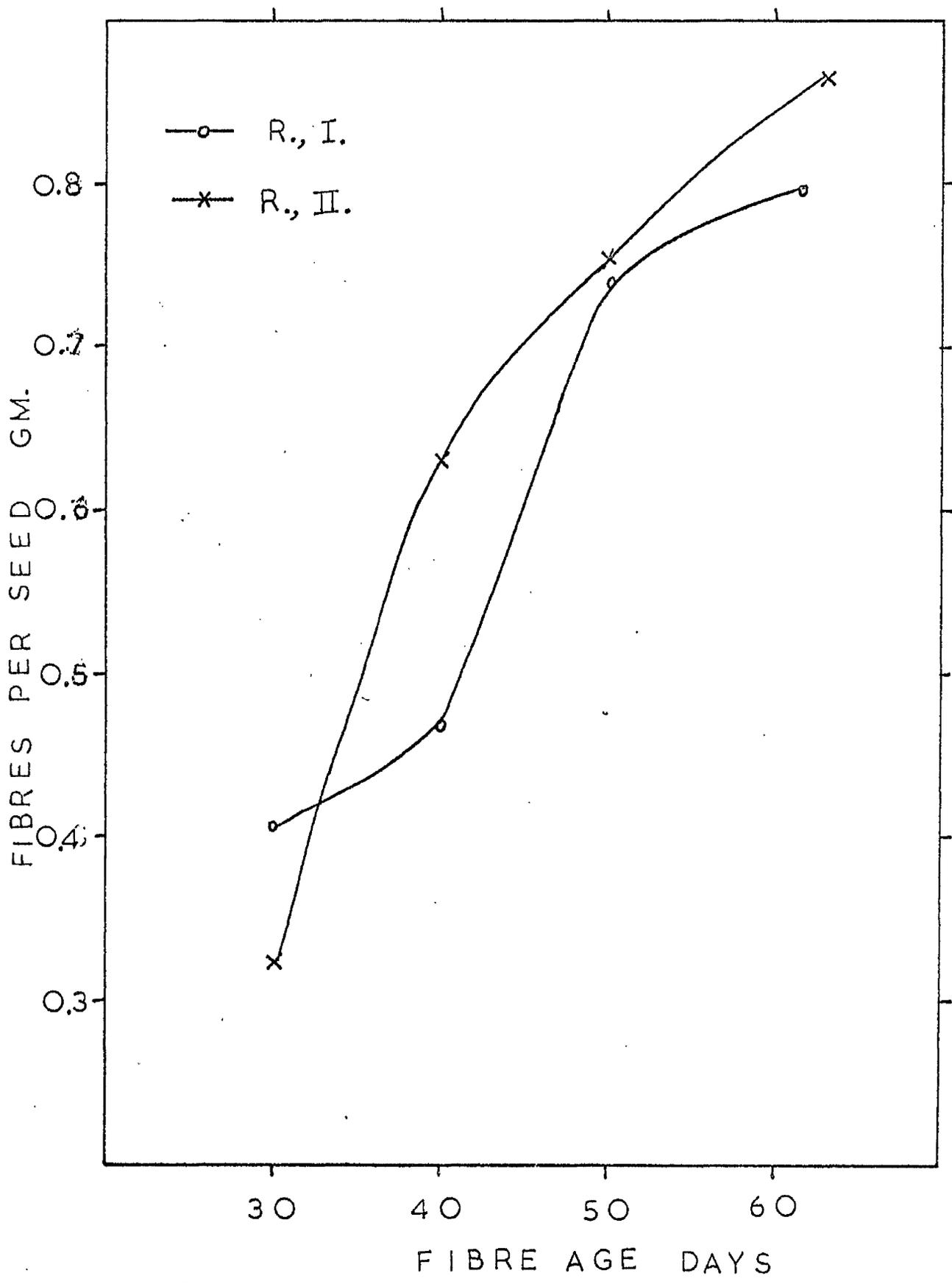


Fig. 8 The rate of cellulose deposition

tubular cells had emerged from the seed coat. However, it is noteworthy that several bolls were treated on each plant which means that for all the bolls, apart from the first one, the effect of gibberellic acid would possibly be felt earlier than the actual treatment of the boll. There is also the possibility that the increase in fibre length which, if it is to be compared to a simple stretching process, might result in a small decrease in the diameter.

#### FIBRE WEIGHT AND FIBRE LINEAR DENSITY

Detailed results are given in tables 3,4 on pages 73-76 and are summarized in figures 8 - 12.

##### (A) The rate of cellulose deposition

The rate of cellulose deposition has been examined for the Rex variety ( R., I. and R., II. ), by taking the total weight of fibres per seed at successive growth stages. Figure 8 shows a marked difference in the shape of the curve between the control ( R., I. ) and the treated ( R., II. ) samples. For the control sample, the curve has an ( S ) shape, with an initial part of slow rate of cellulose deposition, followed by a steeper part during the period 40 to 50 days after flowering, in which most of the cellulose deposition took place, and finally the third part of slow rate of cellulose deposition until it ceased. The curve for the treated sample shows a steeper initial part

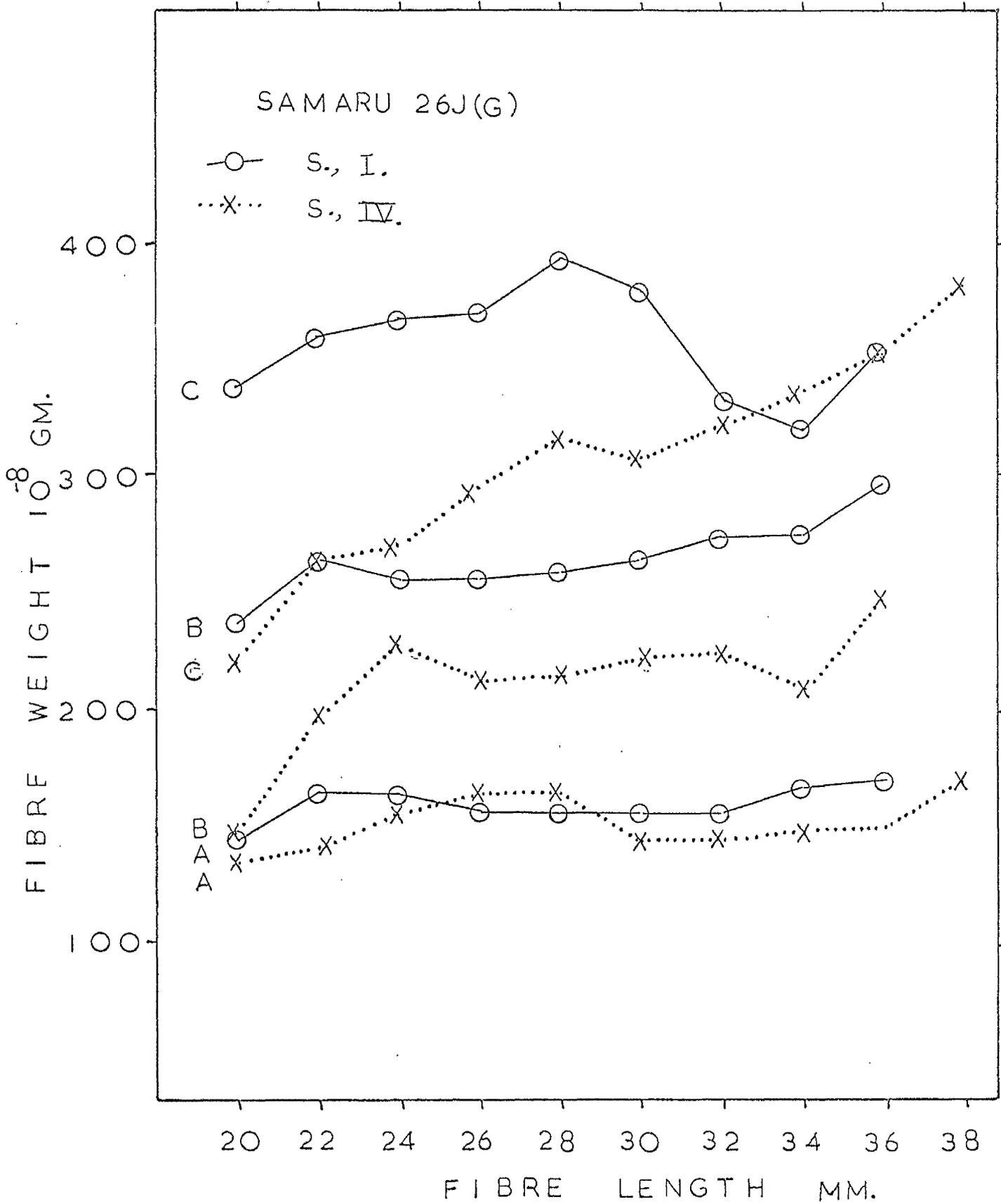


Fig. 9 Fibre weight for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 50 days old

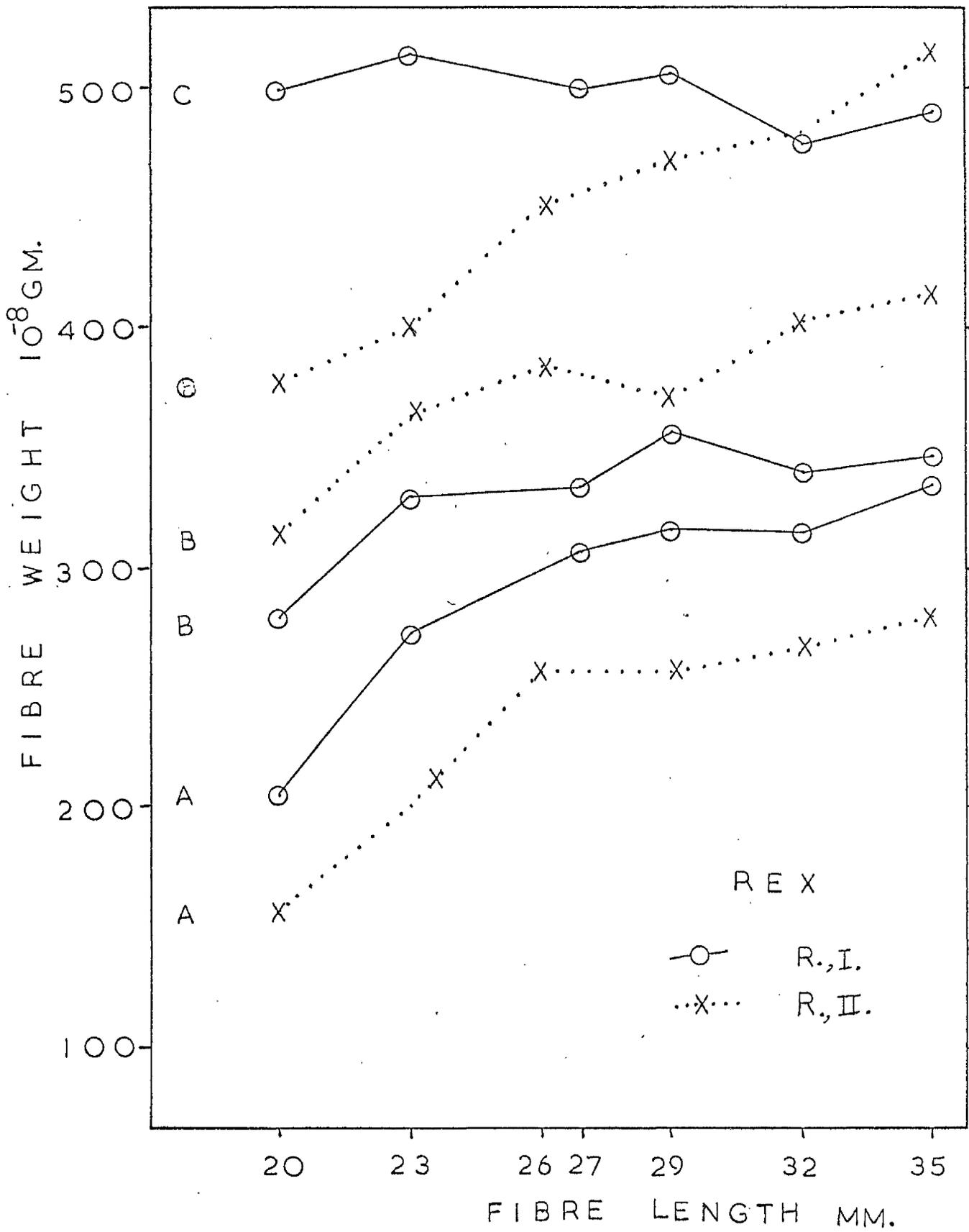


Fig. 10 Fibre weight for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 60 days old

in which more than half the cellulose was deposited. This first stage covered the period 30 to 40 days after flowering. This stage was followed by another one, during the period 40 to 63 days after flowering, in which the rate of cellulose deposition fell off steadily and slowly until it ceased. At 30 days after flowering, the total weight of fibres per seed was smaller in the treated sample than in the control one, which could mean that cellulose deposition started earlier in the control sample. It is also possible that cellulose deposition ceased earlier in the control sample ( the bolls opened on the average 62 and 63 days after flowering in the control and the treated samples respectively).

The rate of cellulose deposition per fibre ( for the mean fibre weight of the sample ) will be similar to the rate of total cellulose deposition per seed, but the rate of cellulose deposition per fibre for each length group will be similar to the rate of cellulose deposition per seed only if the fibre-weight/length-group distribution is similar throughout the cellulose deposition period, which is not always true.

### (B) Fibre weight

Fibre-weight/length-group distribution for fibres of different ages of both Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R.,II. ) varieties are shown in Figures 9,10. The fibre-weight/length-group distribution for the mature fibres

of Samaru 26J (N) ( S., V. and S., VI. ) shows nearly the same pattern as that in the mature 60 days old control sample ( S., I. ). The fibre weight for the control sample ( S., V. ) ranges between 404 and 439 X 10<sup>-8</sup> g., and that for the treated sample ( S., VI. ) ranges between 403 and 452 X 10<sup>-8</sup> g.

From figures 9,10, it is apparent that the distribution among fibres of different lengths of the cellulose to be deposited in the secondary wall is a complex matter. Fibres 30 days old of the Samaru 26J (G) variety ( S., I. and S., IV. ) show a nearly equal fibre weight and this suggests that in the early days of cellulose deposition every fibre receives the same amount of cellulose regardless of its length. This could be explained by the fact that each fibre originates from a single epidermal cell of the seed coat. However, one should take into account the fact that at this early stage of growth the primary wall constitutes a sizeable proportion of total fibre weight. If this manner of distribution of cellulose among fibres of different length groups is maintained throughout the period of cellulose deposition, the final result could be mature fibres of equal fibre weight regardless of fibre length, as is nearly the case in the control sample of Rex ( R., I. ). But if this process is affected by fibre location on the seed coat, with the possibility that fibres in some locations could be in a position to receive more cellulose

than fibres in other locations, then we might expect some fibre length groups to possess more or less fibre weight than others. This is apparent in the Samaru 26J (G) variety ( S., I. ), in which fibres of the extreme length groups, which may possibly have been located on the chalazal and micropylar ends of the seed, possess smaller fibre weight than the remainder of the fibres which are located on the middle portion of the seed. This difference in cellulose distribution among different length groups from one variety to another may explain some of the contrasting findings of the linear-density/length-group relationship reported by several authors.

The effect of gibberellic acid seems to be of an organizing character, and gibberellic acid is a growth regulator substance, it maintains a distribution of cellulose among fibres of different lengths in which short fibres receive smaller amounts of cellulose than long fibres or in other words every fibre receives approximately an amount of cellulose according to its length. As a result of this regulating effect, mature fibres of both Samaru 26J (G) ( S., IV. ) and Rex ( R., II. ) varieties, figures 9, 10, show fibre weight to be dependent on fibre length with short fibres having smaller fibre weight than long ones.

Another effect of gibberellic acid on cellulose deposition in Samaru 26J (G) ( S., IV. ) and Rex ( R., II. ) varieties is

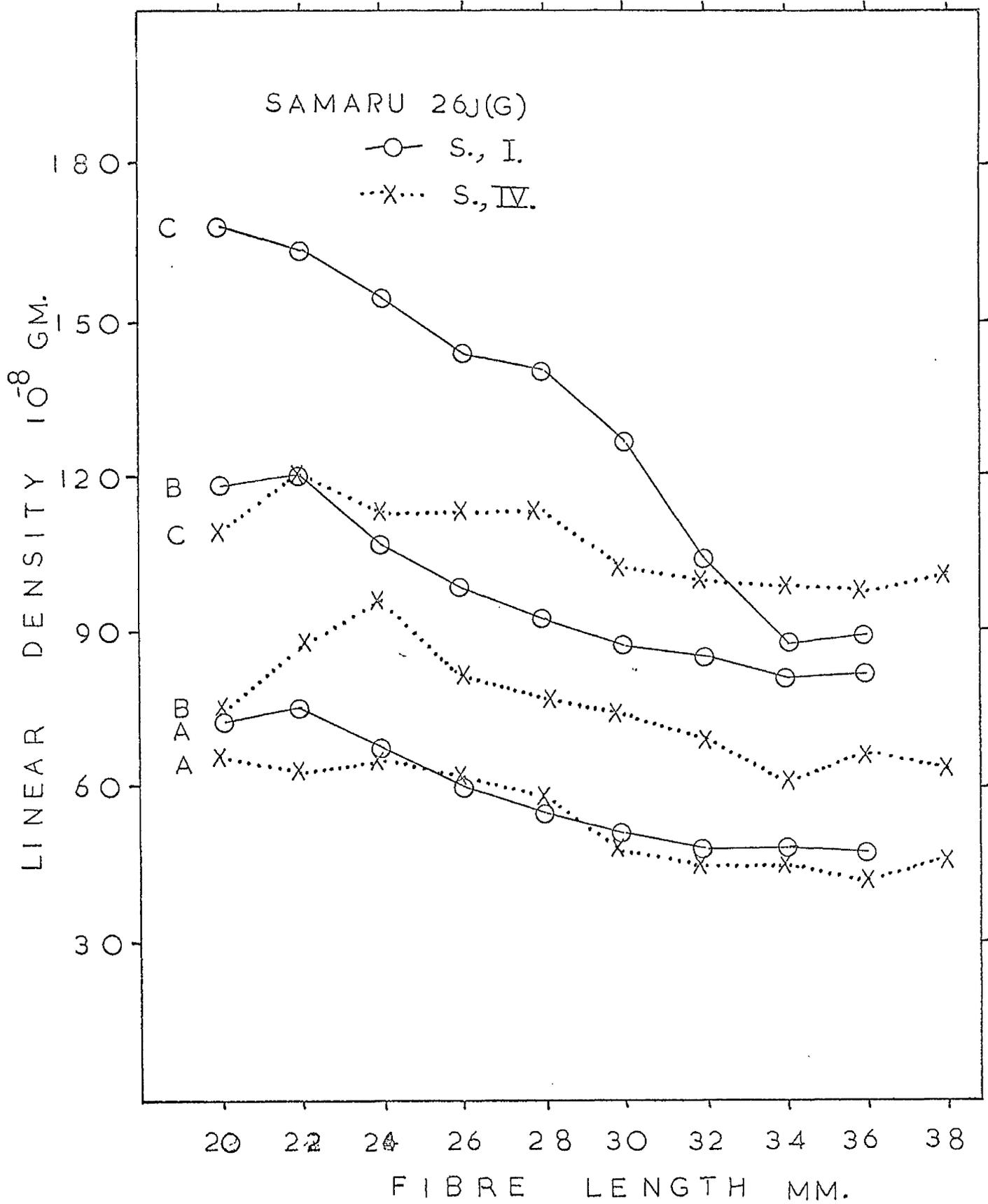


Fig. 11 Fibre linear density for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 50 days old

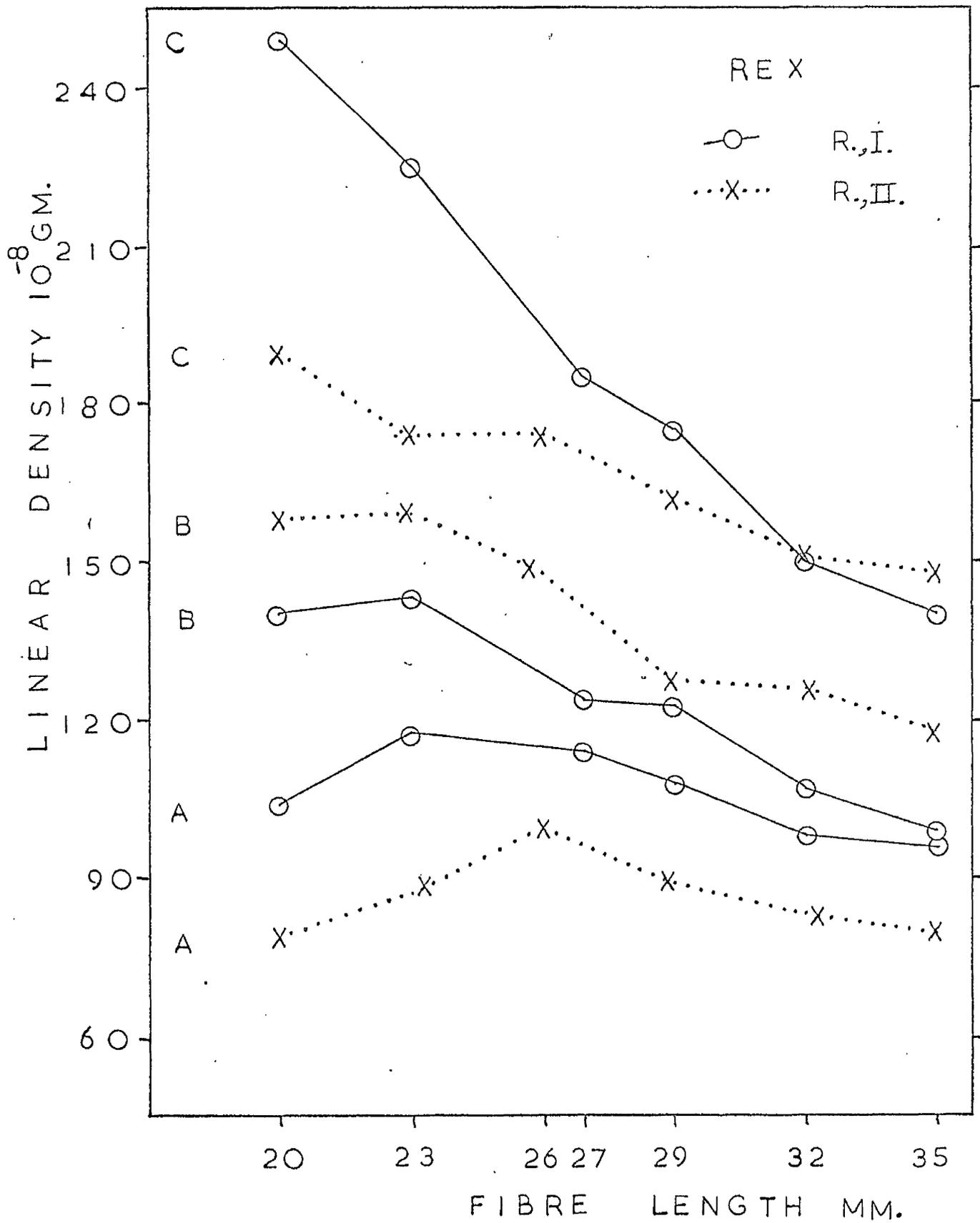


Fig. 12 Fibre linear density for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 60 days old

the appreciable reduction in the fibre weight, and consequently the linear density. The mean value of fibre weight for the mature Rex variety falls from  $501 \times 10^{-8}$  g. in the control sample ( R., I. ) to  $472 \times 10^{-8}$  g. in the treated sample ( R., II. ). The reason for this loss in fibre weight cannot be a loss in the total weight of fibres per seed which is actually higher in the treated sample than in the control one ( Figure 28 ). The only explanation is the possible increase in the number of fibres per seed in the treated sample.

### (C) Fibre linear density

The linear density of the different length groups of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties are shown in figures 11, 12. Fibre linear density varies according to fibre weight in the different length groups of the different samples. Because of the difference in fibre weight between the control and treated samples, this difference is transmitted to the linear density. For Samaru 26J (G), see figure 11, in the control sample, the linear density increases as fibre length decreases, while in the treated sample the linear density shows almost the same value for the different length groups. For the Rex variety, see figure 12, in the control sample the linear density increases steadily as fibre length decreases, while in the treated sample the linear density is somewhat higher for

TABLE 9 . Fibre maturity of the different length groups of Rex.

Sample	Fibre length mm.	Fibre density $\text{g cm}^{-3}$	Polarized light method		Sodium hydroxide swelling method			Mat.factor
			Immature	Mature	D	T	N	
			%	%	%	%	%	
R., I.	35	0.952	21	79	6	29	65	0.995
	32	1.000	19	81	5	41	54	0.947
	29	1.087	15	85	5	40	55	0.950
	27	1.063	11	89	6	37	57	0.955
	23	1.293	13	87	3	28	69	1.031
	20	1.377	9	91	3	26	71	1.040
R., II.	35	1.065	16	84	4	36	60	0.980
	32	0.993	17	83	5	42	53	0.937
	29	0.988	17	83	5	33	62	0.984
	26	1.094	21	79	7	32	61	0.970
	23	1.048	18	82	6	31	63	0.981
	20	1.118	21	79	8	29	63	0.975

the longest length groups. The mean values of the linear density for the control and treated samples are  $178$  and  $157 \times 10^{-8}$  g. respectively.

#### FIBRE MATURITY

Fibre maturity was measured by three methods. The polarized light method differentiates broadly between two groups of fibres: the immature and the mature. The sodium hydroxide method sorts out the sample into three groups according to the degree of wall thickening, and the maturity ratio is a fair measure of the distribution of these three groups. The fibre density method makes use of fibre diameter and linear density and gives a mean value of the degree of filling of the cell tube with cellulose. This method has the advantage of being applicable to all stages of fibre growth.

The results obtained for Rex variety, table 9, show that in the polarized light method, the percentage of immature fibres follows the same trend as the percentage of the dead fibres in the sodium hydroxide swelling method. The higher percentages given by the polarized light method occur because it classes as immature, fibres which are classed as dead plus some which are classed as thin-walled by the sodium hydroxide method. It is difficult to compare the fibre density measurements with the results of the other two methods, but they all show the same trend for the

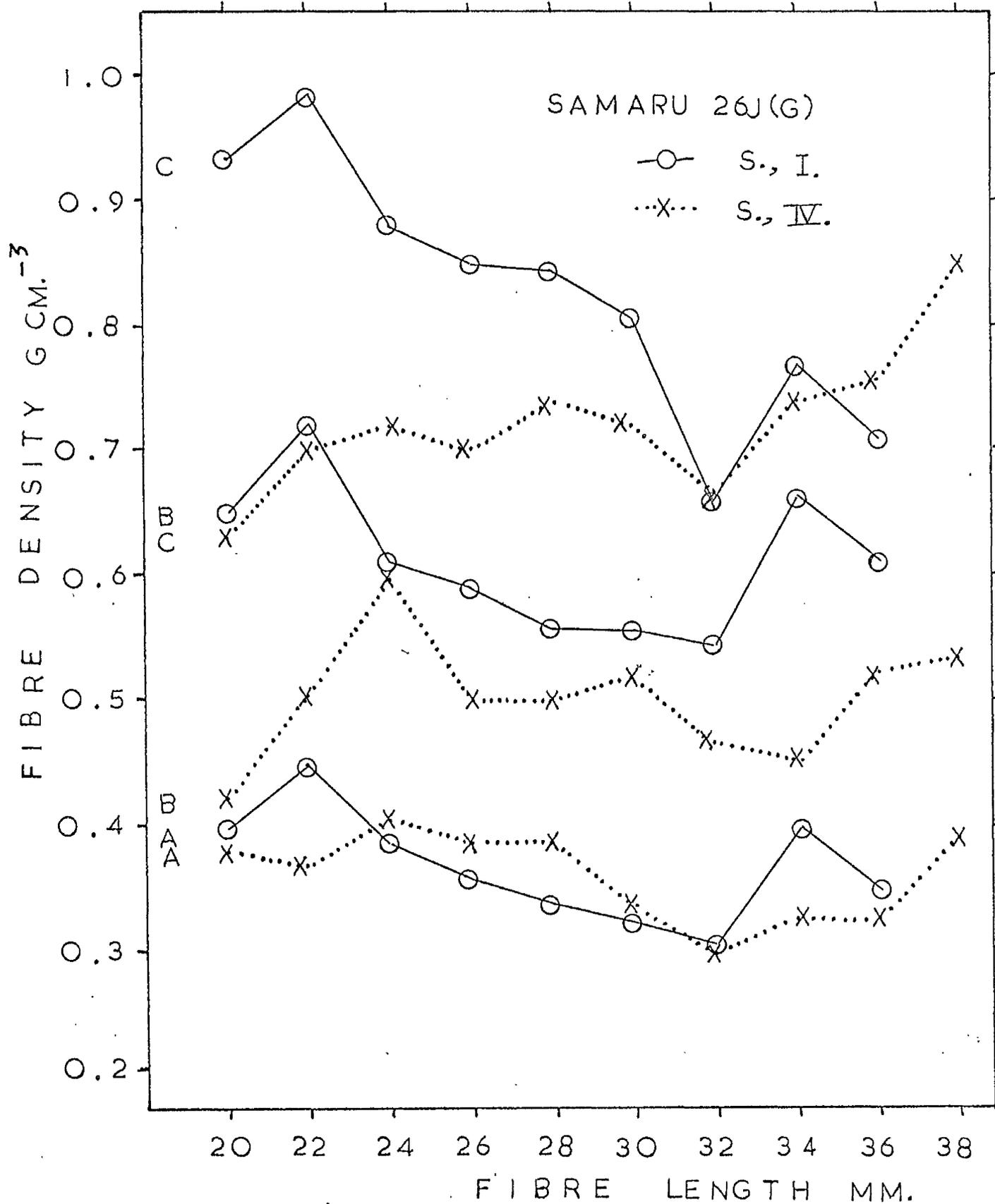


Fig. 13 Fibre density for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 50 days old

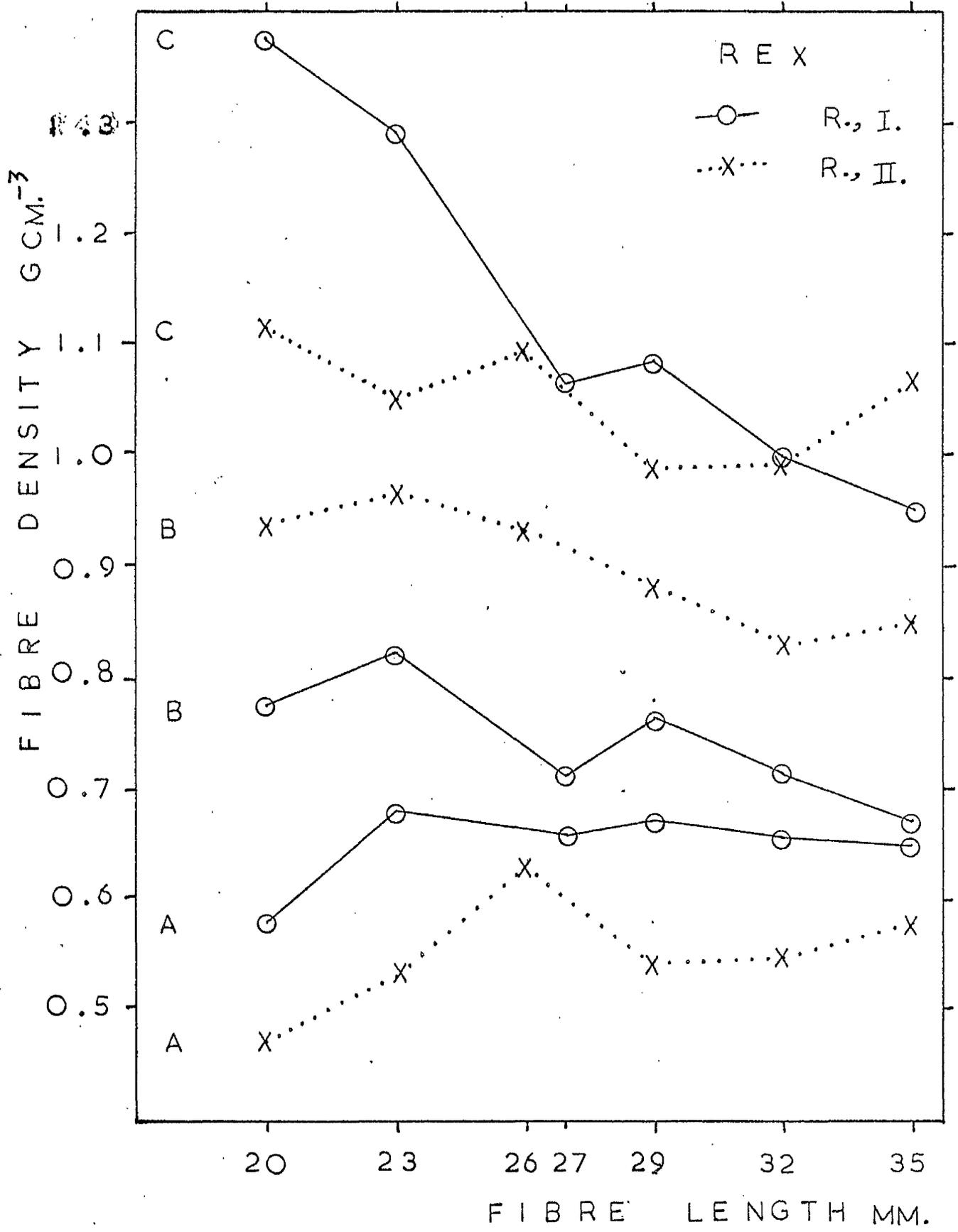


Fig. 14 Fibre density for fibres of different ages  
 (A) 30 days old (B) 40 days old (C) 60 days old

different length groups of the control sample.

Generally speaking, the percentage of the dead fibres and that of the immature fibres shows a tendency to decrease as fibre length decreases in the control sample, while in the treated sample there is a less marked tendency of these percentages to increase as fibre length decreases. The mean percentages of immature fibres in the control and treated samples are 14.5 and 17.5 % respectively. This 3 % increase in the immature fibres in the treated sample is responsible in some part for its lower linear density ( 12 % lower than the control sample ). The values of fibre density of the control sample show a tendency to increase as fibre length decreases, while in the treated sample this tendency is apparent only for the short length groups.

Fibre density has been calculated for fibres of different length groups of three successive ages of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties, in order to compare the degree of the filling of the cell tube in the different length groups as fibre growth progressed. The results obtained, figures 13,14, show that this is dependent on the linear density since fibre diameter is constant for the successive ages.

In figure 13 , the control mature fibres ( 50 days old ) of Samaru 26J (G) variety, show a decrease in fibre density as fibre length increases while the treated sample shows the opposite.

In figure 14, the control mature fibres ( 60 days old ) of the Rex variety, show a marked decrease in fibre density as fibre length increases, while the treated sample shows more or less equal fibre density for the different length groups. This difference in fibre density between the control and treated samples is related to differences in fibre diameter and linear density distributions. In the treated sample, fibre diameter increases as fibre length decreases and the linear density is nearly equal for all the length groups, this results in a decrease in fibre density with the decrease in fibre length. In the control sample, however, fibre diameter increases as fibre length decreases in the same manner as in the treated sample, but the linear density of the short fibres is much higher than that of the long fibres so that the increase in fibre diameter, merely reduces the slope appreciably, but does not alter its direction.

In comparing the results of the linear density with that of the fibre density of the control mature samples of the Samaru 26J (G) and Rex varieties, it is interesting to notice that while the linear densities of the shortest length groups are 69 % and 78 % higher than that of the longest length groups in Samaru 26J (G) and Rex respectively, the differences in fibre density are only 27 % and 40 %. This is understandable because of the increase in fibre diameter as fibre length decreases within a sample or variety.

TABLE 10 . The average and the actual convolution angles of fibres of different fibre densities.

Sample	Fibre age (days)	Fibre density ( $\text{g cm}^{-3}$ )	Ribbon width ( $\mu$ )	No. of convol. per mm.	Average convol. angle ( $\phi^{\circ}$ )	Actual convol. angle ( $\phi'^{\circ}$ )
S., I.	30	0.334	24.9	3.44	7.65	29.8
	40	0.558	22.5	3.74	7.60	28.5
	50	0.843	21.6	4.82	9.30	27.6
S., IV.	30	0.383	26.2	2.60	6.10	31.2
	40	0.500	22.9	3.55	7.25	28.9
	50	0.735	20.9	4.03	7.55	26.8
R., I.	30	0.655	24.6	3.66	8.05	29.6
	40	0.712	23.2	5.15	10.65	28.9
	60	1.063	22.4	4.90	9.75	30.8
R., II.	30	0.543	25.7	2.96	6.85	27.7
	40	0.780	23.6	5.10	10.70	27.6
	60	0.988	21.1	5.65	10.60	28.9

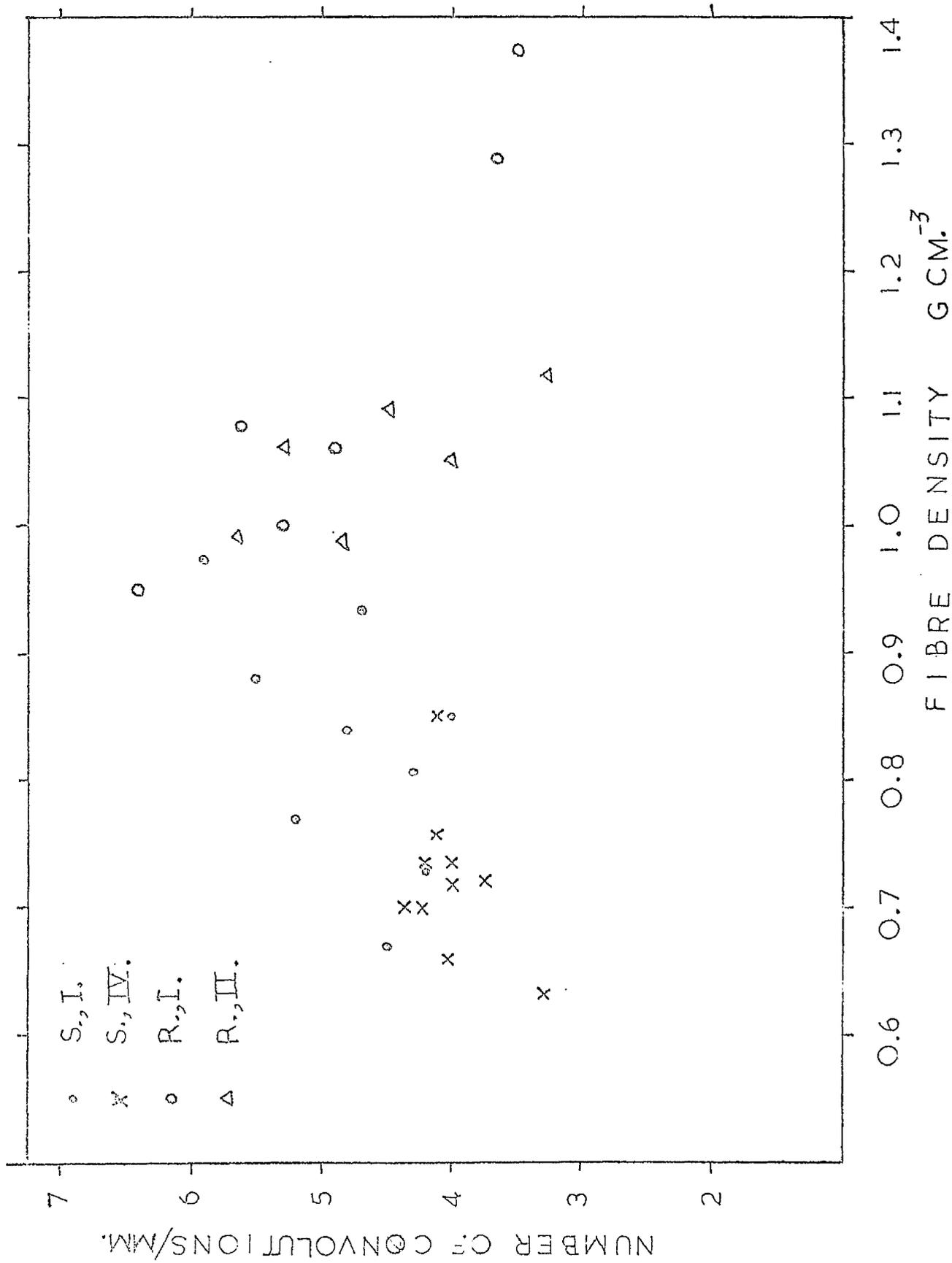


Fig. 15 The number of convolutions per mm. for fibres of different densities

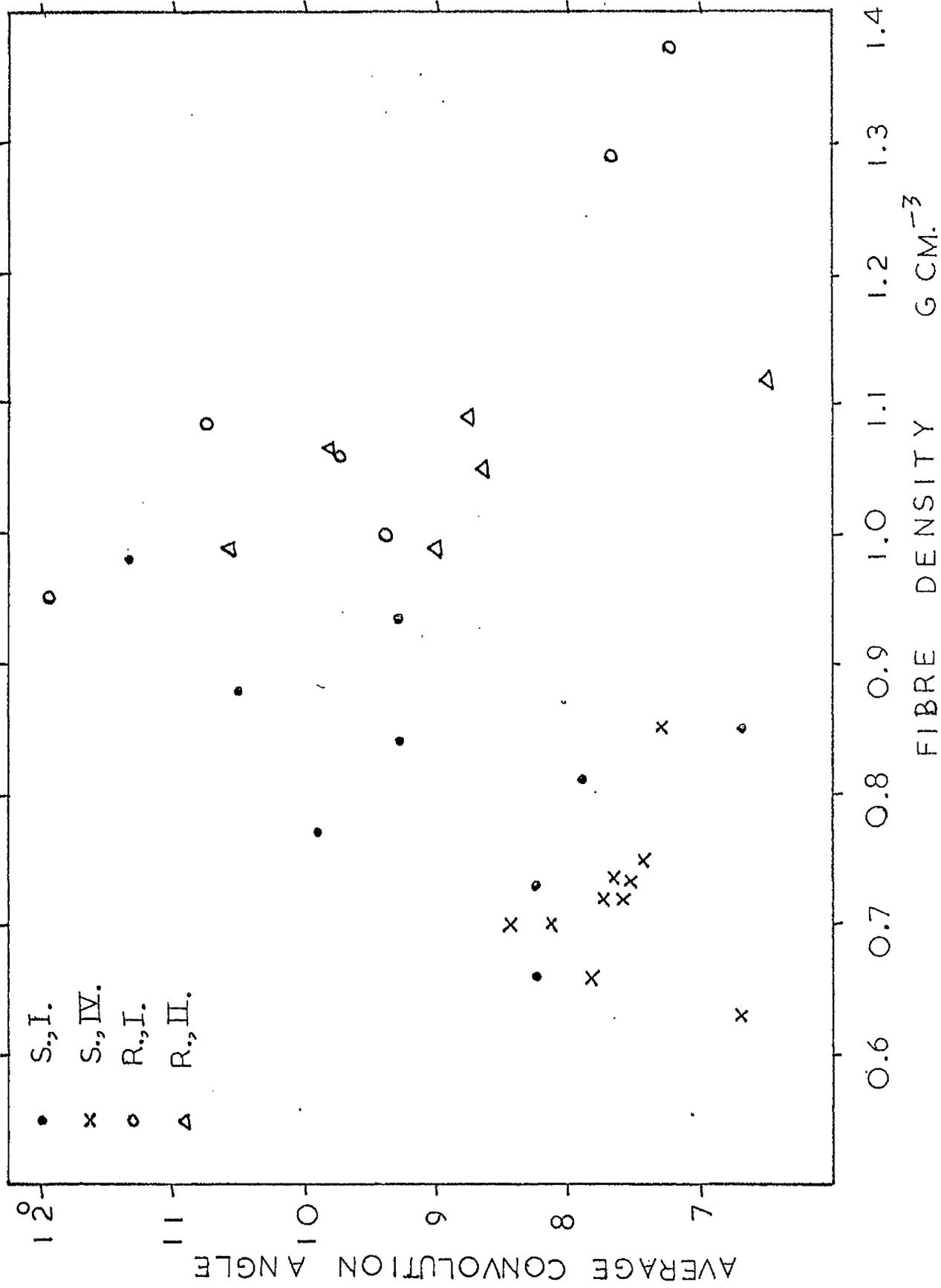


Fig. 16 The average convolution angle for fibres of different densities

## THE CONVOLUTIONS AND THE CONVOLUTION ANGLE

In recent years, the convolutions have attracted much attention because of their possible effect on cellulose orientation, or on the orientation results as measured by the X-ray diffraction method. The possible relationship between the convolutions and the convolution angle and the orientation, as well as the possible effect of the convolutions on fibre extensibility, made it important to find the number of convolutions and the convolution angle for the samples under investigation, and also desirable to carry out a general investigation into this peculiar characteristic of cotton fibres. The relationship between the convolutions and the orientation, as measured either by optical or X-ray diffraction methods, and between the convolutions and the mechanical properties will be discussed later.

The number of convolutions per mm. and the average convolution angle for fibres of different ages and different length groups within the mature samples, of Samaru 26J (G) and Rex varieties are shown in detail in table 2, and are summarized in table 6 and figures 15,16. The number of convolutions per mm. and the average convolution angle are higher in almost all the control sample of Samaru 26J (G) than in the corresponding treated ones ( tables 2,10 ). For the Rex variety, there is no definite difference between the control and treated samples in the number

of convolutions per mm. in the different length groups; nor is there any difference between the two samples in the relationship between convolutions per mm. and degree of maturity ( tables 2,10 )

The average convolution angle is higher for the different length groups within the control sample than in the corresponding groups for the treated sample ( table 2 ) because of differences in ribbon width. For samples of different ages, the control samples of lower and higher degrees of maturity ( 30 and 60 days old ) show higher average convolution angle and samples of intermediate degree of maturity shows no difference ( table 10 ).

It appears that the effect of gibberellic acid treatment on the number of convolutions per mm. and the average convolution angle may be indirect and results mainly from its effect on the degree of wall thickening and the distribution of fibres with different degrees of wall thickening within a sample. In order to examine this indirect effect the frequency of the convolutions and the average convolution angle of the control and treated samples as a whole have been investigated from the point of view of their relationship with fibre maturity as provided by fibres of different ages, as well as fibres with different degrees of maturity within one of the "mature" samples. Also these characteristics have been investigated on fibres of different length groups and different fibre densities and the change in the number of

convolutions and the convolution angle caused by mercerization at constant length has been examined.

The results obtained for fibres of different ages, but of the same length ( equal to the mean length of the test sample ) of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties are shown in table 10 . From these results it is clear that the number of convolutions per mm. increases as fibre age, and consequently fibre density and maturity, increaseS, while the ribbon width shows a gradual decrease. The average convolution angle shows an increase as fibre age increases, except in the case of Rex variety in which the fibres 60 days old show a smaller average convolution angle than fibres 40 days old. The actual convolution angle does not follow the same trend as the average convolution angle, because, although the ribbon width is closely associated with wall thickening, the convolution length is more affected by the distribution of the convolutions along fibre length.

To examine the relationship between fibre maturity and the number of convolutions and the average convolution angle within a sample, sixty fibres ( ten of each length group ) of the mature sample of the Rex variety ( R., I. ) were analyzed according to their maturity under the polarizing microscope and sorted out into three groups; immature, mature, and very mature fibres. From the

results obtained, table 11, it is clear that, within a sample, the number of convolutions and the average convolution angle are much lower in immature and very mature fibres than in fibres with intermediate degrees of maturity, while the ribbon width decreases steadily as the fibres become more mature.

TABLE 11.

	Immature fibres	Mature fibres	Very mature fibres	Mean
Ribbon width ( $\mu$ )	24.0	21.5	17.8	21.5
No. of Conv./mm.	1.68	6.04	1.77	4.6
Conv. pitch (mm.)	0.595	0.166	0.566	0.217
Average Conv. angle ( $\phi^\circ$ )	3.65	11.5	2.85	8.85

The relationship between fibre density and the number of convolutions per mm. and the average convolution angle was examined for the different length groups of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties. For Samaru 26J (G), figure 15, the number of convolutions per mm. increases as fibre density increases with correlation factor  $r = 0.755$ , with the maximum number of convolutions shown by the sample of maximum density. The Rex variety shows the reverse, the number of convolutions

decreases as fibre density increases. Since Samaru 26J(G) has a lower fibre density than Rex, when the two cottons are brought together, figure 15, the number of convolutions is found to increase as fibre density increases until it reaches a maximum at fibre density  $0.9 - 1.1 \text{ g cm}^{-3}$ , after which it decreases for further increase in fibre density.

The average convolution angle has been found, for the two varieties taken together, to increase as fibre density increases until it reaches a maximum after which it decreases for further increase in fibre density. This relationship is apparent in figure 16, but the points are fairly scattered. This scattering can be explained by the following:

(a) In any cotton variety or sample, the fibres show a distribution of the degree of wall thickening. As a result, and because both the immature and the very mature fibres possess fewer convolutions than the fibres of intermediate degree of maturity ( see table 11 ), two varieties or samples may have two different fibre densities and equal numbers of convolutions.

(b) The convolution angle depends on two variables, the ribbon width and the convolution pitch. Both these variables depend on the degree of wall thickening or fibre density. The number of convolutions per mm. increases as fibre density increases until a certain limit after which it will decrease for any further increase

TABLE 12 . The number of convolutions and the average convolution angle of mercerized fibres of different ages.

Sample	Fibre age (days)	Ribbon width ( $\mu$ )	No. of convol. per mm.	Average conv. angle ( $\phi^\circ$ )
	30	20.3	3.23	5.85
S., I.	40	14.8	4.80	6.35
	50	14.6	1.82	2.40
	30	20.1	3.10	5.50
S., IV.	40	17.0	3.22	4.90
	50	14.7	3.26	4.30
	30	15.4	4.22	5.85
R., I.	40	14.8	3.83	5.10
	60	17.4	0.61	0.95
	30	19.4	2.60	4.40
R., II.	40	15.4	3.50	4.85
	60	16.7	1.22	1.85

in fibre density: the convolution pitch will vary in the reverse way, it will decrease as fibre density increases until it reaches a minimum when the number of convolutions reaches the maximum, after that it will increase for any further increase in fibre density as the number of convolutions decreases until it reaches the infinity when the fibres do not convolute at all. The second variable, which is the ribbon width, decreases as fibre density increases and will reach a constant value for the highly mature fibres. As a result of the dependence of these two variables on fibre density, we should expect the average convolution angle to be sensitive to any change in fibre density, and we may assume that it will increase as fibre density increases until a certain limit after which it will decrease for any further increase in fibre density, however, this could be modified from sample to sample by the pattern of distribution of fibre densities within each sample.

Mercerization at constant length ( table 12 ) does not remove the convolutions completely, in fact the number of convolutions has increased in some immature samples and shows no change in others, but only the mature samples with a high fibre density show an appreciable reduction in the number of convolutions. The average convolution angle is always smaller because of the appreciable decrease in the ribbon width.

CHAPTER IV.

COTTON FIBRE FINE STRUCTURE

TABLE 13. The crystallite and the overall molecular orientation

Sample	Fibre age (days)	Fibre length (mm.)	X-ray diffraction			Optical
			50 per cent. angle ( $\psi$ )	40 per cent. cent. angle ( $\gamma$ )	( $\gamma-\phi$ ) ( $\Phi$ )	Av. orient. angle ( $\theta^\circ$ )
	30	28	43.6	47.6	39.9	-
S., I.	40	28	38.1	41.4	33.8	-
	50	28	36.9	40.5	31.2	38.8
	30	28	45.3	48.6	42.5	-
S., IV.	40	28	37.4	40.9	33.6	-
	50	28	35.2	38.4	30.8	38.5
S., V.		28	32.0	35.9	29.1	34.8
S., VI.		28	31.6	35.2	29.7	34.8
	30	27	38.9	43.5	35.5	40.1
R., I.	40	27	40.9	44.6	33.9	39.7
	60	27	38.4	42.5	32.8	39.8
	30	29	41.4	44.7	37.9	40.6
R., II.	40	29	38.4	42.9	32.2	39.5
	60	29	38.3	42.0	31.4	38.5
S.I., I.		35	29.4	33.6	29.2	30.5
S.I., II.		35	29.9	33.6	29.5	30.4

TABLE 14 . The number of reversals, the average orientation angle, and the factor  $\left(\frac{r}{l}\right)$  for fibres of the different length groups.

Sample	Fibre length (mm.)	No. of reversals per mm.	Av. orient. angle ( $\theta^\circ$ )	$\frac{r}{l}$
S., I.	36	2.86	36.6	323
	34	2.35	36.7	327
	32	2.60	37.6	393
	30	2.21	38.5	418
	28	2.84	38.8	461
	26	2.38	39.4	498
	24	2.34	39.9	553
	22	2.20	40.4	587
	20	1.87	40.5	673
S., IV.	38	2.11	35.7	287
	36	2.00	36.2	317
	34	2.04	36.7	341
	32	1.94	37.2	384
	30	1.62	38.0	397
	28	1.76	38.5	443
	26	1.84	39.0	489
	24	1.87	39.4	523

TABLE 14 . ( continued)

	Fibre length (mm.)	No. of reversals per mm.	Av. orient. angle ( $\theta^\circ$ )	$\frac{r}{\tau}$
S., IV.	22	1.75	39.7	595
	20	1.64	40.4	660
S., V.	28	2.44	34.8	462
S., VI.	28	2.26	34.8	462
S.I., I.	35	2.02	30.5	270
S.I., II.	35	1.83	30.4	268
R., I.	35	2.73	37.6	347
	32	2.45	38.4	381
	29	2.31	39.0	438
	27	2.41	39.8	489
	23	2.21	41.0	574
	20	2.07	41.3	672
R., II.	35	2.36	37.6	337
	32	2.24	38.0	386
	29	2.36	38.5	441
	26	2.27	39.7	485
	23	2.00	40.4	559
	20	2.11	40.8	650

TABLE 15 . The average convolution angle and the average orientation angle for fibres of different lengths of four commercial varieties.

Variety	Fibre length	Av. conv.	Av. orient.	
	mm.	angle ( $\phi^\circ$ )	angle ( $\theta^\circ$ )	( $\theta - \phi$ ) ( $^\circ$ )
	48	7.50	27.4	19.90
Sea Island	42	7.20	28.2	21.00
(G. barbadense)	36	5.45	30.5	25.05
	30	5.10	32.0	26.90
	42	7.10	26.0	19.90
Giza 45	36	5.30	28.2	22.90
(G. barbadense)	30	6.20	30.0	23.80
	24	5.15	31.8	26.65
	36	7.25	33.0	25.75
Ashmouni	30	7.20	35.2	28.00
(G. barbadense)	24	6.60	36.1	29.50
	30	5.55	33.8	28.25
Texas	30	5.55	33.8	28.25
(G. hirsutum)	24	5.90	35.0	29.10

## THE CRYSTALLITE ORIENTATION

The crystallite orientation measured by the X-ray diffraction method is possibly modified by the presence of the convolutions. Meredith<sup>136</sup> pointed out that the angle between the long axis of any crystallite and the axis of the collapsed fibre is a function of the angle of convolution of the fibre and the inclination of the crystallite to the axis in the unconvoluted fibre. The analysis of the X-ray diffraction photograph and the calculation of the orientation angle is based on the assumption that the fibres of the sample are perpendicular to the X-ray beam. In the cotton fibre, at the place of a convolution, the fibre is actually inclined to the X-ray beam at an angle equal to the actual convolution angle (  $20 - 30^{\circ}$  ). But since not the whole fibre is convoluted, then the average convolution angle can be taken as a mean value of the actual convolution angle along the fibre length. The real magnitude of the modification of the measured X-ray angle caused by the convolutions is still unknown and is outside the field of this investigation. However, applying Meredith's<sup>136</sup> suggestion of subtracting the average convolution angle from the 40 per cent. X-ray angle, although it is not strictly valid, is helpful.

The results obtained from the X-ray diffraction method,

table 13 , show the following.

(1) The effect of gibberellic acid treatment on crystallite orientation

(a) For fibres of Samaru 26J (G) 40 and 50 days old and for fibres of Rex 40 and 60 days old, treatment with gibberellic acid has caused a small decrease in the orientation angles ( both 40 and 50 per cent. X-ray angles ).

(b) For fibres 30 days old, the X-ray angles are larger in the treated samples of both Samaru 26J (G) and Rex varieties ( S., IV. and R., II. ) than in the control ones ( S., I. and R., I. ).

The explanation of this difference probably lies in the difference in the degree of wall thickening.

(c) A negligible difference in the X-ray angles is found between the control and the treated samples of Samaru 26J (N) and Sea Island.

From these results it is concluded that treatment with gibberellic acid has no direct effect on crystallite orientation. The difference between the control and treated samples of the Rex variety is possibly due to the difference in the fibre lengths of these samples which were 27 and 29 mm. for the control and the treated samples respectively.

(2) The relationship between fibre maturity and the crystallite orientation ( or the crystallite orientation of the successive layers of cellulose in the secondary wall )

It is apparent from table 13 , that both the 40 and 50 per cent.

X-ray angles decrease as the fibre becomes more mature, or as more cellulose layers are laid down in the secondary wall, in both Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties ( see plates 1 - 3 ). These results are not in agreement with Meredith's<sup>136</sup>; he reported that the 40 per cent. X-ray angle tends to increase as the degree of wall thickening increases.

(3) The effect of environmental conditions on the crystallite orientation

It is interesting to notice that Samaru 26J (N) ( both S., V. and S., VI. ) show smaller orientation angles than Samaru 26J (G) ( both S., I. and S., IV. ). This is due to the difference in the environmental conditions under which the plants were grown. This effect is similar to the results found by Berkley et al.<sup>27</sup> and confirms their conclusion that environmental conditions do modify the crystallite orientation. This environmental effect explains the relatively higher values of 40 and 50 per cent. X-ray angles ( table 13 , except that for Samaru 26J (N) ) than those reported by other workers.<sup>25,136</sup>

THE OVERALL MOLECULAR ORIENTATION

The average orientation angle measured optically can be regarded as fairly free from the interference of the convolutions since it was measured only on straight unconvoluted portions of

the fibre. Atsuki and Okajima<sup>7</sup> reported that the higher refractive index ( $n_{11}$ ) decreases as the pitch of convolution decreases and that it will reach the value of the lower refractive index ( $n_1$ ) when the convolution pitch reaches zero, and consequently they concluded that ( $n_{11}$ ) in the unconvoluted fibre may be equal to that of ramie fibres, and the smallness of ( $n_{11}$ ) in cotton fibres is caused by the additive effect of the convolutions on the spiral structure of the fibre. In this work it has been observed that the convoluted portions of the fibre show lower values for ( $n_{11}$ ) than the straight unconvoluted portions. This observation could be explained not by a structural change caused by the convolutions, but by the position of the fibre edge in the convoluted portion. In measuring the higher refractive index ( $n_{11}$ ), the convoluted portion of the fibre will show lower values than the remainder of the fibre because in this convoluted portion the fibre edge is not parallel to the polarized light direction of vibration as it supposed to be but actually inclined at an angle between 20 and 30° which is the actual convolution angle. As the convolution pitch decreases the actual convolution angle increases, and consequently the refractive index ( $n_{11}$ ) at this portion decreases. In the extreme, if the convolution pitch tends towards zero, the actual convolution angle approaches 90°

and the fibre edge is almost perpendicular to the direction of vibration of the polarized light, and therefore the refractive index will tend towards the value of  $(n_1)$  for the unconvoluted portion. When measuring the lower refractive index  $(n_1)$ , the convoluted portion will show higher values than the remainder of the fibre, and these will increase as the actual convolution angle increases and approach the value of  $(n_{11})$  for the unconvoluted portion.

Thus it is concluded that the convolutions may cause faulty measurements unless care be taken. The possible error in measuring  $(n_{11})$  and  $(n_1)$  will be towards smaller values of  $(n_{11})$  and higher values of  $(n_1)$ , especially if the number of convolutions is high and the convoluted portion is great.

(1) The effect of gibberellic acid treatment on molecular orientation

The treatment with gibberellic acid has caused a small decrease in the average orientation angle of Rex variety, this decrease can be attributed to the difference in length ( the sample length was 27 and 29 mm. for the control and the treated samples respectively ) rather than to an effect of gibberellic acid. No effect has been found on Samaru 26J (G), Samaru 26J (N), and Sea Island. The relationship between orientation angle and length is discussed more fully on page 117.

(2) The relation between the average orientation angle and the average convolution angle

The average orientation angle and the average convolution angle were measured for (a) fibres of the different length groups of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties, (b) fibres of the mean length of Samaru 26J (N) and Sea Island, and (c) fibres of different lengths of four commercial cottons ( Sea Island, Giza 45, Ashmouni, and Texas ).

Meredith<sup>136</sup> found that on subtracting the values for the average convolution angles from the respective spiral angle values, the difference was approximately constant. This led to the assumption that the spiral angle of the fibrils in the originally unconvoluted fibres may be about the same irrespective of the variety of cotton. These findings and conclusion of Meredith<sup>136,137</sup> were confirmed by Betrabet et al.<sup>31</sup>

From the results obtained in this work it was found impossible to draw a conclusion on the relationship between the average orientation angle and the average convolution angle and it appears that the two characteristics are independent. Within a variety or sample ( Samaru 26J (G), Rex, Sea Island, Giza 45, Ashmouni, and Texas ), the average orientation angle of fibres of different lengths increases steadily as fibre length decreases ( figure 17 and table 14 ), while the average

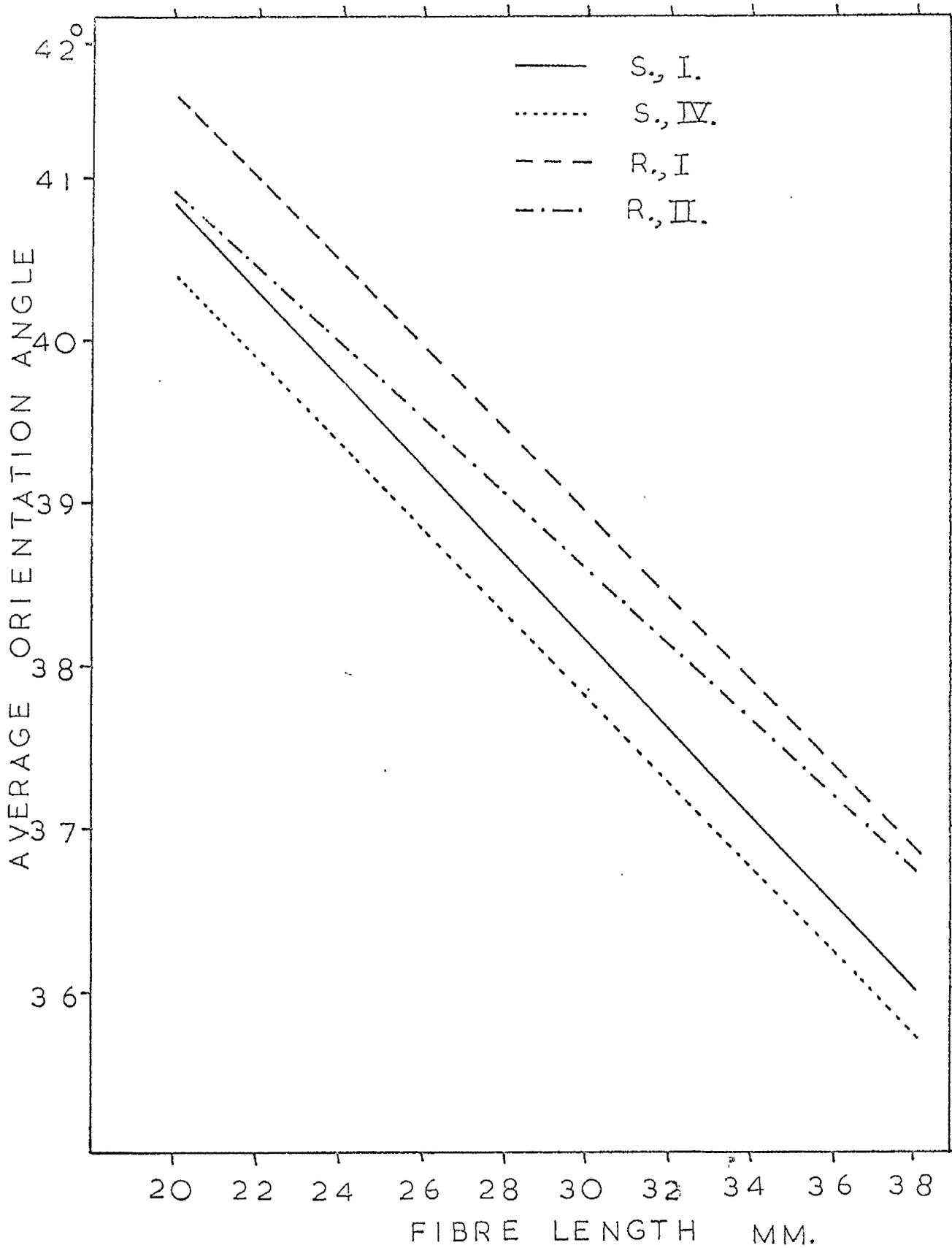


Fig. 17 The average orientation angle for fibres of the different length groups

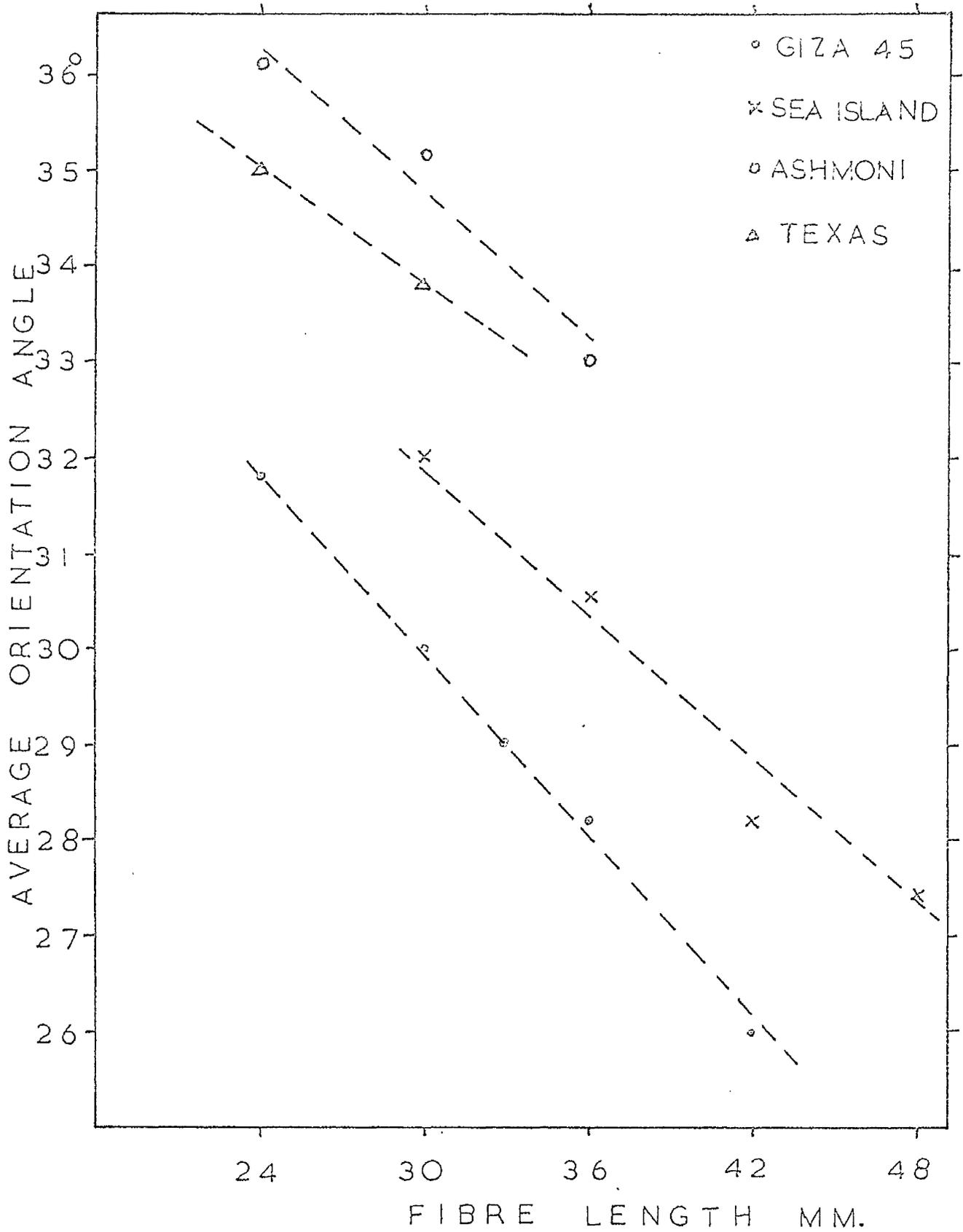


Fig. 18 The average orientation angle for fibres of different lengths

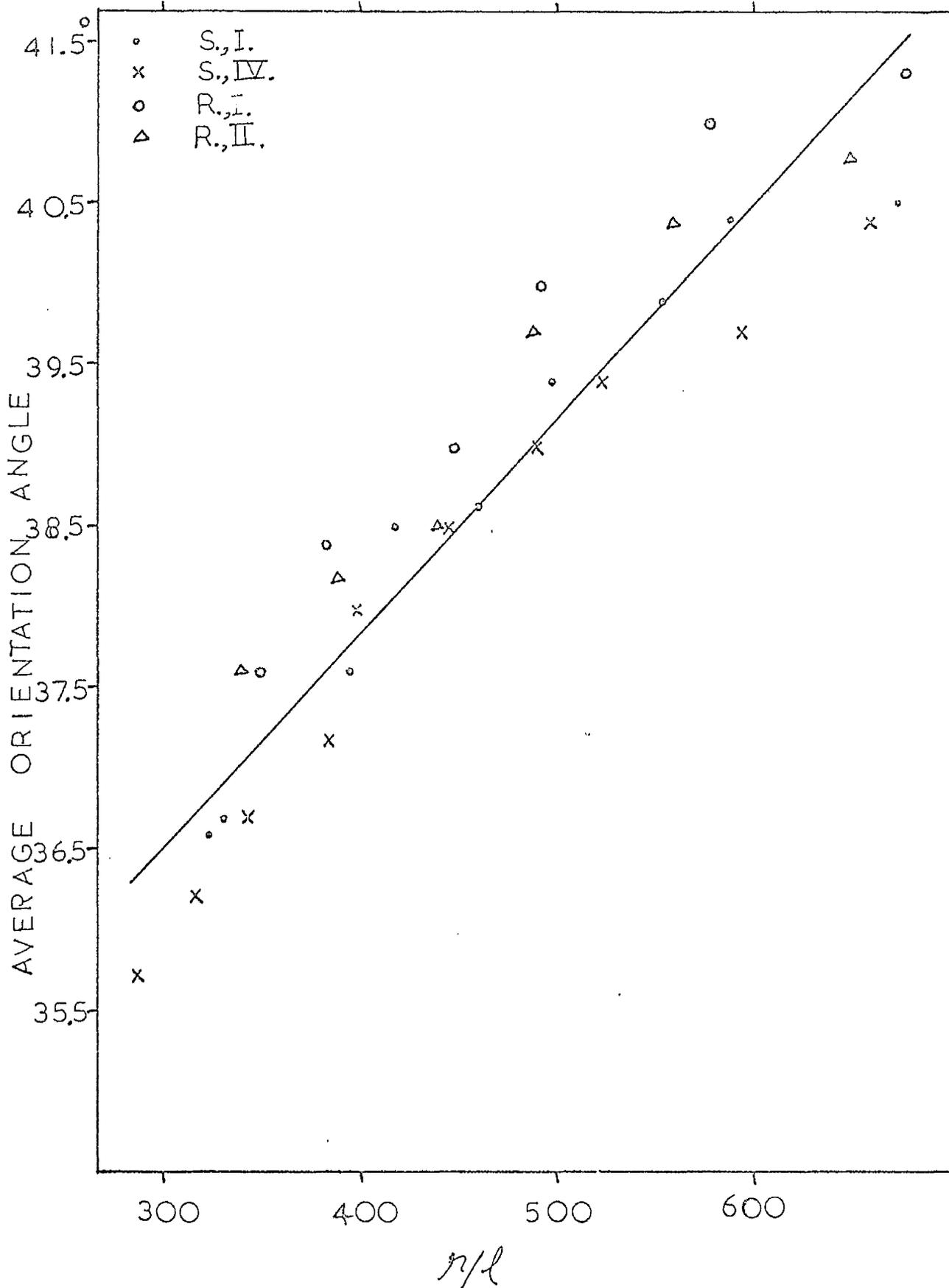


Fig. 19 The change in the average orientation angle in relation to the factor  $r/l$

convolution angle fluctuates according to fibre maturity ( figure 15 ). When the values of the average convolution angles are subtracted from the respective values of the average orientation angles of fibres of different lengths within a variety of the four commercial varieties, table 15, it is apparent that the difference is not constant either within a variety or between the four varieties, and hence it is concluded that the orientation measured for the unconvoluted portions of the fibre is not affected by the presence of the convolutions.

(3) The relation between the molecular orientation and fibre dimensions

From the results obtained for the different test samples under investigation, two points were noticed.

(a) Within a sample or variety, the average orientation angle decreases as fibre length increases, with the longest length group in every variety or sample having the smallest average orientation angle, and the shortest length group having the largest average orientation angle, figures 17, 18.

(b) Between varieties or samples, fibres of equal length may not have equal orientation angles.

In figures 17 and 18 the average orientation angle is plotted against fibre length. The average orientation angle

increases steadily as fibre length decreases. High correlation values between fibre length and the average orientation angle have been obtained (  $r = -0.986, 0.997, 0.990,$  and  $0.980$  for Samaru 26J (G) SS., I., S., IV., Rex, R. I., and R., II. respectively ). This may lead to the conclusion that, within a sample or variety, the average orientation angle depends on fibre length. But such a conclusion may be an over-simplification since we know that these different length groups differ in their diameter as well. It has been reported earlier ( page 90 ) that as fibre length decreases fibre diameter increases. As the fibrils spiral around the fibre axis, then fibre diameter as well as fibre length may be expected to influence, to some extent, the spiral angle. This may explain the variation in the average orientation angle from fibre to fibre within a sample when they have equal lengths but vary in their diameter, and also why fibres of equal length but of different varieties differ in their average orientation angles. Therefore it was thought that it may be helpful to look into the relationship between both fibre length and fibre diameter, or in other words fibre dimensions, on the one hand and the average orientation angle on the other.

The factor  $(\frac{r}{l})$  is proposed to give one value to represent both fibre length and fibre diameter and to describe any change in the values of either or both of them (  $l$  is fibre

length in millimeters,  $r$  is fibre radius in microns multiplied by 1000 in order to get the factor in simple numbers ). This factor has been chosen because if the fibre is regarded as consisting of  $(n)$  segments in each of which the spiral makes one turn, then the following formula is applicable in measuring the spiral angle from the knowledge of spiral pitch and fibre radius:

$$\tan \theta = \frac{2\pi r}{\frac{l}{n}} \quad \text{where } \theta \text{ is the spiral angle,}$$

$r$  is fibre radius, and  $\frac{l}{n}$  is spiral pitch. This factor  $(\frac{r}{l})$  has been found satisfactory in relating fibre length, diameter, and the spiral angle.

In figure 17, in which the average orientation angle is plotted against fibre length for the different length groups of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties, it is apparent that the regression line of each sample is separate. The overall correlation factor between the average orientation angle and fibre length for all the test specimens presented in the figure is - 0.915. In figure 19, in which the average orientation angle is plotted against the factor  $(\frac{r}{l})$  for the same test specimens presented in figure 17 the points became nearer to each other and the overall correlation factor between the average orientation angle and the factor  $(\frac{r}{l})$  is 0.987. This improvement in the correlation factor indicates

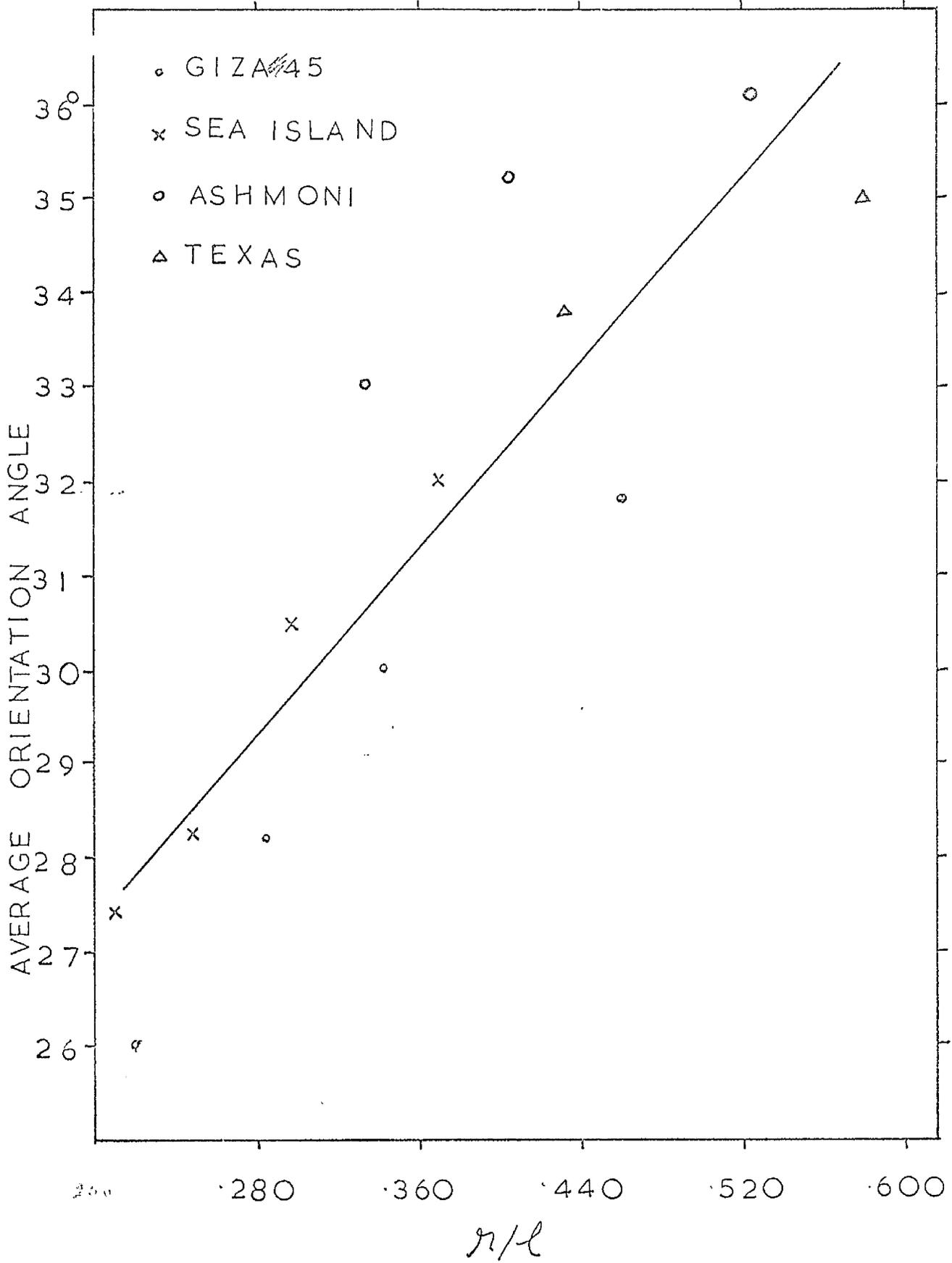
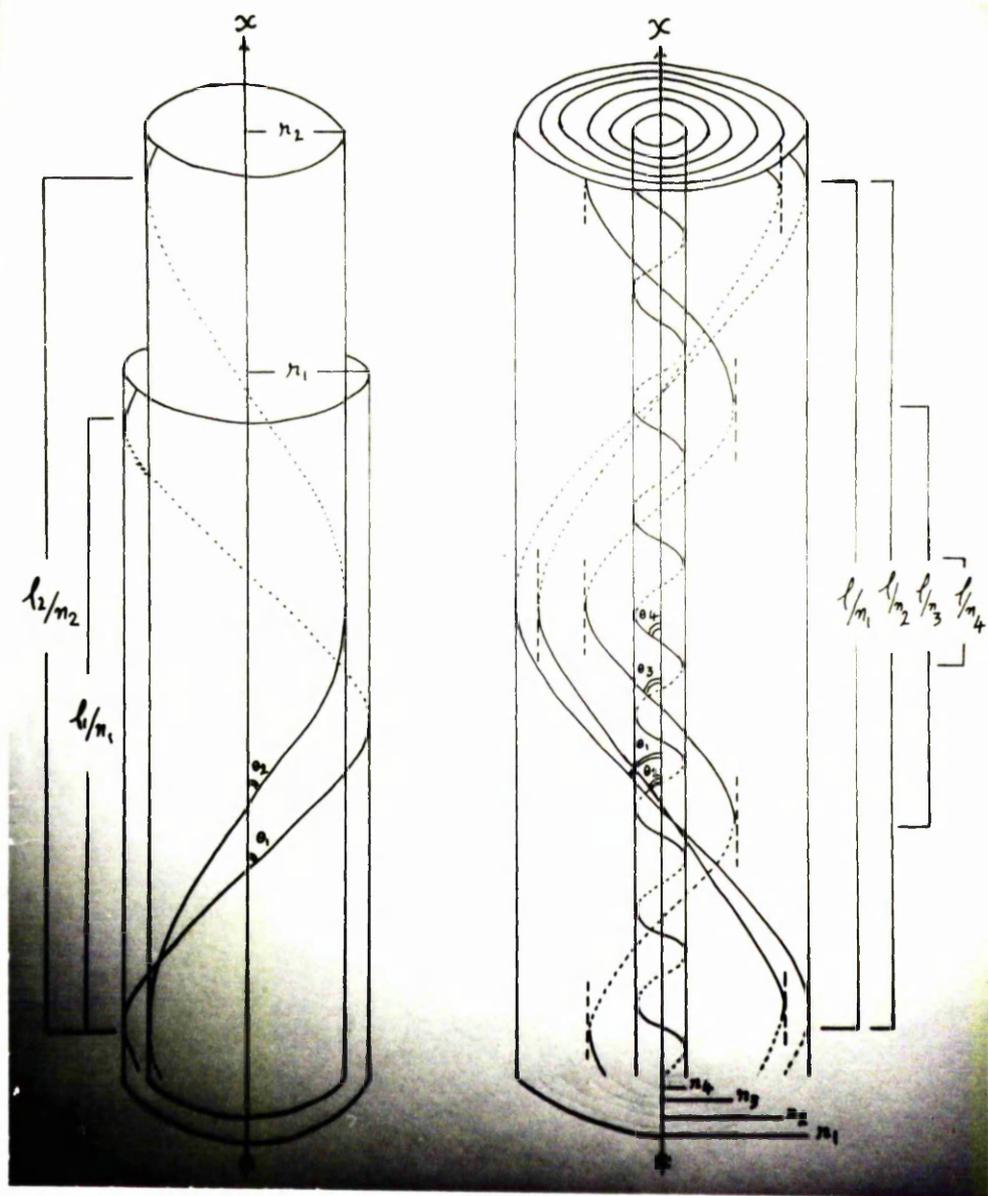


Fig. 20 The change in the average orientation angle in relation to the factor  $r/l$

that part of the difference in the average orientation angle among the different length groups and samples is due to the difference in their diameter. Nearly the same results have been obtained for the four commercial varieties. In comparing figures 18 and 20, it is clear that in figure 18, the points within each variety follow a definite regression line and these regression lines are far apart, while in figure 20, the points of the different varieties became nearer to each other ( the regression lines still being separate can be explained by the fact that these cottons were grown under different environmental conditions which affect the orientation differently, see page 113 ). The overall correlation factors between the average orientation angle and fibre length and between the average orientation angle and the factor  $( \frac{L}{d} )$  are - 0.840 and 0.880 respectively.

This relationship between the average orientation angle and fibre dimensions leads to the assumption that the molecular orientation depends on both the length and the diameter of the fibre; as the fibre becomes longer, and the diameter becomes smaller, the average orientation angle becomes smaller or the



A

B

FIGURE 21

spiral becomes steeper. This relationship is explained by a model, figure 21A.

According to the formula  $\tan \theta = \frac{2\pi r}{\frac{l}{n}}$ ,  $\theta$  will vary according to the variation in either or both fibre diameter and the spiral pitch. In the model, figure 21A, in the longer fibre; the radius ( $r_2$ ) is smaller and the spiral pitch ( $\frac{l_2}{n_2}$ ) is longer than in the short fibre ( $r_1, \frac{l_1}{n_1}$ ) and consequently the spiral angle ( $\theta_2$ ) is smaller than in the short fibre ( $\theta_1$ ) or in other words the spiral is steeper in the long fibre than in the short one.

In this construction of the relationship between the molecular orientation and fibre length and diameter, the fibre is regarded as one cell. It may be argued<sup>31,136</sup> that the cotton fibre is divided by the reversals into segments and that each segment can be regarded as a cell and the fibre as a whole as a series of cells. In this construction ( figure 21A ), the fibre is regarded as only one cell because firstly, the reversals are a forced change<sup>3</sup> in the direction of the spiral at local points and the commonest type of reversal is one in which the fibrils bend around in an arc so as to change their direction, such a

reversal is not expected either to divide the fibre into separate cells or to affect the number of turns which the spiral makes along the fibre length, and consequently is not expected to alter the spiral angle. Secondly, if the fibre is to be regarded as a series of cells, then, since the reversals are not distributed at equal distances along fibre length, it might be expected that the spiral angle would be different in these cells of different length if the number of turns of the spiral were constant in each and this has not been observed. What generally happens is that the spiral makes more or less turns in one segment than in another, according to their length, so as to keep the spiral angle constant in all segments and this means that no relationship exists between the characteristics of the spiral and the presence of reversals.

(4) The molecular orientation of the successive layers of the secondary wall

If the relationship between the average orientation angle and fibre length and diameter holds good for fibres of different length groups within a sample and for fibres of different samples or varieties, one may wonder whether this relation holds for the successive layers of cellulose in the secondary wall as well. Since in these successive layers the length is constant, but their

diameter decreases gradually towards the inner side of the cell, if the relation holds good, one should expect the average orientation angle to decrease for the successive cellulose layers. As an example, let us assume a fibre of  $20 \mu$  diameter and 20 growth layers or lamellae in the secondary wall each of  $0.2 \mu$  thickness, total  $4 \mu$ , and assuming the outer layer having average orientation angle ( or spiral angle ) =  $25^\circ$ . Applying the formula  $\tan \theta = \frac{2 \pi r}{l/n}$ , then there are three possibilities:

(a) If the number of turns of the spiral in the successive layers within a fibre is constant and consequently the spiral pitch is constant for all these layers, then the spiral angle should be  $25^\circ$ ,  $22.8^\circ$ , and  $20.4^\circ$  for the first, tenth, and twentieth layers respectively. The difference in the spiral angle between the outer and innermost layers is great and will lead to a weak fibre.

(b) The spiral angle is constant for all the successive layers and consequently the number of turns of the spiral increases gradually for the successive layers, and the spiral pitch decreases steadily. If this true, then fibres of different degrees of wall thickening should show equal orientation and their initial

Young's moduli should be more or less the same. The results obtained for the orientation and the initial Young's modulus of fibres of successive degrees of wall thickening do not agree with this possibility.

(c) The third possibility is a balance between the previous two, It is possible that for the first few layers of the secondary wall the balance will be in favour of the first factor, in which the spiral angle decreases gradually as the tube diameter decreases, but as the growth process continues for 20 to 30 days there is a possible levelling off in the growth activity. In the middle layers, the two factors may balance each other with the effect of the decrease in the tube diameter equalized by that due to the decrease in the spiral pitch. In the innermost layers, the balance may be reversed, taking into account the possible reduced activity of cellulose deposition, with the decrease in the spiral pitch becoming greater or more effective than that due to the decrease in the tube diameter. The result of this change in the balance between the effect of the decreasing diameter and the decreasing spiral pitch will be: a decrease of the spiral angle, followed by a steady spiral angle, then finally an increase in the spiral angle, of the successive cellulose layers. The difference in the spiral angles between the outer and the inner layers and the middle ones will, however, be small. The measured

average orientation angle of the mature fibres is a mean value of those of the successive layers.

Figure 21B shows a model of the possible spiral structure of the successive layers of the secondary wall as proposed in the third possibility. In the first few layers, represented by layers 1 and 2, the spiral pitch is constant ( $\frac{l}{n_1} = \frac{l}{n_2}$ ) and as the radius decreases ( $r_1 > r_2$ ), the spiral angle decreases ( $\theta_1 > \theta_2$ ). For the middle layers, represented by layer 3, the spiral pitch ( $\frac{l}{n_3}$ ) is smaller than ( $\frac{l}{n_1}$  and  $\frac{l}{n_2}$ ) because the spiral is making more turns, the spiral angle ( $\theta$ ) of these middle layers may be nearly equal but smaller than that of outer layers. In the innermost layers, represented by layer 4, the spiral makes more turns resulting in a marked decrease in the spiral pitch ( $\frac{l}{n_4}$ ) less than ( $\frac{l}{n_1}$ ,  $\frac{l}{n_2}$ , and  $\frac{l}{n_3}$ ) and this overcomes the decrease in the radius ( $r_1 > r_2 > r_3 > r_4$ ): consequently the spiral angle ( $\theta_4$ ) is larger than that in the middle layers ( $\theta_3$ ).

From this hypothetical structure of the successive layers of the secondary wall, one should expect the orientation angle measured for fibres of successive ages or successive degrees of wall thickening but of equal length, to decrease first, level off, as wall thickening increases, and finally might increase as the fibre wall becomes very thick. It is interesting to examine

the results obtained for the Rex variety, table 16, taking fibre density as an indication of the degree of wall thickening.

TABLE 16.

Sample	Fibre age (days)	Fibre density (g cm <sup>-3</sup> )	Orientation	
			40 per cent. X-ray - Av. conv. angle ( $\bar{\Phi}$ )	Average orientation angle ( $\theta$ )
R., I.	30	0.655	35.5	40.1
	40	0.712	33.9	39.7
	60	1.063	32.8	39.8
R., II.	30	0.543	37.9	40.6
	40	0.780	32.2	39.5
	60	0.988	31.4	38.5

The results in table 16 show that, in both R., I. and R., II., both the optically measured average orientation angle and the 40 per cent. X-ray angle, after subtracting the average convolution angle, show a decrease as fibre density increases. The difference in the average orientation angle between fibres 30 days old and fibres 40 days old is greater than that between

fibres 40 days old and fibres 60 days old, showing that there is a levelling off in the decrease in the average orientation angle. If the samples of both R., I. and R., II., are arranged in a manner according to fibre density, the average orientation angle shows a marked decrease first, then a slower decrease, and finally, in the most mature sample, an increase.

These results suggest that the spiral is initially becoming steeper and it falls off more and more gradually in the successive layers of cellulose in the secondary wall. The expected increase in the spiral angle of the innermost layers ( as suggested in figure 21B ) is not quite clear in these samples possibly because these samples do not include any of very high degree of wall thickening or because these innermost layers are too few to affect appreciably the properties of the whole fibre.

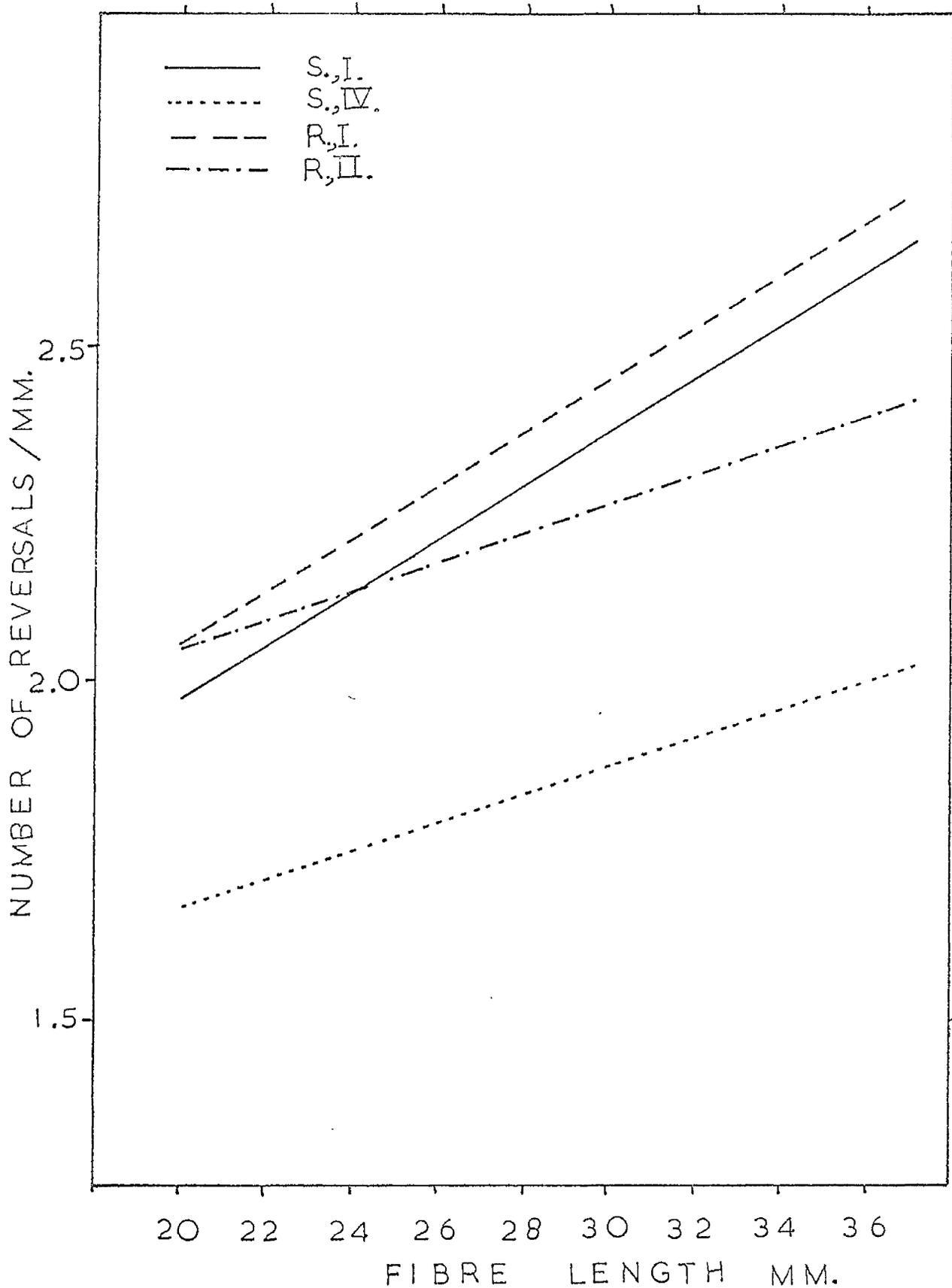


Fig. 22 The number of reversals per mm. for fibres of the different length groups

## THE STRUCTURAL REVERSALS

In order to find the relationship between the number of reversals per unit length and fibre length within a variety or sample and between varieties and the effect of gibberellic acid treatment on this characteristic, the number of reversals per mm. was measured for the different length groups of Samaru 26J (G) ( S., I. and S., IV. ) and Rex ( R., I. and R., II. ) varieties. The results shown in figure 22 , which shows that within a variety or sample, the number of reversals per mm. increases as fibre length increases. Fibres of equal length but of different varieties may possess different numbers of reversals per mm. Treatment with gibberellic acid has resulted in a decrease in the number of reversals per mm. in both Samaru 26J (G) and Rex varieties. This effect is possibly due to the increase in the size of the boll noticed in the treated samples.

CHAPTER V.

COTTON FIBRE MECHANICAL PROPERTIES

TABLE 17. The measured extension to break and breaking load.

Sample	Fibre age (days)	Fibre length (mm.)	Extension to break			Breaking load		
			$E_m$ %	Stand. error	Coef. of var. %	BL (g.)	Stand. error	Coef. of var. %
S., I.	30	28	10.3	0.50	35	0.94	0.05	38
	40	28	8.9	0.50	37	1.43	0.10	46
	50	28	11.0	0.55	35	3.71	0.28	52
	50	36	10.8	0.45	30	2.87	0.20	49
	50	20	11.2	0.55	35	4.33	0.25	40
S., IV.	30	28	9.0	0.50	40	1.01	0.07	53
	40	28	7.6	0.50	50	1.24	0.09	52
	50	28	9.9	0.45	33	3.16	0.27	60
	50	36	9.4	0.45	34	3.25	0.28	61
	50	20	10.3	0.60	40	2.97	0.21	51
S., V.	M	28	10.2	0.50	35	4.34	0.27	43
S., VI.	M	28	9.1	0.47	36	4.14	0.24	41
S.I., I.	M	35	9.3	0.29	22	4.07	0.20	34
S.I., II.	M	35	8.2	0.42	37	3.76	0.26	50

TABLE 17. (continued)

Sample	Fibre		Extension to break			Breaking load		
	age (days)	length (mm.)	$E_m$ %	Stand. error	Coef. of var. %	BL (g)	Stand. error	Coef. of var. %
R., I.	30	27	11.8	0.61	35	1.87	0.11	41
	40	27	12.4	0.64	37	2.66	0.26	69
	60	27	11.4	0.50	33	3.98	0.27	51
R., II.	30	29	13.4	0.61	34	1.62	0.10	48
	40	29	12.5	0.65	37	3.21	0.24	54
	60	29	11.7	0.67	40	4.23	0.29	48
MERCERIZED								
R., I.	30	27	10.8	0.41	25	2.18	0.07	22
	40	27	12.3	0.69	40	3.21	0.21	47
	60	27	11.4	0.56	36	4.55	0.25	42
R., II.	30	29	15.1	0.78	37	1.99	0.10	38
	40	29	9.3	0.50	39	3.87	0.25	45
	60	29	10.9	0.51	35	4.39	0.25	43

TABLE 18 . The measured and projected extension to break and tensile strength and the percentage of normally-broken fibres.

Sample	Fibre age	Fibre length	Ext. to break		Tensile strength		NBF %
	(days)	(mm.)	$E_m$ %	$E_p$ %	$T_m$ (g/tex)	$T_p$ (g/tex)	
S., I.	30	28	10.3	11.7	17.1	20.3	66
	40	28	8.9	10.9	16.5	20.9	64
	50	28	11.0	11.9	26.5	29.8	80
	50	36	10.8	12.2	28.9	34.8	64
	50	20	11.2	11.9	26.5	28.5	88
S., IV.	30	28	9.0	11.2	17.1	21.8	58
	40	28	7.6	10.0	16.2	22.2	58
	50	28	9.9	11.3	28.0	32.5	62
	50	36	9.4	10.9	33.2	41.2	66
	50	20	10.3	11.4	26.9	31.7	74
S., V.	M	28	10.2	11.0	29.5	32.2	87
S., VI.	M	28	9.1	10.3	27.0	30.0	78
S.I., I.	M	35	9.3	9.9	43.7	47.3	86
S.I., II.	M	35	8.2	10.1	38.5	49.9	63

TABLE 18 . (continued)

Sample	Fibre age (days)	Fibre length (mm.)	Ext. to break		Tensile strength		NBF %
			$E_m$ %	$E_p$ %	$T_m$ (g/tex)	$T_p$ (g/tex)	
R., I.	30	27	11.8	12.5	16.4	17.9	80
	40	27	12.4	13.5	21.4	26.4	50
	60	27	11.4	12.4	21.5	23.9	76
R., II.	30	29	13.4	14.7	18.2	21.2	54
	40	29	12.5	13.9	25.1	30.3	56
	60	29	11.7	12.9	26.1	31.1	72
MERCERIZED							
R., I.	30	27	10.8	10.9	19.2	19.4	97
	40	27	12.3	13.6	25.9	30.4	63
	60	27	11.4	12.6	24.6	28.1	77
R., II.	30	29	15.1	15.6	22.3	23.4	90
	40	29	9.3	10.3	30.2	34.7	72
	60	29	10.9	12.0	27.1	31.3	73

TABLE 19 . The percentage extension due to the convolutions  
and fibre fine structure.

Sample	Fibre age (days)	Fibre length (mm.)	$E_c$ %	$E_m - E_c$ %	$E_p - E_c$ ( $E_f$ ) %
S., I.	30	28	3.5	6.8	8.2
	40	28	3.4	5.6	7.6
	50	28	4.0	7.0	7.9
	50	36	3.6	7.2	8.6
	50	20	4.0	7.2	7.9
S., IV.	30	28	3.0	6.0	8.2
	40	28	3.3	4.3	6.7
	50	28	3.1	6.8	8.2
	50	36	3.1	6.3	7.8
	50	20	2.9	7.4	8.5
S., V.	M	28	3.0	7.2	8.0
S., VI.	M	28	2.6	6.5	7.7
S.I., I.	M	35	1.8	7.5	8.1
S.I., II.	M	35	1.5	7.2	8.0

TABLE 19 . (continued)

Sample	Fibre age (days)	Fibre length (mm.)	$E_c$ %	$E_m - E_c$ %	$E_p - E_c$ ( $E_f$ ) %
R., I.	30	27	3.9	7.9	8.7
	40	27	4.9	7.5	8.6
	60	27	4.8	6.5	7.4
R., II.	30	29	3.0	10.4	11.7
	40	29	4.8	7.8	9.1
	60	29	4.9	6.8	8.0
MERCERIZED					
R., I.	30	27	2.3	8.4	8.6
	40	27	2.0	11.3	11.6
	60	27	0.5	11.0	12.1
R., II.	30	29	1.5	13.6	14.1
	40	29	2.0	7.2	8.3
	60	29	0.9	9.9	11.1

TABLE 20 . The initial Young's modulus and the stiffness.

Sample	Fibre age (days)	Fibre length (mm.)	In. Young's mod.			Stiffness		
			(g/tex) <sub>m</sub>	(g/tex) <sub>c</sub>	S <sub>m</sub>	(g/tex) <sub>ma</sub>	S <sub>p</sub>	S <sub>pa</sub>
S., I.	30	28	288	541	166	252	172	248
	40	28	251	646	185	295	192	277
	50	28	370	845	242	381	250	374
	50	36	487	867	267	401	284	404
	50	20	320	866	236	367	239	360
S., IV.	30	28	340	685	189	283	194	265
	40	28	285	715	214	380	222	332
	50	28	470	800	282	412	280	396
	50	36	543	1016	354	525	379	527
	50	20	424	854	261	365	278	375
S., V.	M	28	392	900	289	410	293	402
S., VI.	M	28	376	822	300	417	291	390
S.I., I.	M	35	658	943	470	583	478	584
S.I., II.	M	35	670	963	469	575	499	587

TABLE 20 . (continued)

	Fibre age (days)	Fibre length (mm.)	In. Young's mod.			Stiffness		
			$Y_m$	$Y_c$	$S_m$	$S_{ma}$	$S_p$	$S_{pa}$
R., I.	30	27	197	347	139	207	142	206
	40	27	331	582	173	284	196	306
	60	27	282	623	189	328	195	322
R., II.	30	29	213	287	136	177	144	182
	40	29	395	697	200	323	218	332
	60	29	295	670	223	382	242	389
MERCERIZED								
R., I.	30	27	213	344	167	227	178	226
	40	27	400	512	211	230	224	263
	60	27	328	349	215	233	224	233
R., II.	30	29	222	261	147	164	150	167
	40	29	476	690	327	417	337	418
	60	29	360	431	249	273	261	282

TABLE 21 . The work of rupture and the work factor.

Sample	Fibre age	Fibre length	Work of rupture ( g/tex ) <sup>29.</sup>			Work factor		
	(days)	(mm.)	WR <sub>m</sub>	WR <sub>p</sub>	WR <sub>pa</sub>	WF <sub>m</sub>	WF <sub>p</sub>	WF <sub>pa</sub>
S., I.	30	28	0.820	1.081	0.898	0.464	0.455	0.543
	40	28	0.690	1.064	0.903	0.467	0.467	0.572
	50	28	1.298	1.548	1.290	0.446	0.436	0.546
	50	36	1.490	1.935	1.558	0.476	0.455	0.520
	50	20	1.283	1.475	1.306	0.431	0.436	0.580
S., IV.	30	28	0.778	1.200	1.008	0.505	0.492	0.564
	40	28	0.583	1.033	0.911	0.476	0.464	0.611
	50	28	1.190	1.613	1.290	0.428	0.439	0.486
	50	36	1.453	2.008	1.629	0.467	0.450	0.507
	50	20	1.210	1.515	1.379	0.437	0.419	0.513
S., V.	M	28	1.363	1.638	1.435	0.453	0.464	0.557
S., VI.	M	28	1.100	1.453	1.250	0.450	0.470	0.540
S.I., I.	M	35	1.920	2.193	2.016	0.472	0.468	0.526
S.I., II.	M	35	1.535	2.330	2.177	0.486	0.465	0.513

TABLE 21 . (continued)

Sample	Fibre		Work of rupture			Work factor		
	age (days)	length (mm.)	( g/tex ) <sup>1/2</sup> . )			WF <sub>m</sub>	WF <sub>p</sub>	WF <sub>pa</sub>
			WR <sub>m</sub>	WR <sub>p</sub>	WR <sub>pa</sub>			
R., I.	30	27	0.930	1.048	0.833	0.481	0.468	0.531
	40	27	1.198	1.435	1.064	0.451	0.400	0.465
	60	27	1.140	1.322	0.976	0.464	0.442	0.549
R., II.	30	29	1.160	1.410	1.193	0.476	0.452	0.482
	40	29	1.340	1.725	1.339	0.426	0.411	0.485
	60	29	1.335	1.667	1.346	0.437	0.419	0.540
MERCERIZED								
R., I.	30	27	1.008	1.033	-	0.489	0.489	-
	40	27	1.378	1.740	-	0.434	0.422	-
	60	27	1.200	1.515	-	0.427	0.429	-
R., II.	30	29	1.435	1.548	-	0.425	0.424	-
	40	29	1.410	1.758	-	0.505	0.492	-
	60	29	1.468	1.822	-	0.499	0.486	-

The following is a key for figures 23 - 32.

- ×        The measured breaking point
- ⊙        The extrapolated breaking point
- \_\_\_\_\_ The measured stress-strain curve
- ..... The extrapolated stress-strain curve
- The re-plotted extrapolated stress-strain curve  
( after removing the contribution of the convolutions )

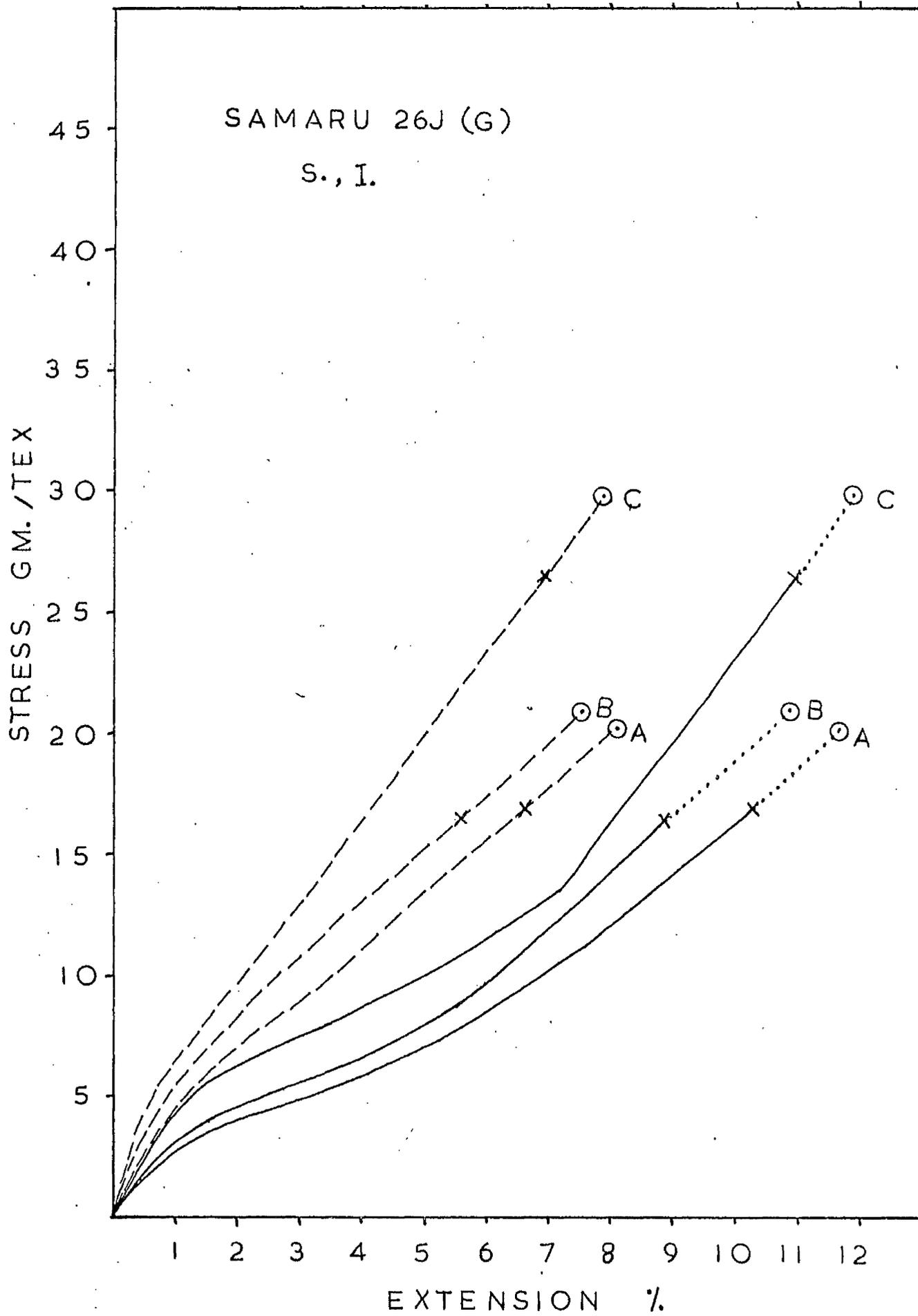


Fig. 23 Stress-strain curves for Samaru 26J (G)  
(A) 30 days old (B) 40 days old (C) 50 days old

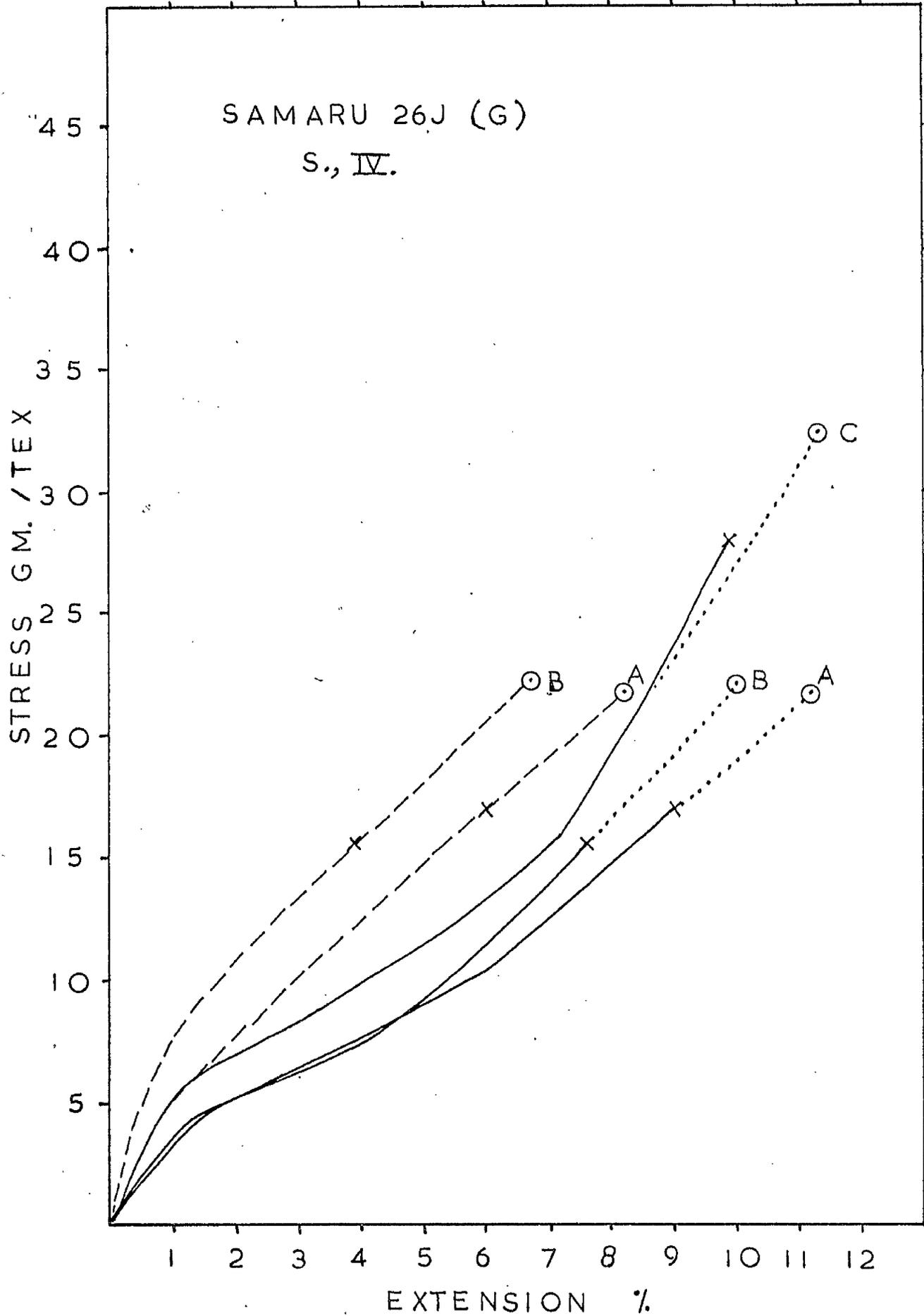


Fig. 24 Stress-strain curves for Samaru 26J (G)  
(A) 30 days old (B) 40 days old (C) 50 days old

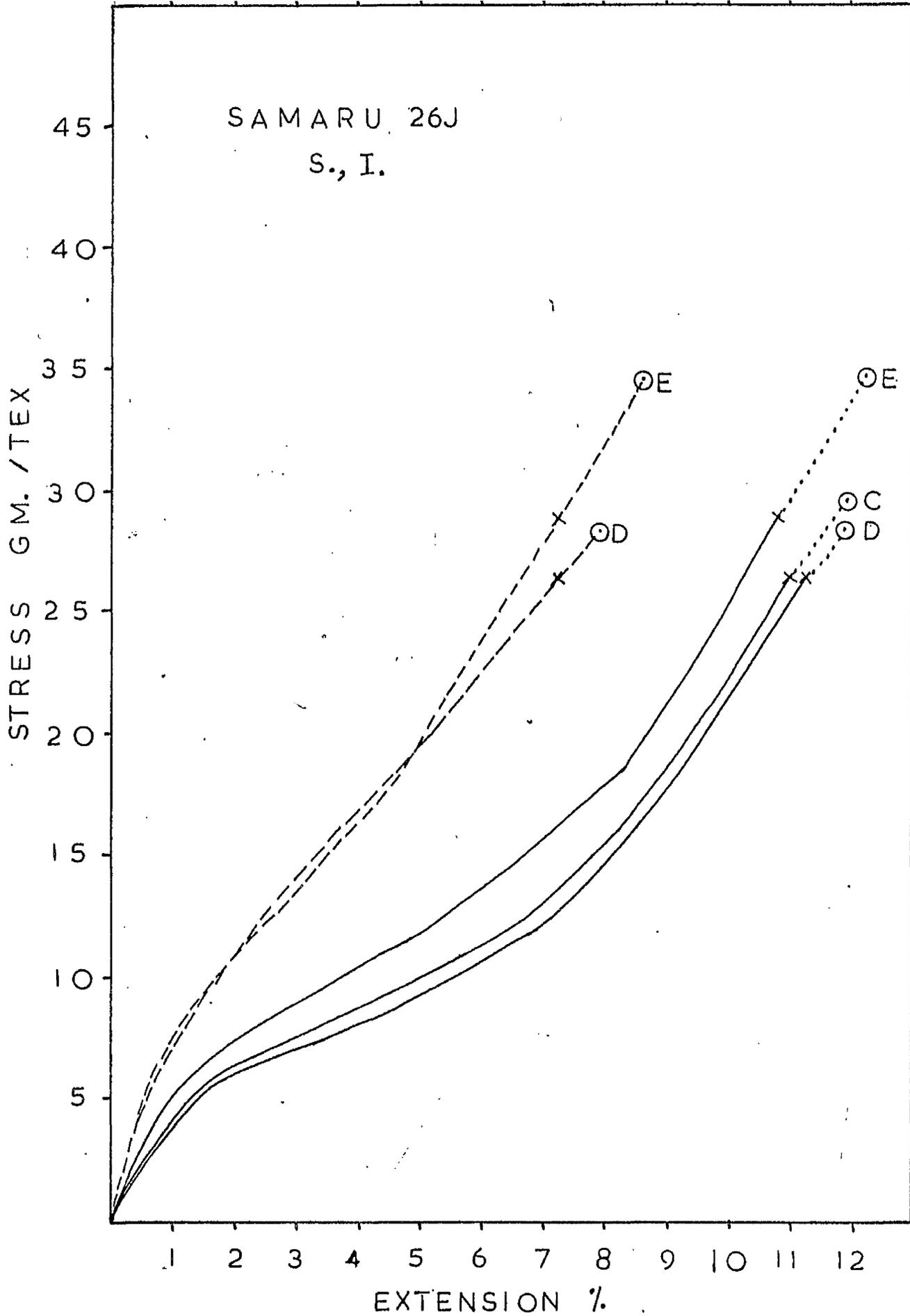


Fig. 25 Stress-strain curves for Samaru 26J (G)  
(C) 28 mm. long. (D) 20 mm. long (E) 36 mm. long

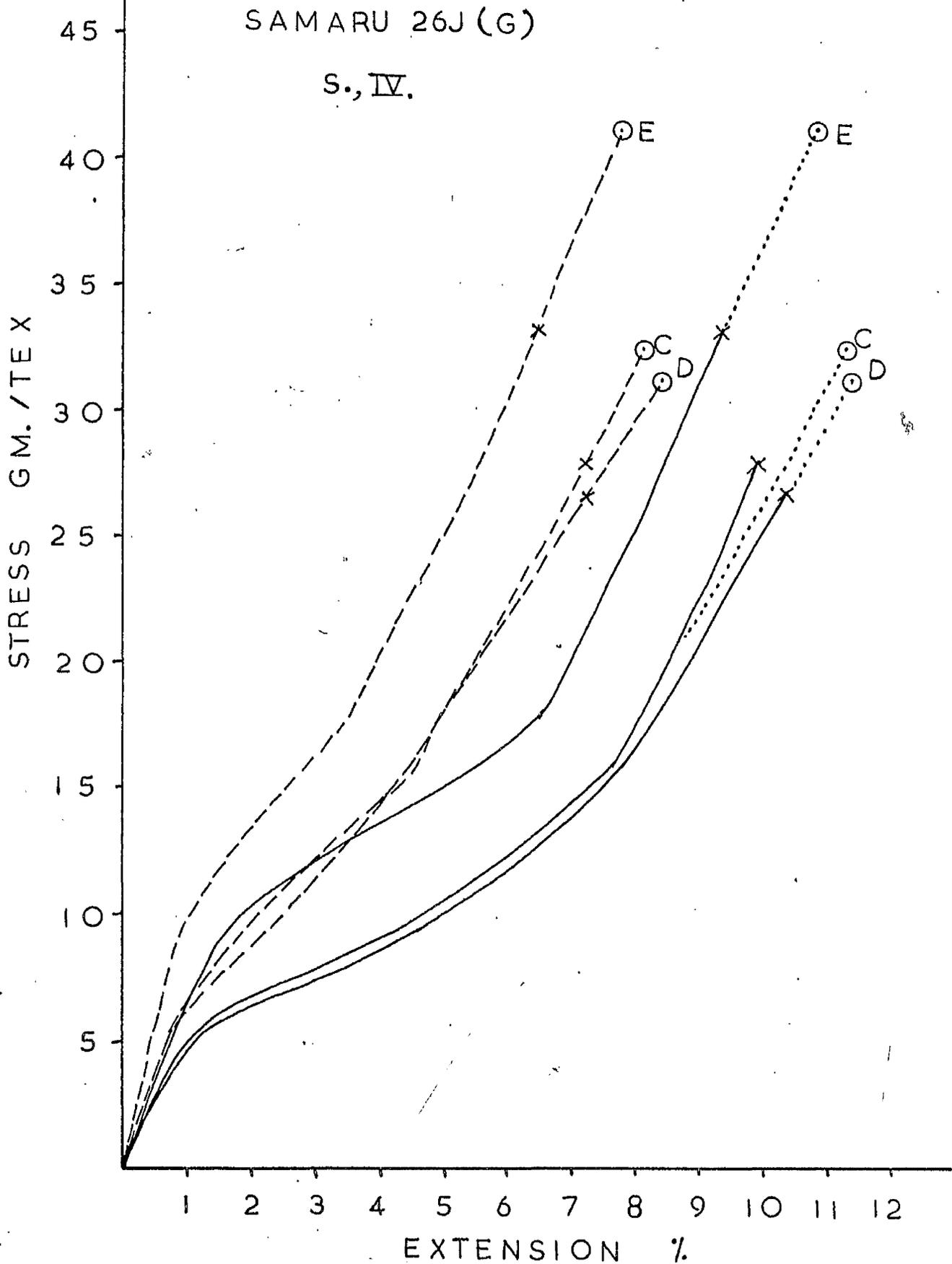


Fig. 26 Stress-strain curves for Samaru 26J (G).  
(C) 28 mm. long (D) 20 mm. long (E) 36 mm. long

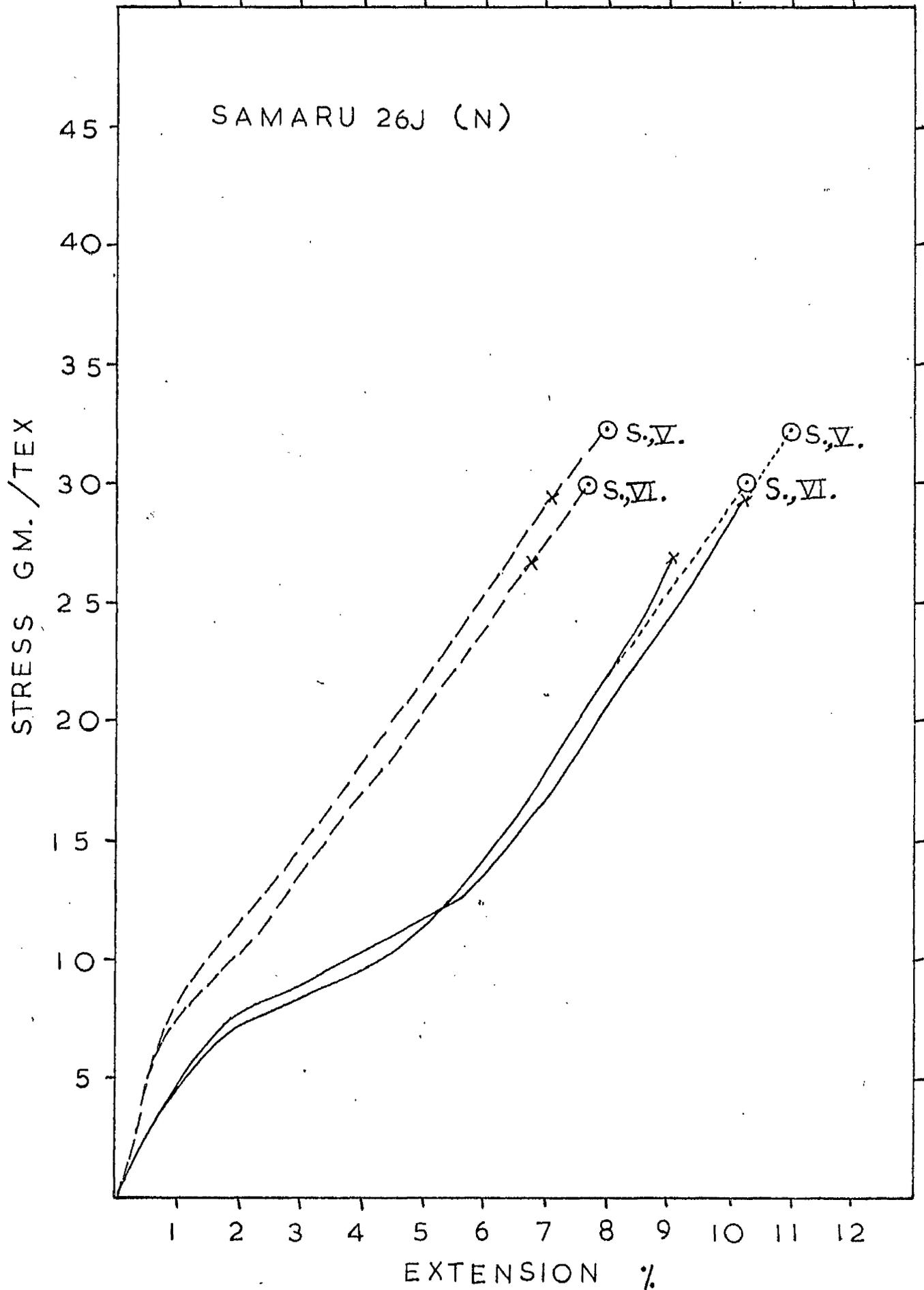


Fig. 27 Stress-strain curves for Samaru 26J (N)

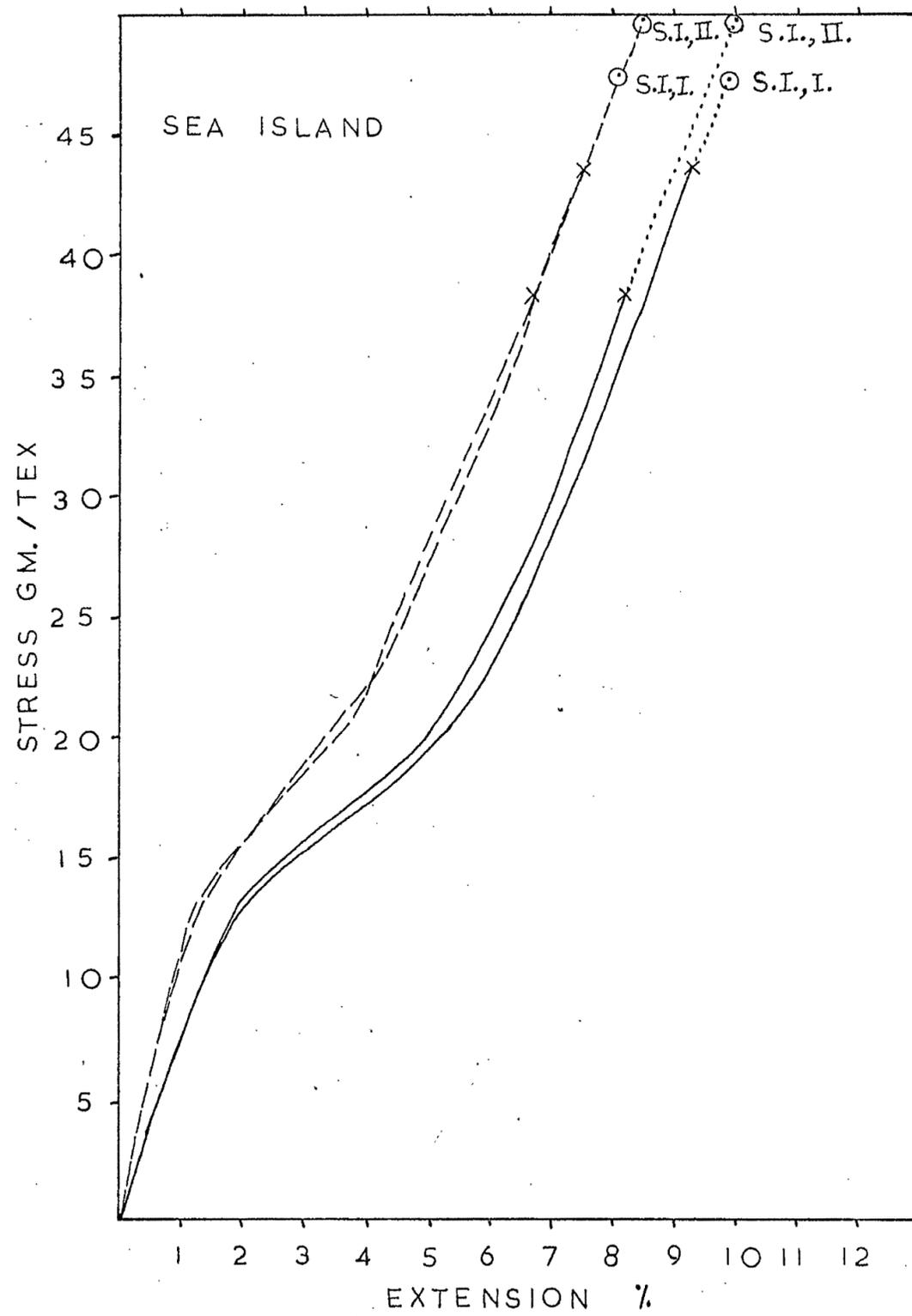


Fig. 28 Stress-strain curves for Sea Island

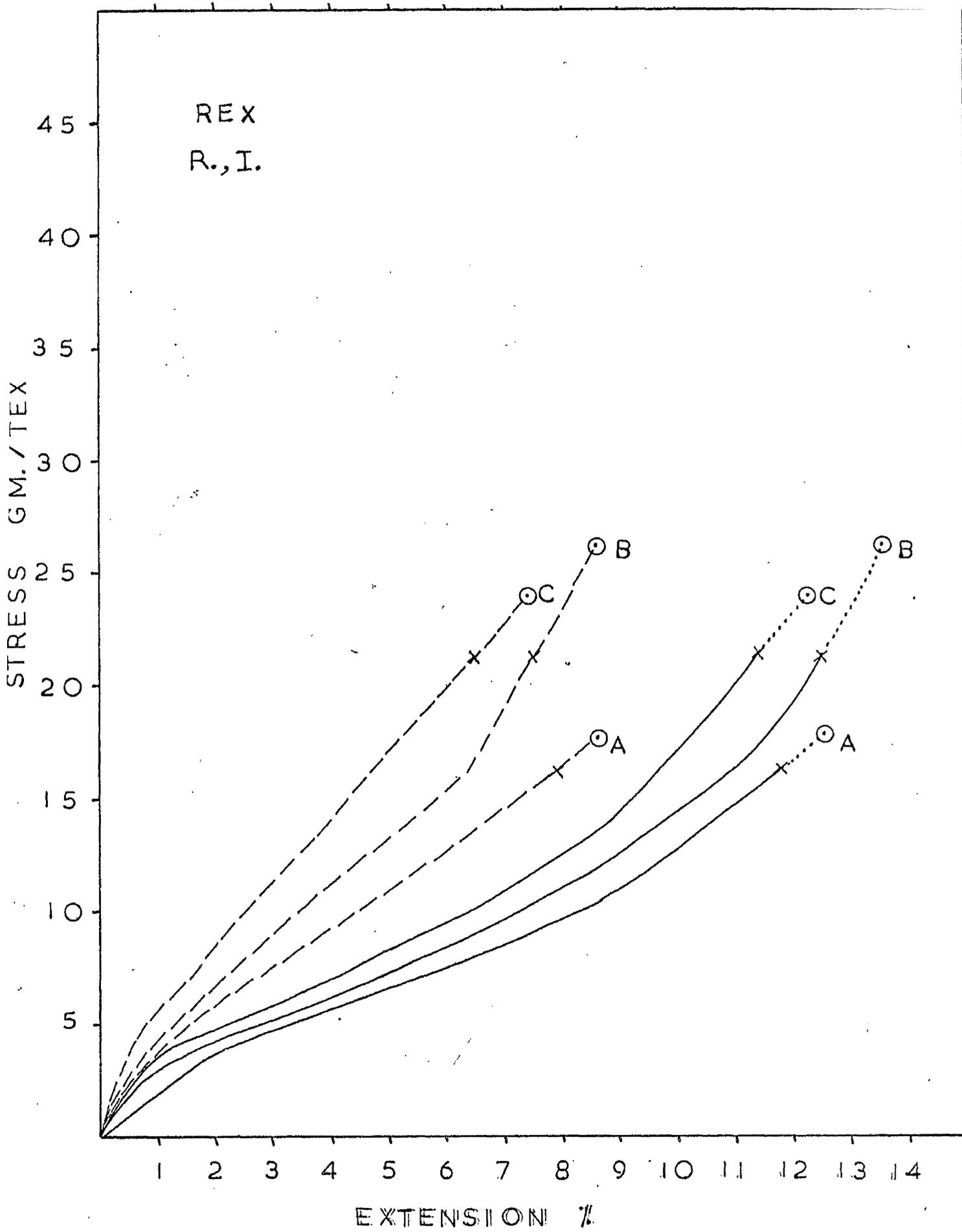


Fig. 29 Stress-strain curves for Rex  
(A) 30 days old (B) 40 days old (C) 60 days old

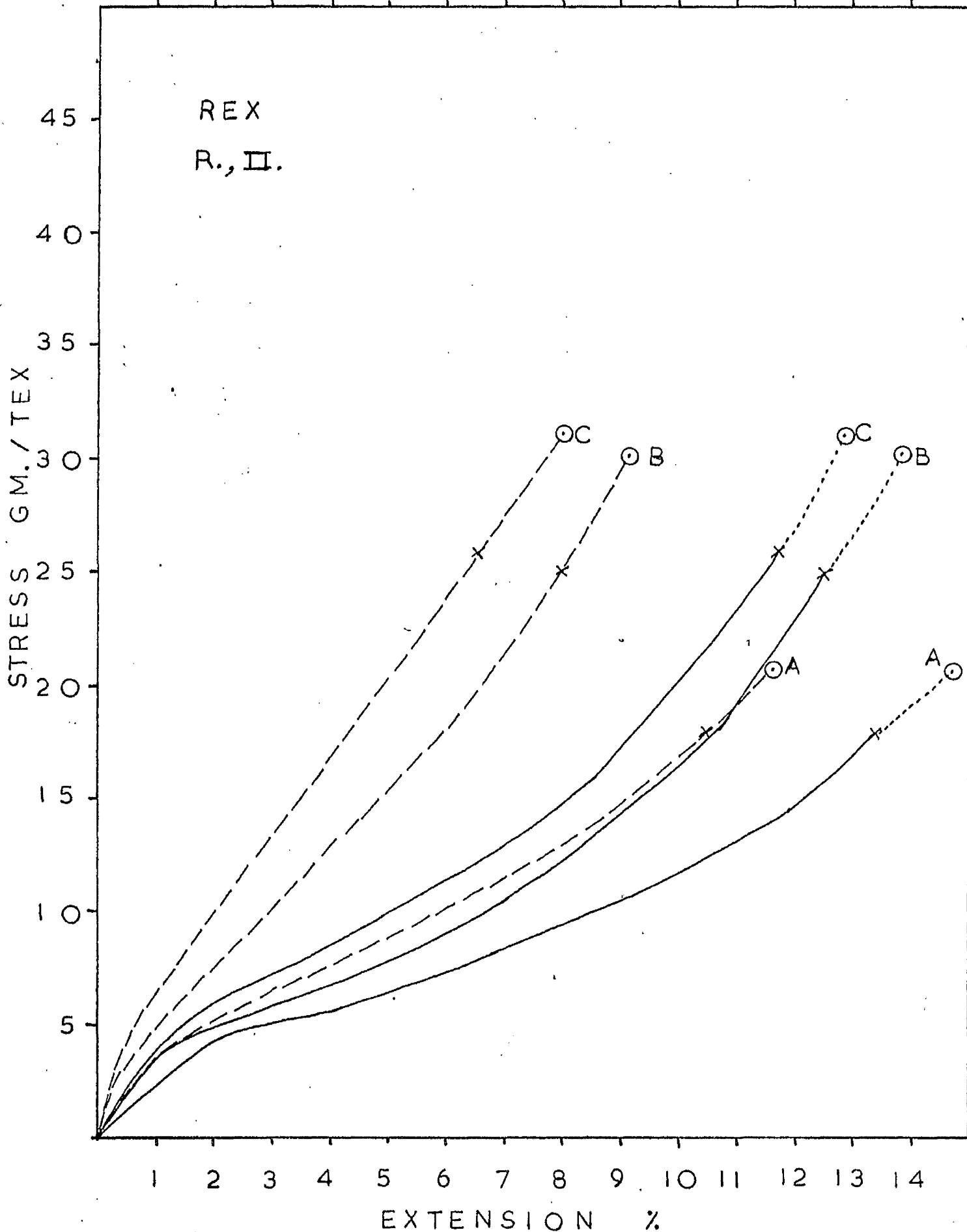


Fig. 30 Stress-strain curves for Rex  
(A) 30 days old (B) 40 days old (C) 60 days old

REX (MERCERIZED)  
R, I.

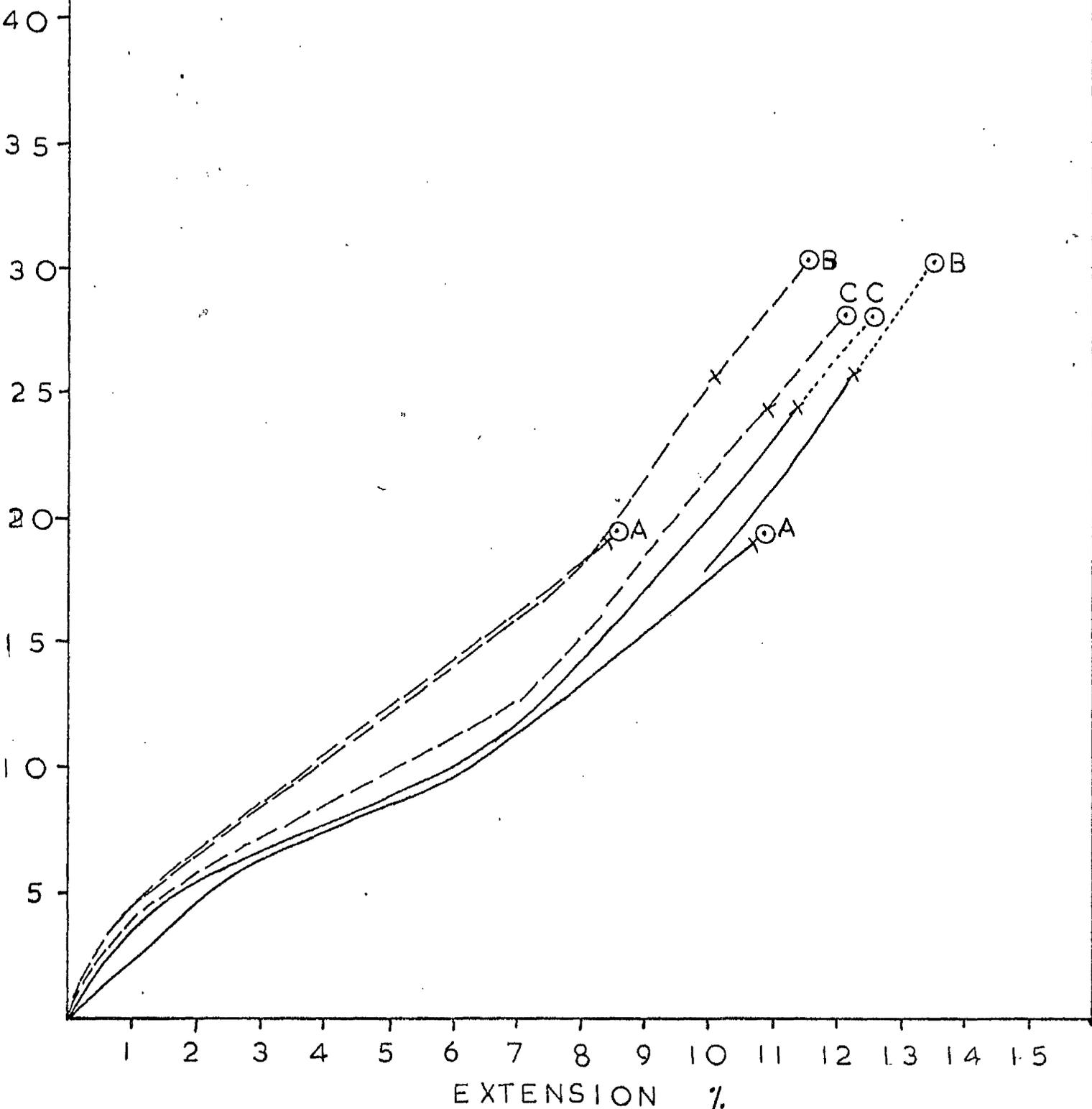


Fig. 31 Stress-strain curves for Rex. (mercerized)  
(A) 30 days old (B) 40 days old (C) 60 days old

REX (MERCERIZED)

R., II.

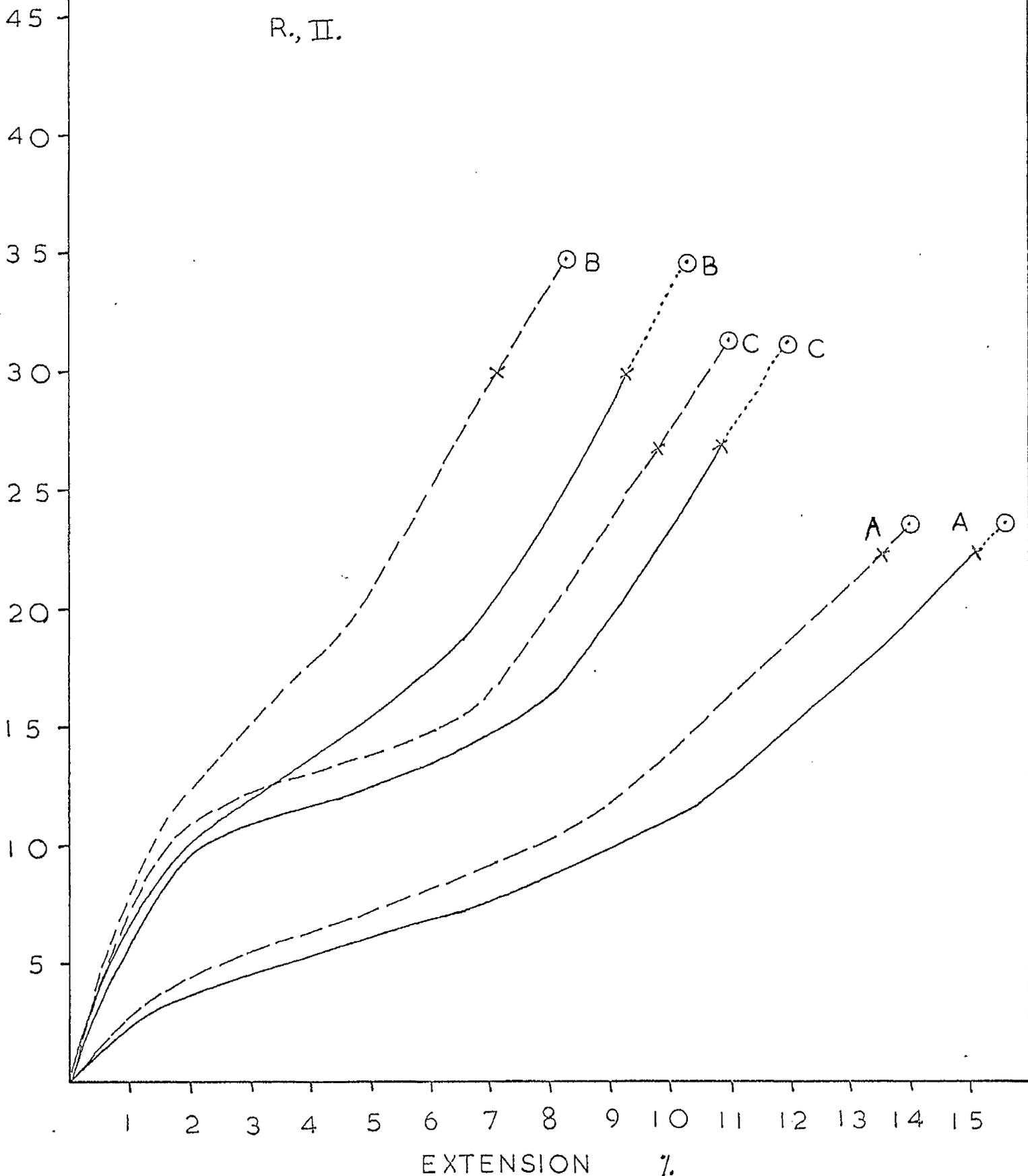


Fig. 32 Stress-strain curves for Rex (mercerized)  
(A) 30 days old (B) 40 days old (C) 60 days old

The mechanical properties of cotton fibres, mainly tensile strength and extension to break, are of paramount importance in their utilization and have been always regarded as the most influential properties in cotton evaluation. Hence, the effect of gibberellic acid treatment on these properties has been given considerable attention. The possible direct as well as the possible indirect effect of gibberellic acid treatment on fibre mechanical properties have been investigated. As gibberellic acid has caused an increase in fibre mean length of some samples ( Samaru 26J (G) and Rex varieties ) and a decrease in fibre maturity ( Samaru 26J (G) and Rex varieties ), the relationship between these two characteristics and fibre mechanical properties has been investigated in order to find the indirect effect of gibberellic acid ( through its effect on fibre length and maturity ) and also the effect of these characteristic features as structural entities on fibre mechanical properties.

The following are comparisons between the measured mechanical properties of the control samples and the samples treated with gibberellic acid:

(a) Extension to break (  $E_m$  ) ( table 17 )

All the treated samples of Samaru 26J (G), Samaru 26J (N), and Sea Island show lower extension to break than the control ones. No difference was found between the control and the treated samples

of the Rex variety.

(b) Tensile strength (  $T_m$  ) ( table 18 )

For Samaru 26J (G); fibres 30 and 40 days old, and mature fibres of 20 mm. length, show no difference in tensile strength due to gibberellic acid treatment. For mature fibres 28, and 36 mm. long, the control samples show lower tensile strength. All the control samples of the Rex variety show lower tensile strength. The mature fibres, 60 days old, of the treated sample show a 30 % increase in tensile strength compared with the mature control sample. The control sample of Samaru 26J (N) and the control sample of Sea Island show higher tensile strength. It is difficult to decide whether all these differences are significant because the tensile strength was calculated from mean values of breaking load and fibre linear density and these two properties are correlated. However, as a guide a difference of 5 % may be taken as being significant.

(c) Work of rupture (  $WR_m$  ) ( table 21 )

For Samaru 26J (G), Samaru 26J (N), and Sea Island, the control samples show higher work of rupture. For Rex, the control samples show lower work of rupture.

(d) The initial Young's modulus and the stiffness (  $Y_m$  and  $S_m$  )  
( table 20 )

For Samaru 26J (G) and Rex varieties, the control samples show

lower initial Young's modulus and stiffness values. For Samaru 26J(N) and Sea Island, both the control and treated samples show nearly equal values.

To explain these differences, it is thought necessary first to investigate carefully the stress-strain curve and the various structural properties that influence and contribute to the measured mechanical properties.

#### ANALYSIS OF THE STRESS-STRAIN CURVE

The stress-strain curve for cotton fibres provides comprehensive information about the behaviour of the fibre as load is applied, a behaviour which is a product of the fine and gross structure of the fibre: the molecular orientation, the crystallinity, the weak places and the convolutions and other fine and gross structural features, all contribute to the stress-strain curve. The curve has been analyzed to reveal their respective contributions. The actual or measured stress-strain curve was first obtained from measurements on a test specimen of fifty single fibres. This curve was then extrapolated for the effect of the major weak places in order to get the projected stress-strain curve. From the latter, the percentage extension provided by the convolutions has been removed in order to get a stress-strain curve which is supposed to be due to the fibre

fine structure, mainly the overall molecular orientation and the percentage crystallinity, unmodified by either the weak places or the convolutions. Finally an attempt has been made to find the role of the orientation and the crystallinity in fibre extensibility independently.

(a) The role of the weak places

Several kinds of major weak places occur along the cotton fibre length. These are mainly the structural reversals, the growth rings, and the structural faults. The presence of such major weak places causes a rapid deterioration in the measured strength and elongation of cotton fibre as the test length is increased. To obtain a stress-strain curve, it has been found that a specimen test length of 1 cm. is satisfactory, test lengths shorter than 1 cm. are not practicable while longer test lengths suffer more from the presence of the weak places. However, even in the chosen test length of 1 cm., the presence of the weak places was apparent, and as the frequency of these major weak places along the fibre length from one sample to another might not be constant and consequently their effect might be different, a method for re-calculating the stress and the strain at the breaking point was applied in order to get the projected stress and strain which are supposed to be the nearest to the stress

and strain resulting from fibre fine and gross structural characteristics. The difference between the projected extension to break, tensile strength, and work of rupture, and the actual values can be attributed to these major weak places and shows the loss in the mechanical properties due to their presence and frequency. The percentage of the prematurely -broken fibres gives an idea about the frequency of the major weak places within a sample.

From the stress-strain curves presented in figures 23 - 32 it is apparent how much of the stress-strain curve is absent because of the presence of the major weak places and how much this is different from one sample to another. From tables 18 , 21 it is clear that the actual or measured extension to break, tensile strength, and work of rupture, are appreciably lower than the projected ones. The magnitude of this difference is broadly in agreement with the percentage of prematurely-broken fibres. The percentage of normally-broken fibres varies from sample to sample and shows some relation with variety, fibre maturity, fibre length, and gibberellic acid treatment. The control sample of Samaru 26J (N) ( S., V. ) shows the lowest percentage of prematurely-broken fibres, followed by Sea Island, Samaru 26J(G), and then Rex.

The effect of maturity on the frequency of the major weak places, which might be a result of the number of growth rings,

is not quite clear. However, it seems that samples of lower and higher maturity possess higher percentages of normally-broken fibres than fibres of intermediate maturity ( S., I. , S., IV. , and R., I. ).

The relation between fibre length, within a sample, and the major weak places ( see table 18 ) is rather confused. However, generally, the long, mature fibres of Hamaru 26J (G) ( S., I. and S., IV. ) show a much lower percentage of normal breakages than the shortest fibres. As shown earlier, the longer fibres possess more reversals per unit length than the short ones, thus this decrease in the percentage of normally-broken fibres as fibre length increases within a variety or a sample is possibly due to the increase in the number of reversals per unit length as fibre length increases.

The effect of gibberellic acid treatment on the frequency of the major weak places is clear. In almost all the samples the percentage of normally-broken fibres in the control samples is appreciably higher than that in the samples treated with gibberellic acid. This effect cannot be related to the frequency of the structural reversals, since control samples possess greater numbers of reversals per unit length than the treated samples. Also it cannot be related to the number of growth rings since these are expected to be equal in both the control and the treated

samples. The third possibility could be the frequency of the structural faults which might increase as a result of treatment with gibberellic acid.

The effect of mercerization at constant length on the frequency of the major weak places, or in other words on the percentage of prematurely-broken fibres, is quite interesting. From table 18 it is clear that this effect is dependent on fibre maturity or in other words the number of growth rings. The greatest reduction in the percentage of prematurely-broken fibres was in the very young fibres ( 30 days old of R., I. and R., II. ), followed by fibres of intermediate maturity ( 40 days old ), while in the mature fibres ( 60 days old ) the reduction is negligible. Thus it is possible that the increase in the tensile strength of the mercerized mature samples is not due to the elimination of the major weak places but rather is due to either the elimination of the minor weak places or other effects, while the increase in the tensile strength of the immature samples is due to the elimination of both major and minor weak places and other factors.

(b) The role of the convolutions in fibre extensibility

It has been proved that the convolutions unfold as load is applied and thus contribute to fibre extension to break ( see page 162 ).

The percentage extension due to unfolding the convolutions has been calculated for the samples under investigation following the method given in page 62. It varies according to variety and maturity and ranges between 1.5 % for Sea Island ( S.I., II. ) and 4.9 % for Rex ( R., II. ) ( see table 19 ).

From tables 18-21 and figures 23-32, it is clear that the convolutions not only contribute substantially to the fibre extension and hence contribute to the work of rupture, but also give the stress-strain curve its typical shape. This effect on the stress-strain curve shape is apparent from the change in the work factor which is always less than 0.5 for the actual or measured stress-strain curves, and which becomes, often, more than 0.5 after the removal of the contribution of the convolutions.

(c) The role of orientation in fibre extensibility

After the removal of the part contributed by the convolutions ( $E_c$ ) from the projected extension to break ( $E_p$ ), the stress-strain curve can be regarded as a product of the fibre fine structure. The orientation and the crystallinity are possibly the most important characteristics contributing to the extension to break ( $E_f$ ).

Since differences in percentage crystallinity between different cottons are negligible<sup>100</sup> ( this might not be strictly

the case for fibres of different degrees of maturity or stages of growth ), the differences in extension to break (  $E_f$  ) between varieties might be due to the differences in their spiral angle or orientation.

In comparing the extension to break (  $E_f$  ) of the mature samples under investigation, it has been found that all of them have more or less equal extension to break (  $E_f$  ) with mean values between 7.4 and 8.1 % . This might be explained in the following:

- (1) The extension to break is due largely to the unfolding of the helix, as presumed by Orr and co-workers,<sup>160</sup> then either:
  - a. all the samples have equal spiral angles ( as suggested by Meredith<sup>136</sup> ), and consequently the unfolding of the spiral structure produces equal extensions, or
  - b. the differences in the spiral angle of the different samples exist but do not show consequent differences in extension to break (  $E_f$  ), possibly because of the failure of the spiral of the samples of greater spiral angle to unfold to an extent comparable to that of samples of smaller spiral angle.
- (2) The unfolding of the helix provides only a small proportion of the extension to break (  $E_f$  ), either because:
  - a. the helix does not actually unfold to a great extent, or
  - b. the spiral angles are much smaller than the empirical

X-ray angles or average orientation angles, hence, even if the spiral unfolds completely, it will not contribute appreciably to the extension to break, and the differences between the different samples will be small.

There is not sufficient evidence to prove that the spiral unfolds either partially or completely during fibre extension to break. The evidence presented by Orr and co-workers<sup>160</sup> which is based on their observation that a fibre untwists when a weight is hung on it does not take into account the possible presence of convolutions.

The measurements of the crystallite orientation and the overall molecular orientation carried out on the samples under investigation here showed that these samples possessed different orientation angles.

DeLuca and co-workers<sup>60</sup> analyzed the ( 002 ) X-ray diffraction arc and divided the 40 per cent. X-ray angle into spiral angle and crystallite angle. In table 22 are given the 40 per cent. X-ray angle and the spiral angle, as reported by DeLuca<sup>60</sup> for two varieties of widely differing X-ray angle, and in the final column the extension of the spiral, if it unfolds completely, which has been calculated from the spiral angle.

Since the deduced values of the spiral angle are approximately half the empirical 40 per cent. X-ray angle, the spiral angles

still differ according to variety in nearly the same ratio as the 40 per cent. X-ray angles.

TABLE 22.

Variety	40 per cent. X-ray angle ( ° )	Spiral angle ( ° )	Ext. due to unfolding the spiral ( % )
Belgien Congo	39.5	17.7	5.0
Strain 330	27.7	11.9	2.2

If the spiral unfolds completely, it will provide a percentage extension which is comparatively small especially in strain 330. The remainder of the extension to break ( $E_f$ ) may probably be attributed to the extension of the amorphous regions. For these amorphous regions to be in a position to contribute to the extension to break, they should be within the fibrils and not in between as suggested by Hearle,<sup>94,95</sup>

Hearle,<sup>95</sup> analyzing the extension to break of the cotton fibre, assumes that the extension of the fibre is due to:

- a. the extension due to unfolding the spiral,
- b. the extension of the crystalline fibrils themselves, and
- c. the extension of the amorphous regions.

As Hearle<sup>95</sup> assumes the fibrils to be wholly crystalline

and the amorphous regions to be in between them, then these amorphous regions will not actually contribute to fibre extension, since the total extension of (a) and (b) would be a limiting factor. Only the amorphous regions within the fibrils could contribute to fibre extensibility.

The possible contribution of the amorphous regions, within fibrils, means that the percentage crystallinity will affect fibre extensibility. If differences in crystallinity exist between the different varieties, then the extension due to the amorphous regions might differ. If no difference in crystallinity exists, then the contribution of the amorphous regions will be nearly equal ( it might be affected to some extent by possible differences in the molecular orientation in these regions ).

As raw cottons possess equal percentage crystallinity,<sup>100</sup> the possible contribution of the amorphous regions could be examined only indirectly. As mercerization at constant length does alter the percentage crystallinity ( 70 % and 40 % of cellulose is in <sup>the</sup> crystalline state in raw and mercerized cotton respectively<sup>100</sup> ), but does not alter greatly the spiral angle,<sup>61</sup> this procedure was used in investigating the role of the percentage crystallinity. The six samples of Rex variety were mercerized at constant length. Their structural characteristics and mechanical properties were investigated. The extension to

break ( $E_f$ ) for the raw and the mercerized samples is shown in table 23 .

TABLE 23.

Sample	Fibre age (days)	$E_f$ (raw ) ( % )	$E_f$ (merc.) ( % )	$E_f(\text{raw}) - E_f(\text{merc.})$ ( % )
	30	8.66	8.57	+ 0.09
R., I.	40	8.64	11.59	+ 2.95
	60	7.43	12.10	+ 4.67
	30	11.66	14.08	+ 2.42
R., II.	40	9.11	8.28	+ 0.73
	60	8.00	11.07	+ 3.07

The difference in the extension to break between the raw and mercerized samples, ( $E_f$  (raw) -  $E_f$  (mercerized)), is equal to the difference in extension resulting from the increased percentage amorphous regions and the possible loss in extension due to improvement in orientation in the mercerized samples. This difference is appreciable in the mature samples, showing that the contribution of the added amorphous regions is appreciable although it is not possible at this stage of work to find quantitatively how much extension is due to the increase in

the percentage amorphous regions. However it seems reasonable to say that the original amorphous regions in the raw cotton contribute appreciably to fibre extension to break.

The stress-strain curve, ( after removing the contribution of the convolutions ), of the mercerized samples has the same shape as the original stress-strain curve of the raw samples. This is possibly due to the increase in the percentage amorphous regions which, by contributing to fibre extension, have a similar effect to that of the convolutions.

In summing up the previous discussion it is concluded that the extension to break (  $E_f$  ) is due largely to the extension of the amorphous regions within the fibrils, and partly to the partial unfolding of the spiral itself. The proposed fibrillar structure put forward by FreyWyssling,<sup>83</sup> in which he assumes the fibrils to consist of crystalline microfibrils which are connected to each other through paracrystalline or amorphous regions, would account very well for the extension to break discussed before. If the spiral of the cotton fibre consists of fibrils connected to each other through amorphous regions, and these fibrils in turn consist of crystalline microfibrils which are also connected to each other through amorphous regions, then the extension to break (  $E_f$  ) will be due to;

- a. the unfolding of the spiral, and

b. the extension of the amorphous regions within the fibrils.

As the complete unfolding of the spiral provides only a small extension and the amorphous regions might provide much higher extension by orientating their molecules, the total theoretical extension ( which might be called the inherent extension ) is possibly much greater than the recorded extension to break. The failure of the spiral to unfold completely as well as the failure of the molecules in the amorphous regions to orientate themselves before the fibre breaks leads to only part of the theoretical or inherent extension being realized.

## (1) EFFECT OF MATURITY ON FIBRE MECHANICAL PROPERTIES

The effect of maturity or the degree of wall thickening on fibre mechanical properties is apparent in figures 23,24,29-32. It is clear that as fibre wall thickness increases, the stress-strain curve shifts towards higher stress per unit strain. The changes in the mechanical properties for the three successive degrees of wall thickening of Samaru 26J (G) and Rex varieties are given in the following.

(a) Extension to break ( tables 18,19 )

The four samples behave somewhat differently; for Samaru 26J (G)  $E_m$ ,  $E_p$ , and  $E_f$  are higher for samples of lower and higher degrees of wall thickening ( fibres 30 and 50 days old ) than the sample of intermediate degree of wall thickening ( 40 days old ). For the Rex variety, the sample of intermediate degree of wall thickening ( 40 days old ) of R., I., shows higher  $E_m$  and  $E_p$  than the other two samples of lower and higher degrees of wall thickening ( fibres 30 and 60 days old ). While for R., II. both  $E_m$  and  $E_p$  decrease as the degree of wall thickening increases. In both R., I. and R., II.,  $E_f$  decreases as the degree of wall thickening increases.

(b) Tensile strength ( table 18 )

The sample of intermediate degree of wall thickening

of Samaru 26J (G) shows a somewhat smaller measured tensile strength ( $T_m$ ) than the other two samples of lower and higher degrees of wall thickening. The projected tensile strength ( $T_p$ ) shows a small increase for fibres of intermediate degree of wall thickening than fibres of lower degree of wall thickening, and a marked increase for fibres of higher wall thickening (60 days old). For the Rex variety both the measured and projected tensile strength increase as the degree of wall thickening increases. The difference between fibres of lower and intermediate degrees of wall thickening is greater than that between fibres of intermediate and higher degrees of wall thickening.

(c) Work of Rupture ( table 21 )

The sample of intermediate degree of wall thickening of Samaru 26J (G) shows a smaller work of rupture ( $WR_m$  and  $WR_p$ ) than the other two samples of lower and higher degrees of wall thickening. The mature sample (50 days old) shows appreciably higher work of rupture. For the Rex variety, the sample of intermediate degree of wall thickening shows a somewhat higher work of rupture ( $WR_m$  and  $WR_p$ ) than the mature sample, and appreciably higher than the sample of lower degree of wall thickening.

(d) Initial Young's modulus and stiffness ( table 20 )

For Samaru 26J (G), the sample of intermediate degree of

wall thickening shows a smaller initial Young's modulus than the other two samples of lower and higher degrees of wall thickening. For the Rex variety, the sample of intermediate degree of wall thickening shows a higher measured initial Young's modulus than the other two samples. For both Samaru 26J (G) and Rex varieties, the calculated initial Young's modulus increases as the degree of wall thickening increases. Also the stiffness increases as the degree of wall thickening increases.

The changes in the initial Young's modulus and stiffness with the increase in the degree of wall thickening ( table 20 ), especially for R., I. and R., II. ( table 24 ), agree with the changes in the orientation for the successive cellulose layers discussed before ( page 122 ).

## (2) EFFECT OF FIBRE LENGTH ON FIBRE MECHANICAL PROPERTIES

( tables 17 - 21 )

The effect of fibre length, within a sample, on the stress-strain curve is apparent in figures 25, 26. The stress-strain curve shifts towards higher stress per unit strain for the longer fibres. This difference is more apparent between fibres 28 mm. long and fibres 36 mm. long. The extension to break (  $E_m$  and  $E_p$  ) is somewhat higher for shorter fibres. The tensile strength (  $T_m$  and  $T_p$  ), the work of rupture (  $WR_m$  and  $WR_p$  ),

TABLE 24 . The orientation, initial Young's modulus, and stiffness of fibres of successive degrees of wall thickening.

Sample	Fibre age (days)	Fibre density ( $\text{g cm}^{-3}$ )	Orientation 40 per cent. Av. X-ray - Av. orien. conv. angle		Initial Young's modulus ( $\text{g/tex}$ )	Stiffness ( $\text{g/tex}$ )
			$\bar{\Phi}$	$\theta^\circ$		
					$Y_c$	$S_{pa}$
R., I.	30	0.655	35.5	40.1	347	206
	40	0.712	33.9	39.7	582	306
	60	1.063	32.8	39.8	623	322
R., II.	30	0.543	37.9	40.6	287	182
	40	0.780	32.2	39.5	697	332
	60	0.988	31.4	38.5	670	389

the initial Young's modulus, and the stiffness are higher for longer fibres with the biggest difference between fibres 28 mm. and 36 mm. long.

#### EFFECT OF GIBBERELLIC ACID TREATMENT ON FIBRE MECHANICAL PROPERTIES

The effect of gibberellic acid treatment on measured extension to break, tensile strength, and work of rupture has been reported earlier ( see page 139 ). After analyzing the stress-strain curve for the various factors that contribute to or modify the curve, the effect of gibberellic acid could be further examined in the following comparison between the control samples and the samples treated with gibberellic acid.

##### (a) Extension to break

(1) The projected extension to break (  $E_p$  ) ( table 18 )

The difference between the control and treated samples of Samaru 26J (G) and Samaru 26J (N), which ranges between 0.5 and 1.3 percentage extension is small, but the trend is consistent with the control samples having the higher extension to break. The control and the treated samples of Sea Island show nearly equal extension to break. The control samples of the Rex variety show lower extensions to break varying between 0.4 and 2.2 percentage extension.

(2) The extension to break due to fibre fine structure (  $E_f$  )  
 ( table 19 )

For Samaru 26J (G), Samaru 26J (N), and Sea Island the differences between the control and the treated samples are not significant. For Rex, the control samples show lower extensions varying between 0.5 and 0.3.0 percentage extension.

(b) Tensile strength (  $T_p$  ) ( table 18 )

For all the samples under investigation, except Samaru 26J (N), the control samples show lower tensile strength. For Samaru 26J (G); the immature fibres 30 and 40 days old of the treated samples show tensile strength 7.5 and 6.5 % higher than the respective control samples. The mature fibres, 50 days old, of 20, 28, and 36 mm. length the treated samples are 11.2, 9.0, and 18.1 % stronger respectively than the control samples. For Sea Island, the treated sample is 5.5 % stronger than the control one. For Rex; samples 30, 40, and 60 days old, the treated samples are 18.4, 14.8, and 30.1 % , stronger, respectively, than the respective control samples. For Samaru 26J (N), the treated sample is 6.8 % weaker than the control one.

(c) The projected work of rupture (  $WR_p$  ) ( table 21 )

For Samaru 26J (N), the control sample shows higher work of rupture. For Samaru 26J (G) and Sea Island, the control samples

show slightly lower work of rupture. For the Rex variety, the control samples show appreciably lower work of rupture.

The work of rupture due to fibre fine structure ( $WR_{pa}$ ) shows nearly the same trend as the projected work of rupture ( $WR_p$ ).

(d) The initial Young's modulus and the stiffness ( table 20 )

For the Samaru 26J (G) and Rex varieties, the control samples show lower initial Young's moduli and stiffness. For Samaru 26J(N) and Sea Island, both the control and the treated samples show nearly equal values.

The lower extension to break ( $E_m$ ) shown by the treated samples of Samaru 26J (N) and Sea Island is due in part to the lower contribution of the convolutions to the extension to break in these samples ( see table 19 ), and in part to the effect of the increased major weak places. In the Rex variety, the contribution of the convolutions is nearly equal in the control and treated samples, and hence ( $E_m$ ) is nearly equal. Both the projected extension to break ( $E_p$ ) and the extension due to fibre fine structure ( $E_f$ ) are higher for the treated samples of Rex. These samples possessed higher tensile strength than the control samples, this possibly enabled the fibres to make use of a greater proportion of the inherent extension before breakage.

The increase in the projected tensile strength of all the treated samples ( except Samaru 26J (N) ) is possibly due partly to the difference in orientation ( as in the Rex variety ) and partly to other factors one of which might be the smaller diameter in the treated samples ( Samaru 26J (G) and Rex ).

The work of rupture, which is approximately the product of the extension to break and the tensile strength is consequently affected by the differences in them.

It is concluded that gibberellic acid treatment has resulted in an increase in the projected extension to break, tensile strength, work of rupture, initial Young's modulus, and stiffness of Samaru 26J (G), Rex, and Sea Island varieties. The effect on Samaru 26J (N) has been nil. This positive effect on extension to break, tensile strength, and work of rupture has been largely offset in several samples ( Samaru 26J (G) and Sea Island ) by the opposite effect of the increased major weak places caused by gibberellic acid treatment.

The dependence of the effect of gibberellic acid treatment on variety and environmental conditions is similar to its effect on fibre length. The effect of gibberellic acid treatment is appreciable and positive on the Rex variety, small and negative on Sea Island: this is a varietal effect. It is also positive ( with regard to the projected values ) on Samaru 26J (G) and negative on Samaru 26J (N): this is an environmental effect.

## A P P E N D I X I

## THE ROLE OF THE CONVOLUTIONS IN FIBRE EXTENSIBILITY

The presence of the convolutions is one of the characteristic features of cotton fibres. As these convolutions run in opposite directions at different intervals, there is no reason why they should not unfold as the fibre is subjected to load. By their unfolding, the convolutions could contribute to fibre extensibility.

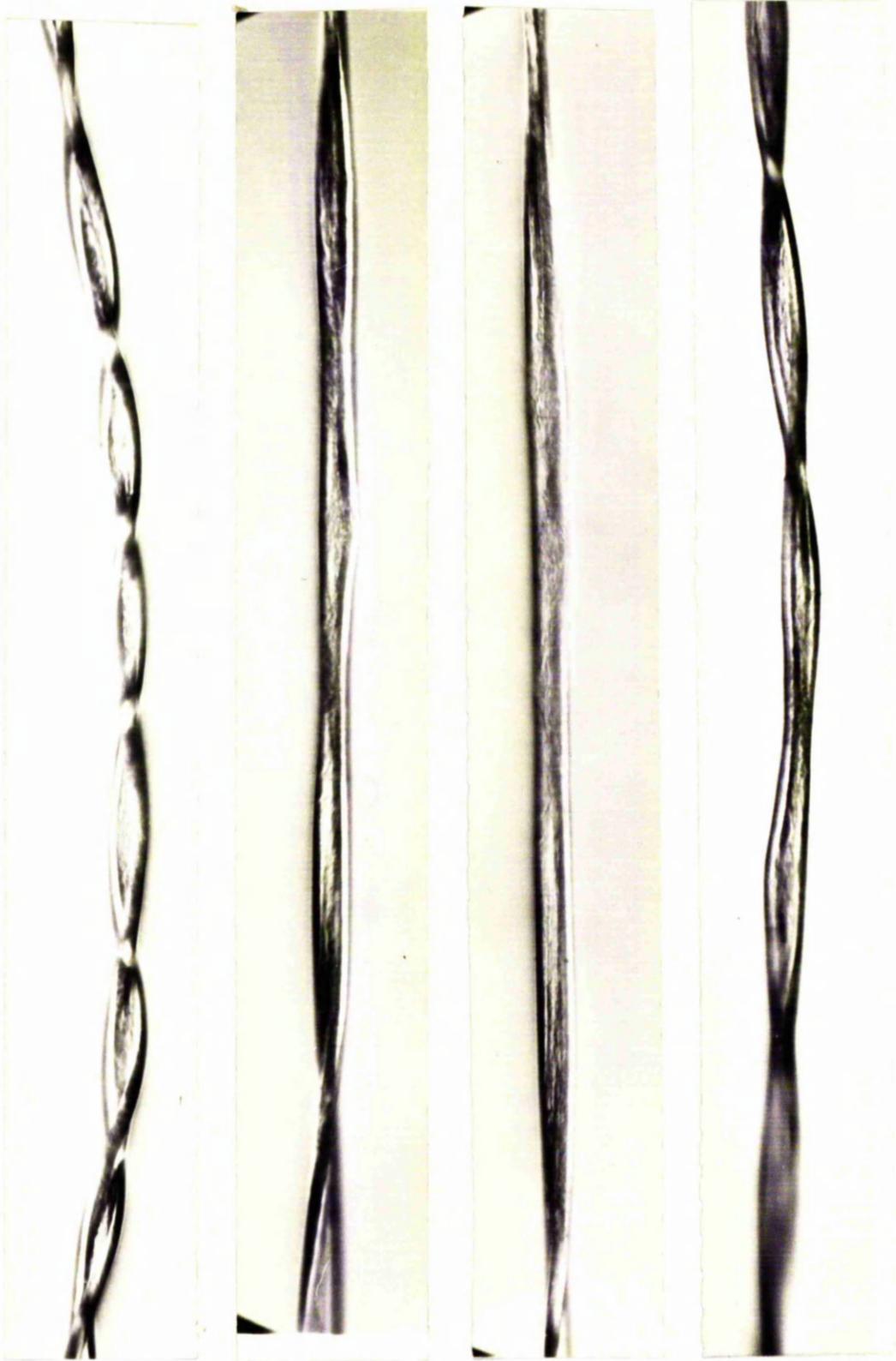
In order to find whether the convolutions contribute to fibre extensibility or not, an experiment was carried out on the mature sample ( 60 days old ) of the Rex variety ( R., I. ) of 27 mm. long. The fibres were sorted out, under the polarizing microscope, into three groups according to their maturity and consequently the number of convolutions per unit length; the immature and the very mature fibres with a small number of convolutions per unit length, and fibres of intermediate maturity with a greater number of convolutions per unit length. The actual convolution angle and the percentage convoluted portion were measured for each group and the percentage extension due to unfolding the convolutions was calculated. The fibres were tested on the Cambridge Extensometer at a constant rate of extension under the conditions recorded in page 57 and the results are shown in table 25.

TABLE 25.

	Immature fibres	Mature fibres	Very mature fibres
Number of convolutions/mm.	1.5	7.75	1.3
Actual convolution angle	26.3°	26.3°	23.5°
Convolute <sup>d</sup> portion %	9.0	41.0	6.5
% Extension due to unfolding the convol. ( $E_c$ )	1.04	4.72	0.6
Fibre extension to break % ( $E_m$ )	11.5	13.1	10.7

Fibres of intermediate degree of maturity and the highest number of convolutions per mm. showed a higher extension to break than fibres of either low or very high maturity which possessed fewer convolutions. These results could be taken as evidence that at least a sizeable proportion of the convolutions does unfold during fibre extension to break as load is applied, and thus contributes to fibre extensibility.

The unfolding of the convolutions on stretching was further investigated by stretching single fibres in a special frame ( see page 60 ) under the microscope. It was observed



A

B

C

D

FIGURE 33



A

B

C

D

FIGURE 3A

that, as load was applied and the fibre was stretched from both ends, the convolutions moved giving the fibre the appearance of rotating about its axis. When two convolutions running in opposite directions met, they neutralized each other and unfolded or disappeared. The remaining convolutions followed in the same manner. When all the convolutions were unfolded, the fibre appeared stable without rotating around its axis until it broke. It was observed also that if two convolutions running in the opposite directions neutralized each other and unfolded during the early process of extension, and then the load was removed or the direction of stretching was reversed so that the fibre returned to its original length, they <sup>re-</sup>appeared at once, but if the stretching process was carried out further until most of the convolutions disappeared, only a few convolutions re-appeared when the load was removed. Figures 33 and 34 show the unfolding of the convolutions as a fibre was extended. In figure 33 the photographs were taken under the polarizing microscope with the analyzer parallel to the polarizer, while in figure 34 the analyzer was set perpendicular to the polarizer, so that two reversals appear as dark bands and can be taken as definition points. In each of the two figures, the photographs were taken for one fibre at successive stages of extension.

Figures 33,34 show the following:

- (a) The convoluted fibre before being extended, in figure 34 six convolutions appear between the two reversals.
- (b) As the fibre was extended: the convolutions have begun to unfold and many of them have actually disappeared.
- (c) As the fibre was extended further: almost all the convolutions have unfolded and disappeared. The fibre appears as a flat and straight ribbon.
- (d) When the extension direction was reversed and the fibre was left free without being subject to any load ( not returned to the original length because in such a case focusing was found impossible ): some convolutions start to re-appear ( the photograph was taken 15 minutes after the removal of the load ).

From figures 33,34 it is concluded that the convolutions do unfold during fibre extension and thus contribute to fibre extensibility. The percentage extension due to unfolding the convolutions is dependent on the actual convolution angle and the percentage convoluted portion of the fibre, and as these two latter factors are dependent on the cotton variety and the maturity, the percentage extension due to unfolding the convolutions is consequently dependent on variety and maturity. It varied between 1.5 and 4.9 contributing 18 % and 41 % of the

total extension to break (  $E_{in}$  ) of Sea Island ( S.I., II. ) and Rex ( R., II. ) respectively.

To determine the role of the convolutions in the stress-strain curve, three points should be known:

- a. how much extension is due to the unfolding of the convolutions,
- b. at what stage of the stress-strain curve the convolutions completely unfold, and
- c. what stress is required to unfold the convolutions.

a. has already been investigated in the observations of stretching under the microscope and in order to investigate b. and c., an experiment was carried out on a sample of the Rex variety ( R., I. - 60 days old - 27 mm. long ). Fifty fibres were examined with 1 cm. test length. The single fibres were examined under the microscope at fairly constant rate of extension of approximately 4 mm./minute. The percentage extension at which the convolutions disappeared and the percentage extension at break were measured and the mean values found. The convolutions were found to disappear at 8.4 % extension, which is very near to the extension at the second point of inflexion ( 8.9 % ) in the measured stress-strain curve of this sample. The extension to break ( 10.9 % ) coincided fairly with that recorded on the Cambridge Extensometer for this sample ( 11.4 % ). This means

that most of the extension contributed by the unfolding of the convolutions is confined to the first and second stages of the stress-strain curve. Consequently, the contribution of the convolutions to fibre extensibility may be subtracted from the extension at the second point of inflexion and the stress-strain curve re-plotted. The stress required to unfold the convolutions must be equal to the stress at the second point of inflexion and therefore the convolutions do not make any contribution to the tensile strength of the fibres.

The replotted stress-strain curves show definite difference in shape from the original ones. The curves show only two stages of extension ( instead of three stages in the original ones ), the initial stage followed by a second stage in which stress is proportional to strain ( see figures 23 - 32 ). The calculated initial Young's modulus (  $Y_c$  ) and stiffness (  $S_{pa}$  ) are appreciably higher than the measured ones (  $Y_m$  and  $S_p$  ). The contribution of the convolutions in the work of rupture is appreciable and comparable to their contribution to fibre extension to break ( see tables 20, 21 ).

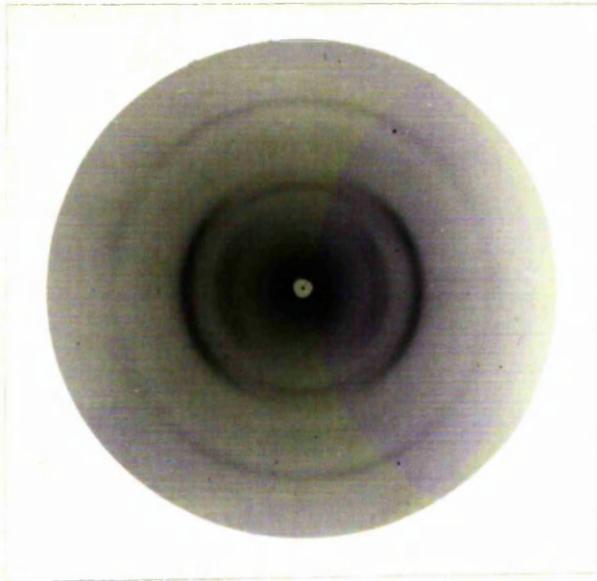


Plate 1. Samaru 26J (G),  
( S., IV. )  
30 days old .

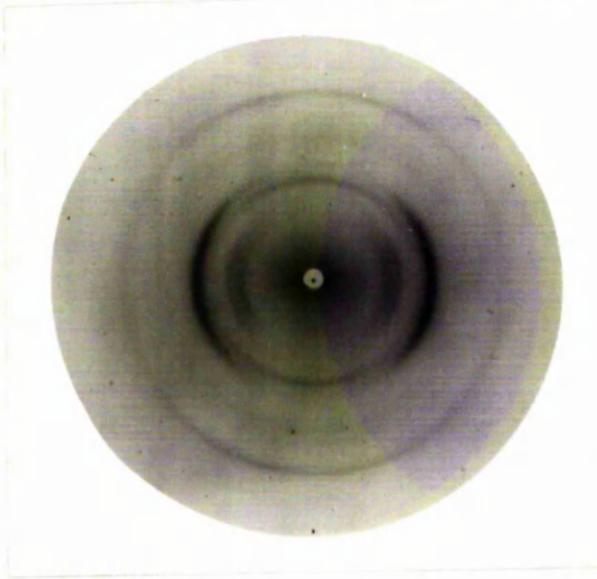


Plate 2. Samaru 26J (G),  
( S., IV. )  
40 days old.

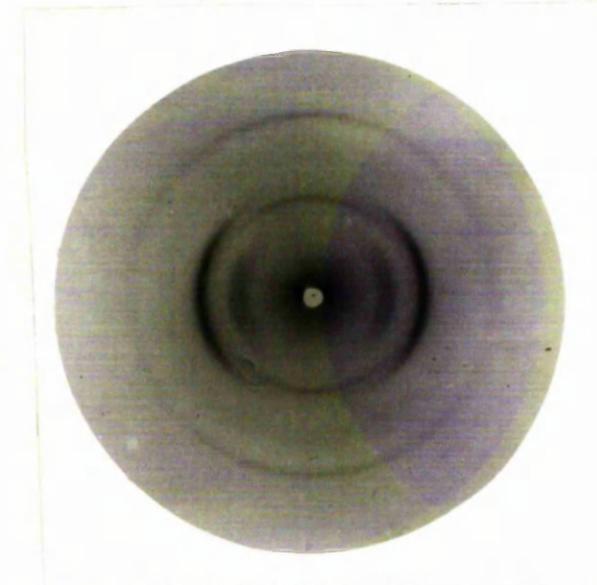


Plate 3. Samaru 26J (G),  
( S., IV. )  
50 days old.

## R E F E R E N C E S

1. Adderley, A. Shirley Inst. Mem., 1 , 151, (1922)
2. Ahmad, N. Indian Central Cotton Comitte, Tech. Lab. Bull., Series A, No.69, 18, (1933)
3. Anderson, D.B., and Kerr, T. Ind. & Eng. Chem., 30, 48, (1932)
4. Anderson, D.B., and Kerr, T. Bot. Rev., 1 , 52, (1935)
5. Anderson, D.B., and Moore, J.H. Amer. J. Botany, 24 , 503, (1937)
6. A.S.T.M. Standards on Textile Materials, (1954)
7. Atsuki, K., and Okajima, S. The Journal of the Soc. of Chem. Ind., Japan, 40 , 361, (1937)
8. Audus, L.J. "Plant Growth Substances," London, (1959)
9. Ayyar, V.R., and Ayyangar, G.S. Emp. Cotton Gr. Rev., 10 , 21, (1933)
10. Baily, A.J., and Brown, R.M. Ind. & Eng. Chem., 32 , 57, (1940)
11. Bailey, I.W. Ibid, 30 , 40, (1938)
12. Balls, W.L. "The Development and Properties of Raw Cotton," London, (1915)
13. Balls, W.L. Proc. Roy. Soc.(London), B90, 542, (1919)
14. Balls, W.L. ibid, B95 , 72, (1923)

15. Balls, W.L. "Studies of Quality in Cotton,"  
London, (1928)
16. Balls, W.L., and  
Hancock, H.A. Proc. Roy. Soc., London, B93, 426,  
(1922)
17. Balls, W.L., and  
Hancock, H.A. *ibid*, B99 , 130, (1926)
18. Barker, H.D., and  
Berkley, E. U.S. Dept. Agric., Tech. Bull., No.931,  
36, (1946)
19. Barker, H.D. U.S. Bur. of Plant Industry, Soils,  
and Agric. Eng., (1946)
20. Barritt, N.W. Ann. App. Biol., 16 , 438, (1929)
21. Barritt, N.W. Emp. Cotton Gr. Rev., 9 , 126, (1932)
22. Barrows, F.L. Contrib. Boyce Thompson Inst., 11 ,  
161, (1940)
23. Becke, F. Sitzungber. Akad. Wiss. Wien., 102(i),  
358, (1895)
24. Berkley, E.E., and  
Woodyard Ind. & Eng. Chem., 10 , 451, (1938)
25. Berkley, E.E. Text. Res., 9 , 355, (1939)
26. Berkley, E.E. Text. Res. J., 18, 71, (1948)
27. Berkley, E.E., and  
King, C.J. U.S. Dept. Agric., Tech. Bull.,  
No. 949, (1948)
28. Berkley, E.E. Text. Res. J., 19 , 362, (1949)

29. Berkley, E.H., et al. U.S. Dept. Agric., Tech. Bull.,  
No. 949, (1948)
30. Betrabet, S.M., et al. J. Sci. and Indus. Res., 19A, 91, (1960)
31. Betrabet, S.M., et al. Text. Res. J., 33 , 720, (1963)
32. Bigler, N. J. Text. Inst., 53 , A578, (1962)
33. Bowman, F.H. "Structure of the Cotton Fibre," (1908)
34. Bradford, W.W., and Agron. J., 50 , 648, (1958)  
Ewing, E.C.
35. Brian, P.W., and Physiol. Plantarum, 8, 669, (1955)  
Hemming, H.G.
36. Brown, K.C., et al. J. Text. Inst., 21 , T186, (1930)
37. Brown, K.C., et al. Shirley Inst. Mem., 9 , 1, (1930)
38. B.S. Handbook No. 11, "Methods of Test for Textiles," 3rd ed.,  
(1963)
39. Bunn, C.W. "Chemical Crystallography," Oxford, (1945)
40. Burley, S.T. jr., and U.S. Dept. Agric. Publication, (1953)  
Rouse, J.T.
41. Calkins, E.W.S. Text. Res. J., 31 , 176, (1961)
42. Campbell, K.S. Text. Manufacturer, 87, 201, (1961)
43. Catlett, M.S., et al. Text. Res. J., 21 , 880, (1951)
44. Christidis, B.G. "Cotton Growing Problems," New York, (1955)
45. Chytiris, T. Text. Res. J., 31 , 175, (1961)
46. Clegg, G.G., and J. Text. Inst., 15 , T14, (1924)  
Harland, S.C.

47. Clegg, G.G. Shirley Inst. Mem., 5 , 223, (1926)
48. Clegg, G.G. J. Text. Inst., 23 , T35, (1932)
49. Clark, G.L. Ind. & Eng. Chem., 22 , 474, (1930)
50. Clavert, M., et al. J. Text. Inst., 15 , T8, (1924)
51. Clor, N.A., et al. Fla. Physiol., 33 , 39, (1958)
52. Collins, G.E. Shirley Inst. Mem., 1 , 133, (1922)
53. Compton, J., and  
Haver, F.E. Contrib. Boyce Thompson Inst.,  
11 , 105, (1940)
54. Compton, J. Ind. & Eng. Chem., 31 , 1250, (1939)
55. Compton, J. Div. Cellulose Chem., Am. Chem. Soc.  
Cincinnati, Ohio, (1940)
56. Conrad, C.M., and  
Berkley, E.E. Text. Res., 8 , 341, (1938)
57. Conrad, C.M., et al. Text. Res. J., 21 , 726, (1951)
58. Coplan, M.J. Tech. Report, 53 - 21, U.S.A. Air Force.
59. Greely, J.J., et al. Text. Res. J., 26 , 789, (1956)
60. DeLuca, L.B., and  
Orr, R.S. J. Polymer Sci., 54 , 471, (1961)
61. DeLuca, L.B., and  
Orr, R.S. ibid, 54 , 475, (1961)
62. Denham, H.J. Shirley Inst. Mem., 2 , 61, (1923)
63. Dorset, R.C.M. Text. Manufacturer, 83 , 460, (1957)
64. Dransfield, M. Emp. Cotton Gr. Rev., 38 , 3, (1961)

65. DuPre jr., M. Text. Res. J., 29 , 151, (1959)
66. Ergle, D.R. Plant Physiol., 33 , 344, (1958)
67. Ergle, D.R. Plant Dis. Repr., 42 , 320, (1958)
68. Ergle, D.R. 54 th Annu. Proc. Assoc. Southern Agric. Workers, 227, (1957)
69. Farr, W.K. Contrib. Boyce Thompson Inst., 3 , 441, (1931)
70. Farr, W.K., and Eckerston *ibid*, 6 , 189, (1934)
71. Farr, W.K. *ibid*, 10 , 71, (1938)
72. Faust, R.C. Proc. Phys. Soc., London, B68, 1081, (1955)
73. Faust, R.C. "Physical Methods of Investigating Textiles," ed. R. Meredith & J. Hearle, New York, (1959)
74. Fiori, L.A., and Brown, J.J. Text. Res. J., 21 , 750, (1951)
75. Fiori, L.A., et al. *ibid*, 29 , 706, (1959)
76. Fiori, L.A. *ibid*, 31 , 178, (1961)
77. Fiori, L.A., and Grant, J.A. J. Text. Inst., 54 , P79, (1963)
78. Flint, E.A. Biological Rev., 25 , 414, (1950)
79. Fox, K., and Finch, K.B. Text. Res., 11 , 62, (1940)
80. Trendenberg, K., et al. Liebigs Ann., 494 , 41, (1932)

81. Frey-Wyssling, A. Kolloid Chem. Beih., 23 , 40, (1927)
82. Frey-Wyssling, A. Sci. Progr. Twent. Cent., 34, 249, (1939)
83. Frey-Wyssling, A. Science, 119 , 80, (1954)
84. Fuwa, T. Textile World, 73 , 49, (1923)
85. Goldwait, G.F., et al. ibid, 97, 105, (1947)
86. Grove, C.B.E., et al. Chem. & Ind. Rev., 954, (1956)
87. Grover, E.B., and "Handbook of Textile Testing and  
Hamby, D.S. Quality Control," New York, (1960)
88. Gulati, A.N. Agric. J. India, 25 , 313, (1930)
89. Gulati, A.N. Indian J. Agric. Sci., 4 , 471, (1934)
90. Hartshorne, N.H., and "Crystals and the Polarizing Microscope,"  
Stuart, A. 2nd ed., London, (1950)
91. Hartshorne, N.H. Science Progress, 50 , 10, (1962)
92. Haven, G.P. "Textile World Yearbook and Catalogue,"  
New York, (1939)
93. Hawkins, R.S., and J. Agric. Res., 40 , 1017, (1930)  
Serviss, G.H.
94. Hearle, J.W.S. J. Text. Inst., 53 , P449, (1962)
95. Hearle, J.W.S. J. Applied Polymer Sci., 7, 1207, (1963)
96. Hearle, J.W.S. "Fibre Structure," ed. J. Hearle &  
R. Peter, Manchester, (1963)
97. Hermans, P.H. "Contribution to the Physics of Cellulose  
Fibres," Elsevier, Amsterdam, (1946)
- 97a. Haworth, W.W. Helv. Chim. Acta, 11 , 547 (1928)

98. Hermans, P.H., et al. Rec. Trav. Chim., 65 , 427, (1946)
99. Hermans, P.H. "Physics and Chemistry of Cellulose  
Fibres," Elsevier, New York, (1949)
100. Hermans, P.H. Makromol. Chem., 4 , 204, (1954)
101. Hermans, P.H., and J. Polymer Sci., 14 , 397, (1954)  
Weidinger, A.
102. Hertel, K., and Text. Res. J., 26 , 479, (1956)  
Craven, C.J.
103. Hess, K., et al. Planta, 25 , 419, (1936)
104. Hessler, E., et al. Text. Res. J., 18 , 628, (1948)
105. Hessler, E., et al. ibid, 27 , 412, (1957)
106. Hessler, E., and ibid, 29 , 487, (1959)  
Wakeham, H.
107. Hessler, E. ibid, 29 , 858, (1959)
108. Hessler, E. ibid, 31 , 38, (1961)
109. Hessler, E. ibid, 18 , 679, (1948)
110. Heyn, A.N.J. ibid, 23 , 782, (1953)
111. Hirzog, M., and ibid, 18 , 71, (1948)  
Berkley, E.E.
112. Hock, C.W., et al. ibid, 11 , 200, (1941)
113. Honnegger, E. J. Text. Inst., 42 , P51, (1951)
114. Indian Central Cotton Committee, Tech. Lab. Bull., Series A,  
No. 69, 18, (1948)

115. Iyengar, R.L.N., and J. Text. Inst., 21 , T417, (1930)  
Turner, A.J.
116. Iyengar, R.L.N. Text. Res. J., 31 , 176, (1961)
117. Jackson, J.E., and Emp. Cotton Gr. Rev., 53 ,125,(1962)  
Fadda, N.R.
118. Kapadia, D.F. Indian Textile J., 45 , 127, (1935)
119. Kerr, T., and J. Arnold Arbor., 15 , 327, (1934)  
Bailey, I.W.
120. Kerr, T. Protoplasma, 27 , 229, (1937)
121. Kerr, T. Text. Res. J., 16 , 249, (1946)
122. Kohler, S. J. Text. Inst., 25 , T141, (1934)
123. Koshal, R.S., and ibid, 23 , T211, (1932)  
Ahmed, N.
124. Lang, A.G. J. Agric. Res., 56 , 507, (1938)
125. Levine, B.S. Science, 40, 906, (1914)
126. Liang, G.Y., and J. Polymer Sci., 37 , 385, (1959)  
Marchessault, R.H.
127. Lord, E. "Manual of Cotton Spinning," Volume 2,  
Part 1, Manchester, (1961)
128. Mark, H., and Z. Physik. Chem., B2 , 115, (1929)  
Meyer, K.H.
129. Mark, H. Chem., Revs., 26 , 169, (1940)
130. Menn, J.C. Shirley Inst. Mem., 4 , 53, (1925)
- 130a. Knatky, O. Kolloid-Z., 120, 24, (1951)

131. Mann, J.C., and Shirley Inst. Mem., 5 , 7, (1926)  
Peirce, F.T.
132. McDonald, A.W., et al. Text. Res. J., 27 , 641, (1957)
133. Meredith, R. J. Text. Inst., 36 , T107, (1945)
134. Meredith, R. ibid, 37 , T205, (1946)
135. Meredith, R. ibid, 42 , T275, (1951)
136. Meredith, R. ibid, 42 , T291, (1951)
137. Meredith, R. British J. of Appl. Phys., 4 , 369, (1953)
138. Meredith, R. J. Text. Inst., 47 , T499, (1956)
139. Merritt, J.M. J. Agric. & Food Chem., 6 , 184, (1958)
140. Meyer, K.H., and Ber., 70B , 266, (1937)  
Misch, L.
141. Meyer, K.H., and Helv. Chim. Acta, 20 , 232, (1937)  
Misch, L.
142. Morey, D.R. Text. Res., 5 , 483, (1935)
143. Moore, J.H. J. Am. Soc. Agron., 30 , 604, (1938)
144. Morlier, O.W., et al. Text. Res. J., 21 , 6 , (1951)
145. Morton, W.E., and J. Text. Inst., 45 , T774, (1954)  
Radhakrishnan, N.
146. Nanjundayya, C., and ibid, 29 , T75, (1938)  
Ahmed, N.
147. Nanjundayya, C. Indian Cotton Gr. Rev., 6 , 171, (1952)
148. Navkal, H., and ibid, 3 , 144, (1949)  
Sen, D.L.

149. Nickerson, R.F., and Leape, C.B. Ind. & Eng. Chem., 33 , 83, (1941)
150. Nishikawa, S., and Ono, S. Proc. Math.-Phys. Soc.-Tokyo, 7 , 131, (1913)
151. Nishikawa, S., and Ono, S. *ibid*, 8 , 296, (1914)
152. Obermiller, R.L. Text. Res., 6 , 325, (1936)
153. O'Kelley, J.C. Iowa State College J. of Sci., 26, (1952)
156. Orr, R.S., et al. Text. Res. J., 24 , 399, (1954)
157. Orr, R.S. *ibid*, 25 , 939, (1955)
158. Orr, R.S. *ibid*, 29 , 144, (1959)
159. Orr, R.S. *ibid*, 29 , 349, (1959)
160. Orr, R.S., et al. *ibid*, 31 , 302, (1961)
161. Osborne, G.G. Text. Res., 5 , 275, (1935)
162. Pearson, N.L. Text. Res. J., 20 , 152, (1950)
163. Pearson, N.L., et al. *ibid*, 25 , 961, (1955)
164. Peirce, F.T., and Stephenson, R.J. Shirley Inst. Mem., 5 , 239, (1926)
165. Peirce, F.T., and Lord, E. *ibid*, 17 , 25, (1939)
166. Phinney, B.O. "Encyclopedia of Plant Physiology," Volume 14, 1185, (1960)
167. Pillay, K.P.R., and Shankaranarayana, K. Text. Res. J., 31 , 515, (1961)

168. Polanyi, M. Naturwissenschaften, 9 , 288, (1921)
169. Prakash, J., and Text. Res. J., 32 , 954, (1962)  
Iyengar, R.L.N.
170. Preston, J.M. "Modern Textile Microscopy," London, (1933)
171. Preston, R.D. Biol. Rev., 14 , 281, (1939)
172. Preston, R.D., and Biochem. Biophys. Acta, 3 , 549, (1949)  
Wardrop, A.B.
173. Preston, R.D. "Molecular Architecture of Plant Cell  
Walls," London, (1952)
174. Radhakrishnan, T., et al. Text. Res. J., 29 , 392, (1959)
175. Rebenfeld, L., et al. *ibid*, 27 , 286, (1957)
176. Rebenfeld, L. *ibid*, 28 , 585, (1958)
177. Rebenfeld, L. *ibid*, 31 , 253, (1961)
178. Rebenfeld, L. *ibid*, 31 , 311, (1961)
179. Rebenfeld, L. *ibid*, 32 , 154, (1962)
180. Rebenfeld, L. *ibid*, 32 , 202, (1962)
181. Richardson, R.P., et al. U.S. Dept. Agric. Tech. Bull.,  
No. 545, (1937)
182. Richardson, R.P. Textile Digest, 69 , 1, (1940)
183. Rollins, M.R. Text. Res. J., 15 , 65, (1945)
184. Sands, J.E., et al. *ibid*, 30 , 620, (1960)
185. Segal, L., et al. Rev. Sci. Instr., 21 , 431, (1950)
186. Sen, K.R., and J. Text. Inst., 29 , T258, (1938)  
Ahmed, N.

187. Shifffield, F.M.L. Emp. Cotton Gr. Rev., 13 , 277, (1936)
188. Shah, J. Text. Res. J., 30 , 618, (1960)
189. Singh, T.C.N. Ann. Bot., 45 , 378, (1931)
190. Sisson, W.A., and Ind. & Eng. Chem., 5 , 296, (1933)  
Clark , G.L.
191. Sisson, W.A. Contrib. Boyce Thompson Inst., 8 ,  
389, (1937)
192. Sisson, W.A. ibid, 9 , 239, (1938)
193. Sisson, W.A. Text. Res., 7 , 425, (1937)
194. Sisson, W.A. Ind. & Eng. Chem., 27 , 51, (1935)
195. Sisson, W.A. J. Phys. Chem., 40 , 343, (1936)
196. Sisson, W.A. "Cellulose and Cellulose Derivatives,"  
(High Polymers, Volume 15), London, (1943)
197. Sookne, A.M., and J. Res. Natl. Bur. Standards, 26 ,  
Harris, M. 65, (1941)
198. Sookne, A.M. Am. Dyestuff Repr., 30, 29, (1941)
199. Sookne, A.M. Text. Res., 11 , 307, (1941)
200. Steinberger, R.L. ibid, 6 , 325, (1936)
201. Stowe, B.B., and Ann. Rev. of Plant Physiology, 8,  
Yamaki, T. (1957)
202. Sundaram, V., and Ind. Cotton Gr. Rev., 11, 490, (1957)  
Nanjundayya, C.
203. Sundaram, V. ibid, 15 , 74, (1961)
- 203a. Spons Lex, O.L. J. Gen. Physiol., 9 , 677, (1926)

204. Tallant, J.D., et al. Text. Res. J. 30 , 792, (1960)
205. Tallant, J.D.            ibid, 29 , 687, (1959)
206. Tallant, J.D., et al. ibid, 31 , 866, (1961)
207. Tripp, V.W., et al.    ibid, 21 , 887, (1951)
208. Tripp, V.W.            Anal. Chem., 24 , 1721, (1952)
209. Tripp, V.W.            Text. Res. J., 27 , 419, (1957)
210. Tripp, V.W.            ibid, 27 , 427, (1957)
211. Turner, A.J.            J. Text. Inst., 19 , T156, (1928)
212. Turner, A.J.            ibid, 20 , T233, (1929)
213. Turner, A.J., and     Indian Central Cotton Comitte, Tech.Lab.,  
Venkataramen, V.        Tech. Bull., Ser.B, No.17,48, (1933)
214. Tsien, P.C.            Text. Res. J., 19 , 330, (1949)
215. Underwood, G.J.        J. Text. Inst., 26 , T309, (1935)
216. Virgin, W., and        Text. Res. J., 26 , 177, (1956)  
Wakeham, H.
217. Wakeham, H., and     ibid, 21 , 187, (1951)  
Spicer, N.
218. Wakeham, H., and     ibid, 25 , 585, (1955)  
Spicer, N.
219. Wakeham, H., et al.   ibid, 29 , 450, (1959)
220. Whistler, R.L., et al. J. Res. Natl. Bur. Standards, 25 ,  
305, (1940)
221. Walhood, V.T.         Proc. 13th Ann. Beltsville Cotton Conf.,  
27, (1958)

222. Walhood, V.T. Proc. 13<sup>th</sup> Ann. Beltsville Cotton  
Conf., 30, (1958)
223. Webb, R.W., and Off. of Marketing Services, U.S. Dept.  
Richardson, H.B. Agric., Washington, D.C., (1945)
224. Weiss, L.C., et al. Text. Res. J., 31 , 787, (1961)
225. Wergin, W. Angew. Chem., 49 , 843, (1936)
226. Wergin, W. Kolloid-Z., 100 , 436, (1942)
227. Woods, H.J. "Recent Advances in the Chemistry of  
Cellulose and Starch," ed. Honeyman, J.,  
London, (1959)
228. Yoseif, A. The Second Cotton Conf., Cairo, (1958)
229. Yoseif, A., et al The Third Cotton Conf., Cairo, (1962)