INFLUENCE OF SOME CHARACTERISTICS OF CONIFEROUS WOOD TISSUES ON SHORT-TERM CREEP

by

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ABSTRACT

The hypothesis is examined that short-term creep response of earlywood and latewood tissues of some coniferous species, stressed in tension parallel to the grain, is a function of microfibril angle of the S 2 layer of and relative degree of crystallinity in the tracheid cell wall, along with specific gravity of that wood tissue and its extractives content.

A new technique was developed to measure the total creep that occurred over a 60-minute period of time for small specimens (nominally 0.010 in. thick) of Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) (normal and compression wood), Sitka spruce (Picea sitchensis (Bong.) Carr.) and western hemlock (Tsuga heterophylla (Raf.) Sarg.), taken from earlywood and latewood zones of the same increment. Total creep was determined at two initial deformation levels, 3,000 microin. per in. (strain level No. 1) and 6,000 microin. per in. (strain level No. 2).

Microfibril angle was measured by a modified mercury impregnation method, while cell wall crystallinity was determined on small, unoriented pellets by the X-ray diffraction technique. Air-dry specific gravity (oven-dry weight) and alcohol-benzene plus hot water extractives

were also determined by conventional methods.

Multiple regression analyses were carried out and prediction equations, based on the experimental results, have been constructed. It is shown that the variability in total creep response can best be explained by using the prediction equation which contains microfibril angle of the S2 layer, specific gravity and extractives content. The multiple coefficients of determination (R^2) using this subset of variables are 0.7680 and 0.8550 for initial strain Numbers 1 and 2, respectively.

Cell wall crystallinity was eliminated from the prediction equations as the least important variable due to its high inverse correlation with the microfibril angle of the S2 layer (r=0.9284). Two possible reasons are suggested to explain this correlation. First, in the case of a small angle, the scatter around the mean microfibril angle is smaller and the microfibrils probably lie almost parallel to each other. As a result, the relative degree of amorphous material required to fill the micro-spaces between microfibrils would be smaller. Considering the case of a large microfibril angle, the microfibrils are probably not parallel to each other; consequently, relatively large micro-spaces would be occupied by the amorphous material.

A second possible reason for this relationship may be that cellulose chain molecules, in the case of a small microfibril angle, will have a better chance for increased

frequency of cross links (bonding between neighbouring chains) along their unit length. Consequently, a tendency of improved geometric order should be observed with better chain coherence in the resulting cellulose as compared to situations associated with tracheids characterized by larger microfibril angle. It must be indicated that reasons for this high degree of correlation, as noted above, remain conjectural.

Among the structural features studied, microfibril angle was shown to control creep response to the greatest extent As it increases, total creep increases, the reason being that with a small angle, microfibrils are in a position to bear most of the applied load and therefore their relative movement towards a smaller angle would be less. This results in a small plastic deformation. In the case of a large angle, there is a possibility that the microfibrils have a large tendency to move to a smaller angle causing a large creep response.

Wood samples of low specific gravity creep more than those with high specific gravity. This behavior is explained by the higher relative per cent of the S2 layer in the latter.

Extractives are shown to contribute significantly to the variation in total creep. They probably act as plasticizers causing a reduction in the primary and secondary bonding between microfibrils. This would facili-

tate the movement of the stiff inextensible microfibrils to accommodate the creep-inducing stresses.

Results obtained in this study were compatible with the proposed hypothesis.

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INTRODUCTION

Wood has been used for many years as a structural material in buildings, bridges and innumerable other engineered fabrications. Its mechanical behavior is found to be influenced by numerous factors such as moisture content, temperature, species of wood and direction of applied stress or strain. In addition, the factor of time has been given especial consideration with respect to stress-strain relationships.

Although several applications of wood are based on its elastic properties, rheological studies reveal that wood exhibits both elastic and plastic properties. Frequently, the plastic properties dominate in its behavior. Time-dependent behavior manifests itself in several ways, namely creep, stress relaxation and damping capacity. There is evidence from linear viscoelastic studies, conducted at low stress levels, that these three rheological phenomena are related mechanistically.

Creep, one of the important rheological properties exhibited by wood, can be defined as a continuing deformation due to a constant load imposed over time. Since wood requires several modifications for conversion to a usable form, it undergoes applied stresses in manufacturing

processes such as machining, drying and pressing. In addition, it can be subjected, in use, to complex loading conditions. Whatever the source of stress, a resultant strain occurs and increases with elapsed time.

The rheological response of wood under constant stress has been studied almost exclusively using macro-samples. Very little is known about the microscopic and submicroscopic characteristics contributing to variations in creep response.

Wood is an extremely complex material. Basically its elastic properties, under the same environmental conditions, are determined by factors inherent in its structure. These may be summarized as follows:

1. The amount of cell wall substance present in a given volume of wood (specific gravity);
2. The proportionate chemical composition of the primary components of the cell wall (cellulose, hemicelluloses and lignin);
3. The supermolecular arrangement and orientation of wall material in the different tissues;
4. The kind, size, proportions and arrangement of the cells making up the woody tissues.

It is anticipated that these factors would also influence the rheological properties of wood. But virtually nothing is known about the role which the abovenoted variables play in controlling creep response of wood.

A thorough knowledge of the relationships between

creep response and microscopic and submicroscopic characteristics of wood is important. At the micro-level, such information is of fundamental scientific interest because it allows some inference as to the mechanism of creep. In addition, information obtained from well designed experiments could be combined with knowledge on other variables, which might be resolved at the macro-level, to provide guidence to the user of structural timber as well as to the wood scientist in selecting material of the required strength for a specific purpose.

The objective of this thesis is to examine the hypothesis that short-term creep of earlywood and latewood tissues of some coniferous species, stressed in tension parallel to the grain, is a function of microfibril angle of the S2 layer of tracheid wall, and relative degree of crystallinity in the cell wall, along with specific gravity of that wood tissue and its extractives content. Wood tissues taken from Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) (normal and compression wood), Sitka spruce (Picea sitchensis (Bong.) Carr.), and western hemlock (Tsuga heterophylla (Raf.) Sarg.) were used in the present study.

It is hoped that knowledge derived from a study of this nature will provide the means to better understand the rheological properties of wood.

LITERATURE REVIEW

A literature review on creep response as a function of structural features of the woody cell wall, specific gravity and extractives content can be divided logically into distinct areas, which are described below.

I. Minute Structure of Coniferous Woods

Coniferous woods consist of two major anatomical elements, the longitudinal tracheids and the radially extended ray parenchyma cells (wood rays) (105). The former constitutes up to 90 per cent of the wood volume and over 95 per cent of the wood weight (93, 105, 110, 124). In addition, different kinds of minor cell types may also be present, depending on species. The walls of tracheids consist of two ontogenetically distinct structures, the primary wall and secondary wall. The primary wall, the outermost layer, is formed at the time of cell division in the cambial zone of a tree. It surrounds the protoplast during the surface growth phase of the plant cell under differentiation (147). After the surface growth has ceased, a secondary cell wall is formed (Fig. 1).

Anatomical elements in wood are bonded together by an inter-cellular layer called the middle lamella (ML).

Wood cell walls consist of three groups of structural substances which Wardrop (147) has classified as framework, matrix and encrusting substances. The framework material of the cell wall is cellulose, which is aggregated in the form of microfibrils as developed during wall formation. These microfibrils were predicted by Frey-Wyssling (28) as early as 1937 and confirmed by others as structural units of cellulose. Hemicelluloses and other carbohydrate materials, excluding cellulose, are incorporated into the cell wall as the matrix forming substance, and are considered to be amorphous materials. Lignin is the main encrusting substance which begins to form with the matrix after some degree of cell-wall formation has taken place (147). Its conclusive chemical association with the framework (cellulose) is still widely debated. Thus, the cell wall can be considered as a three-phase material.

Ray parenchyma cells are different in their structure from tracheids and may affect creep response of wood due to the dislocation which occurs at the contact area between ray cells and tracheids. However, tracheids are only considered in this literature review since they constitute the main elements in coniferous wood structure.

II. Physical Nature of Cellulosic Cell Wall Structure

A. Cell Wall Organization

Cell wall organization studies are concerned 1) with the pattern of microfibril orientation with respect to cell axes and 2) with the layering within the secondary wall.

1. Primary Wall (P)

The primary wall consists of two kinds of microfibril orientation. On its inner surface, the microfibrils are oriented approximately in a transverse direction to the cell axis, but the orientation differs appreciably from this on the outer surface (52, 145) (Figures 1 and 2). No lamellation has been observed by Wardrop (147) in the primary wall layer which is considered to be thinner (0.1 to 0.2 μ (138)) relative to the thickness of other layers in the cell wall. Differences in thickness and proportioning of primary wall layer among wood species, and between trees, their growth zones, and tissues are also expected.

2. Secondary Wall

This wall is characterized by two features: microscopic layering and submicroscopic lamellation (147)(Figures 1 and 2). Its layering feature was observed by Bailey and Kerr (4), who interpreted the optical heterogeneity of the cell wall as being due to a differing microfibrillar

arrangement in the various layers. These layers are regarded by them as a thin outer layer (S1), a broad middle layer (S2) and a thin inner layer (S3) which may be absent in some genera such as Picea (147). These layers also differ in thickness. Timell (138) has reported values of 0.1 to 0.3μ, 1.0 to 5.0μ, and 0.1μ for the thickness of S1, S2 and S3, respectively. The different microfibril orientations in tracheid wall layers have been confirmed on the basis of polarization microscopy (154), light microscopy (156), X-ray diffraction analysis (113) and electron microscopy (83, 147).

Microfibrils in the S1 layer are oriented at a relatively large angle (close to 45°) with the longitudinal cell axis (146). Lamellation of the S1 layer was also indicated by the aid of electron microscopy. It consists of a few lamellae of alternating S and Z helical orientation; the number of these lamellae is different from one species to the other (23, 27, 52, 82, 146). However, further studies have shown that there may or may not be intermediate microfibril orientation (S12) between S1 and S2 layers (34, 147) (Fig. 2B).

The middle layer of the secondary wall (S2) accounts for as much as 80 to 95 per cent of the tracheid wall volume, while the primary cell wall and S1 layer account for only 5 to 15 per cent of the cell wall volume (59). Therefore, the S2 layer has been the one most thoroughly studied. In

this layer, the microfibrils are arranged at a steep (small) angle to the longitudinal cell axis, usually not in excess of about 30° (76), depending to some degree on the species. Furthermore, these microfibrils are arranged in numerous concentric lamellae making almost the same angle with the longitudinal cell axis (147) and exhibiting, therefore, a high degree of parallelism (52) (Figures 1 and 2).

The microfibrils in the inner layer of the secondary wall, S3, are arranged at quite a large, but variable angle to the longitudinal cell axis (19, 34). It is reported that this angle ranged from 70 to 90° (152). Lamellation of the S3 layer, with alternating S or Z helical microfibrillar orientation, was also observed (147). An intermediate microfibril arrangement (S23) has been found between S2 and S3 layers by Harada et al. (34) (Fig. 2B).

Besides the aforementioned layers, some species have an innermost layer called the warty layer (T) (75). It may consist of the remains of cytoplasmic contents of the cell which were deposited on the inner wall at the conclusion of wall development. However, existence of this layer is still a controversial issue in the literature. Forgacs (26), for example, used the warty layer to refer to the layer that was considered by the others as S3, while Harada et al. (34) used it to represent a layer additional to S1, S2 and S3. It should also be noted that local deviations occur in the aforementioned microfibril organization due to pit cavities in the cell wall (34).

In the case of compression wood tracheids (wood formed on the lower side of leaning stems of conifers), the secondary cell wall layers are reduced to the S1 and a modified S2 layer (16). This modified S2 layer has microfibrils oriented at an angle of about 45° to the longitudinal cell axis (16), which is larger than that found in the S2 layer of normal wood. It is also characterized by the presence of an extra layer of lignin between S1 and S2 (138). Furthermore, Côté and Day (16) have reported that the S1 layer of compression wood tracheids is thicker than that in normal wood. Another ultrastructural feature of compression wood was observed by Wardrop and Davies (153), who indicated the presence of the so-called 'helical checks' which follow the microfibril orientation in the S2 layer. Côté and Day (16) agreed with Wardrop and Davies on the reality of these checks which presumably originate during the formation of the S2 layer and which are not caused by drying of the cell wall. The chemical composition of compression wood is also different from that of normal wood. In general, the cellulose content is lower, as is the amount of galactoglucomannan, whereas galactan content is greater and lignin content is considerably higher than in normal wood (138).

B. Supermolecular Arrangement of Cellulose Chains

Based on light microscopy studies, it was found that tracheid walls consist largely of a great number of filaments (fibrils) wound helically with respect to the tracheid axis. However, after the advent of electron microscopy, it has been revealed that these fibrils are aggregations of finer filamentous units called microfibrils. Some research workers refer to these microfibrils as the basic supermolecular structural units in the plant cell wall (16, 118), while others have indicated that the microfibrils in turn are composed of finer or smaller elementary fibrils (35 Å by 35 Å) (3, 30, 79, 91). The main component of microfibrils and/or elementary fibrils is cellulose, generally considered as being composed of β-D-glucopyranose units linked together by a 1, 4 - glycosidic bond (41). The actual state of hemicelluloses in the living tree is largely unknown. Examples taken from other carbohydrate containing plants and living organisms suggest the possibility that the ability to crystallize or to form microfibrils are characteristics expandable to all native polysaccharides.

Cellulose is partly crystalline and gives an X-ray diffraction diagram by which the dimensions of the crystallographic unit cell can be determined. Based on this evidence, Meyer and Misch (86) were able to establish the crystallographic dimensions for a monoclinic unit cell of Cellulose I.

Thereby, each unit cell contains four glucose residues (Fig. 3). The glucose residues are joined together by three kinds of forces: along the a-axis by fairly strong hydrogen bonds; along the c-axis by much weaker van der Waal's forces; and along the b-axis, i.e., along the cellulose chain, by the β-1, 4-glucosidic bonds (70). Consequently, the cellulose lattice is both a chain lattice and a layer lattice. A convenient way of expressing the length of the chain molecules in cellulosic fibers is by using the term 'degree of polymerization (DP)', which represents the number of glucose residues in the chain molecule.

The supermolecular arrangement of the polymer chains within the wall structure has been the subject of many models. There is now a vast literature on the subject with some divergence of opinion among experts in the field. The most widely used models are described below (Figures 4 to 7).

1. Fringed Micellar Model

The fringed micellar model was proposed by Herrmann et al. (42) early in 1930 for cell walls, and for regenerated celluloses by many others such as Hermans (41) and Mark (80). Based on X-ray diffraction studies, Hengstenberg and Mark (40) suggested that crystalline regions in natural cellulose fibers had dimensions of 50 Å by 500 Å. Since the molecular length was much longer than 500 Å, it was proposed, according to the fringed micellar model, that the molecules pass

alternately through many crystalline and non-crystalline regions (Fig.4). In addition, it was assumed that the molecules are essentially fully extended chains (36). Many mechanical and physical properties of polymer behavior were explained on the basis of this model in spite of some divergences of view which appeared particularly in the early days and also recently (134).

2. Fringed Fibrillar Model

Electron microscopy studies have brought evidence for the existence of fine fibrils with widths of the order of 100 Å. Since it is impossible to fit fibrils of this size inside fibrils of the fringed micelle structure, Hearle (35, 37) proposed the fringed fibrillar model. According to this model, the fibrils are assumed to be long, imperfect crystals winding their way through the fiber structure and separated by non-crystalline regions (Fig.5). The long-chain molecules are also assumed to pass alternately through crystalline and non-crystalline regions, giving rise to a continuous network structure. The main difference between the two models, fringed fibril and fringed micelle, is that in the latter the non-crystalline (amorphous) and crystalline regions alternate along the fibrillar axis, whereas in the former the non-crystalline regions are located along the periphery of the fibril.

Jentzen (60) has found that the calculated Young's modulus of delignified longleaf pine (Pinus palustris Mill.)

tracheids agreed with the established theoretical value for perfectly oriented crystalline cellulose. Therefore, he suggested that the fringed fibrillar model is a better representation of tracheid structure than the fringed micellar structure. This model also received the support of Tripp et al. (140), and Rebenfeld (123) for the structure of cotton cellulose. But it has been criticized by Michie et al. (89), due to the inability of the model to explain the observed mechanical properties of regenerated cellulose.

3. Continuous Model

This model was suggested by Hess et al. (43), Ränby (122), and Stuart (133). According to their model, the crystalline lattice in cellulose fibers is continuous throughout the structure, although it includes regions of lower order which occur at intervals along the length, rather than the width of the microfibril. Two such proposals are shown in Fig.6.

4. Folded Chain Model

In the abovementioned supermolecular organization of cellulose chains, it has been assumed that the molecules are essentially fully extended chains. Recently there has been increasing evidence from ultrastructural studies that this organization is an over-simplification. Consequently, it has been suggested that the molecules in single polymer crystals are folded back and forth in layers. Since 1960,

at least five folded configurations have been proposed for native cellulose (3, 7, 21, 79, 139). According to Manley's model (79), the molecules form flat ribbons by folding back and forth, and the molecular chain axis is inclined at some preferred angle to the ribbon axis (Fig. 7b). If the ribbon is wound as a helix, the molecular chain axis becomes parallel to the fibril axis (Fig. 7a). This model was recently supported by Sullivan (134) for celluloses from six pines and two pored woods. Asunmaa (3) criticized Manley's model based on the concept that this model produces a lower apparent density for the cellulose in a microfibril than that measured for crystalline cellulose. Mark (81) has also criticized the folded chain model because of its inability to explain the low extensibility, very high strength, and lack of glass transition[1] behavior typical of synthetic polymers.

It must be indicated that microfibrils composed of fringed micelles, fringed fibrils, continuous or folded chain configuration, would all have different strengths and elastic properties from one another (36, 81). Therefore, by studying the strength and elastic properties of wood in any form or direction would, in fact, only reflect its ultrastructure.

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[1]Glass transition is described as the narrow region on the temperature scale where the thermal expansion coefficient undergoes a discontinuity and below which configurational re-arrangement of polymer chain backbones, if they occur at all, are extremely slow (1).

C. Cell Wall Crystallinity

1. Concept

Extensive studies over a period of years have led to the acceptance of the hypothesis that cellulose fiber and wood tracheids consist of both ordered or crystalline and disordered or amorphous regions. However, a proper definition of crystallinity has not been established. Those who deal with crystallinity from physical points of view define the 'crystalline region' as the portion which is in a state of perfect, three dimensional order and which gives rise to selective X-ray diffraction patterns (41, 143). On the other hand, those who approach the fine structure of fibers by chemical means consider the crystalline region as the portion having extreme resistance to chemical treatments, as compared with the accessible amorphous region (41). It should be pointed out that there is no sharply defined borderline which can be drawn between the two states of a cellulosic fiber, amorphous and crystalline regions. Rather, the distinction is made approximate (41). Furthermore, none of the physical or chemical methods actually measures lateral order or crystallinity distribution of the sample, but they do rank various cellulose materials according to their degree of crystallinity and they are thus useful for comparative purposes.

Mark (81) has recently questioned the reality of crystallinity measurements based on the idea that the presence

of voids on the surface of crystallites contributes also to X-ray scattering. Consequently he did not place much faith in relating mechanical behavior of fibers to crystallinity. However, one cannot ignore this parameter because it is one of the most important molecular characteristics of cellulosic fiber structure.

2. Relation to Mechanical Properties

A large volume of work has been done to relate mechanical properties of natural textile fibers to their crystallinity. Early in 1945 Conrad and Scroggie (15) found that elongation of regenerated cellulose yarns increased with accessibility, which is related inversely to crystallinity number.[2] Tripp et al. (141) and Ward (143) also indicated that high strength and low extensibility were associated with a high percentage of crystallinity.

In an attempt to relate crystallinity of cotton fibers to tensile strength properties, Krässig and Kitchen (72) used an approach similar to that used for regenerated cellulose. They assumed numbers of links between fringed

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[2] crystallinity number = $\frac{\bar{I}_1 - \bar{I}_M}{\bar{I}_1}$

where: $\bar{I}_1$ = average intensity of 101 interference, and

$\bar{I}_M$ = average intensity of minimum between 101 and the $10\bar{1}$ interference.

micelles. Such a study was criticized by Mark (81) who stated that this approach is unsound from the standpoint of theoretical mechanics. Unfortunately, he did not give reasons for his disagreement on their approach.

The studies of amine decrystallization treatment carried out by Orr et al. (102), Parker (106), Segal et al. (127), and Ward (143) indicate that the ultimate elongation increases but the strength may increase or decrease slightly due to decrystallization. Parker also found that this treatment caused abrupt decreases in elastic modulus and zero-span tensile strength of paper handsheets. However, the first creep (creep of specimens which have not been previously subjected to external loading) increased sharply, while an increase in recoverability of test specimens was observed.

Mark (80) indicated that elasticity and tenacity are functionally related to the amount of crystalline material, while swelling, drying and chemical reactions are to be associated with the amorphous parts. From the theoretical point of view, the crystallite forms the firm reinforcing parts of the structure, whereas the amorphous regions are the actual points of weakness (80, 100).

The change of crystallinity when cellulosic fibers are stretched was also a subject for investigation. This was based on the hypothesis that cellulosic fibers undergo a process during stretching similar to that experienced with natural rubber samples. Cellulose suffers a partial

conversion of the amorphous into the crystallized state. The only difference is that this change is not as reversible as with rubber and very much less pronounced (80), as was observed by Sisson (130).

Berkley and Kerr (8) investigated the structure of undried, fresh cotton fiber with X-rays, using photographic methods. X-ray patterns taken for these fibers showed little or no evidence of crystallinity, whereas upon drying the usual diffraction diagram of cellulose appeared. If, before drying, the cotton fibers had also been stretched in the wet state, a faint cellulose diffraction diagram appeared. The diffraction diagram remained present even after a dried bundle of cotton was rewetted. The authors interpreted these findings to indicate that undried cotton fiber, of the ages which they studied, occurs only with an amorphous structure. Their model of undried cotton fiber contained sets of long-chain cellulose molecules which, upon drying, undergo internal rotation, bringing adjacent hydroxyl groups into close and partially unseparable proximity. The chains then unite at intervals to form a permanent crystalline structure, leaving unordered material between the crystalline domains.

Preston (116) did not consider Berkley and Kerr's results as final, since the absence of an X-ray diagram does not necessarily imply the absence of crystalline regions. These regions may be small in size and, in addition, the

cellulose diagram of the fresh material may be masked by the water halos. But it still remained to be explained why, in the rewetted fiber, the water does not mask the cellulose pattern.

Quantitative X-ray diffraction methods have been used by Ingersoll (57) to study the lateral ordering and the orientation of the cellulose chains around the long chain axes in various commercial and experimental rayons. His results showed that breaking length increased with lateral disordering of the cellulose chains when the other factor was constant. He supports the concept that local chain disordering in any linear polymer favors higher extensibility. In addition, he stated that it is important to make correction for the difference in orientation, if samples differing widely in this factor are to be compared with regard to their lateral order. This last concept is also supported by Ward (143), who noted the difficulty in obtaining a series of fibers which differ only in their percentage of crystalline material.

The crystallinity of cellulose in the undried cotton fiber, fresh from the unopened but mature boll, was studied by Heyn (45) using X-ray diffractometer methods. It was compared with the crystallinity of portions of fibers from the same samples, either dried directly or from which water had been removed by various indirect ways. He found that stretching of the fiber in water before drying resulted in

slightly higher diffraction peaks in the dried samples as compared to unstretched samples, regardless of what the subsequent preparation had been. It was also estimated by Nickerson (100) that a normal coagulated viscose filament had about 40 per cent crystalline and 60 per cent amorphous material, whereas stretched filaments of the same material contained about 70 per cent crystalline and 30 per cent amorphous material. This indicates the effect of stretching on crystallinity; however, Patil et al. (107) indicated that stretching of swollen fibers "has next to no effect on crystallinity or crystallite size."

Until a few years ago, the question of crystallinity as a factor influencing the mechanical behavior of wood was ignored. This was probably due to the difficulties involved in developing a technique suitable for determining crystallinity in wood and also the variability inherent in wood structure. Kouris et al. (71) examined the effect of various modes of drying upon crystallinity of softwoods, using X-ray diffraction. They did not observe any change in crystallinity index when previously undried, pure cellulose fibers were dried from water under a wide range of conditions. On the other hand, they obtained lower values when the fibers were dried from benzene.

Crystallinity of earlywood and latewood of a longleaf pine holocellulose pulp was determined by Jentzen (60) as fibers were dried under various axial tensile loads.

Although he observed an increase in Young's modulus, tensile strength, work to rupture, and crystallite orientation, he found a decrease in ultimate elongation of individual fibers, and no significant change in crystallinity. A similar study carried out by Hill (47) on the same species supported Jentzen's finding. It should be pointed out that these results are not in agreement with the observed change in crystallinity of cotton and regenerated cellulose fibers following stretching (8, 45, 57).

The first study relating cell wall crystallinity directly to mechanical behavior of wood was conducted by Murphey (92). According to him, the crystallinity of samples taken from yellow birch (Betula alleghaniensis Britton) and sugar maple (Acer saccharum Marsh.) increased at a decreasing rate as applied load increased. Although the crystallinity was constant over a 24 hour period, it did not return to the original level after removal of the applied load. Thus, upon application of tensile stress, some increase in molecular lattice perfection appears to take place causing an increase in crystallinity. Part of this observed increase is retained after removal of the applied stress. Murphey compared his results with those of published rheological studies of strain versus the time elapsed after loading or unloading. He concluded that elastic strain as measured by X-ray diffraction remained unchanged over a 24 hour

period, whereas a continued change in strain occurred during the same period. This indicated to him that continued deformation under load, after initial strain, is viscous in nature. Furthermore, the observed immediate recovery upon removal of the load was an elastic response. Murphey interpreted the residual deformation to be due to the bonds formed through tensile loading which thus restrict the return of the molecules to their non-stressed condition. Recently, Ziegler (161) supported Murphey's results using Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) and noted that tensile loads resulted in increasing crystallinity and deformation. The actual extent of increased crystallinity was not published.

The only criticism the writer has of Murphey's work is the uncertainty of crystallinity determination with the type of specimen he used. He utilized samples cut on the microtome at 45° from the radial direction. These samples have, of course, preferred orientation, whereas a randomly oriented specimen is recommended for X-ray diffraction work. Therefore, his values are not considered to be precise.

3. Variation in Wood and Tracheids

A few studies have been carried out by various workers on variation of crystallinity in wood and tracheids. Preston et al. (120) compared the crystallinity of Cross and Bevan cellulose taken from radiata pine (Pinus radiata D. Don)

of different ages. Their results revealed that crystallinity decreased from pith to bark. Lee (73) studied the same relationship, using two pulps prepared from western hemlock (Tsuga heterophylla (Raf.) Sarg.) by peracetic acid and chlorite method, but found that crystallinity increased significantly from pith to about 15 increments from pith, after which it became almost constant. He further indicated that crystallinity of delignified tracheid skeletons from latewood was significantly higher than that from earlywood. Similar results were also obtained by Taniguchi (136), who found that the total crystallinity content of Akamatsu (Pinus densiflora Sieb. et Zucc.), as determined by an acid hydrolysis method, increased slowly with age in the early stage of growth.

An X-ray diffraction study was conducted by Holzer and Lewis (53) on Douglas-fir earlywood and latewood tracheid skeletons. The latter exhibited an unusually high degree of preferred orientation among the crystallites, whereas the fibers of the former were found to be much more amorphous. Unfortunately, these workers did not give numerical values. Lindgren (77) has also pointed out that Swedish spruce (Picea excelsa Link.) latewood gave a sharper diagram, which indicated a higher degree of crystalline order than the corresponding earlywood. In addition, delignified longleaf pine latewood tracheids exhibit greater crystallinity than earlywood tracheids (60).

The microscopic and submicroscopic structure of tension wood has been investigated thoroughly by Wardrop (148), Wardrop and Dadswell (150, 151), using several Australian species. It is reported that degree of crystallinity in tension wood is higher than in normal wood (150, 151). These workers attributed the difference to the existence of the inner thick gelatinous layer which contains a highly crystalline cellulose, provided that the paracrystalline (amorphous) phase is similar in both normal and tension wood. Lee (73) found similar results for his black cottonwood (Populus trichocarpa Torr, and Gray) pulps. In contrast, crystallinity of Douglas fir compression wood was considerably lower than that of normal wood (73).

D. Microfibril Angle

1. Definition

There is general agreement among research workers that microfibrils in cell wall layers are oriented at an angle to the cell axis. Since the S2 layer constitutes the bulk of the cell wall, the composite microfibril angle is usually referred to as that which the microfibrils in the S2 layer make with the cell axis. Several terms are used to describe this kind of orientation; for example, Wardrop and Preston (155) used the term micellar angle, which has been criticized by Mark (81) because the orientation forms a

helix, not a spiral and the term micelle implies an 'archaic' concept. X-ray angle is also used by Weiss et al. (157) to describe the orientation of the microfibrils about the cell axis. This angle is determined by X-ray technique, which is considered by Mark (81) not to be a very accurate method for measurement of microfibril angle, probably because the X-ray diffraction diagram has to be interpreted before the angle can be determined. The term convolution angle is utilized by Betrabet and Iyengar (9) as an alternative for microfibril orientation. Recently, Mark (81) applied the standard industrial term 'filament winding angle' to describe the helical organization of the microfibrils with respect to the tracheid axis. Even Mark's term has been misinterpreted to mean that the fiber wall is built up from the inside out.

Microfibril angle is the most common term for describing microfibril organization. It is used by Cave (13), Meylan (87), Meylan and Probine (88), Page (104) and many others. It has also been shown by Hearle (37), Preston (116) and Preston and Astbury (119), that the average crystallite angle of inclination in a cell wall layer of valonia, ramie, bamboo, cotton, sisal and conifer tracheids, corresponds to the mean microfibrillar orientation in the same layer. Consequently these two expressions are used interchangeably in the literature.

2. Relation to Mechanical Properties

Microfibril angle of the S2 layer has been shown to be one of the most important supermolecular factors in evaluating the strength properties of wood and other cellulosic fibers. Microfibrillar arrangement of the S2 layer has been correlated with elongation, modulus of elasticity, tenacity and resilience of cotton fibers. Tripp et al. (141), for example, observed the relatively high elongation of cotton fiber concurrent with the application of axial stress which they attributed to the spiral arrangement of the fibrils. An increase in Young's modulus and resilience, i.e., the ability of a cotton fiber to absorb work without suffering permanent set, were also reported by them. Helical structure was also correlated significantly to the Pressley strength index (lb per mg) (44), shear strength (25) and to Young's modulus as well as tenacity (the number of unit lengths of the textile material which can be supported before rupture) of a cotton bundle (123, 157). Similarly, an inverse relationship between convolution angle of 67 cotton varieties and their respective strength properties has been also obtained by Betrabet and Iyengar (9).

Change of microfibrillar organization by stretching under tensile load was also studied. In 1946, Berkley and Kerr (8) found that undried cotton fibers exhibited considerable plasticity and, when stretched, they increased

in length and decreased approximately 25 per cent in diameter. At the same time, the cellulose spiral in the secondary wall approached the parallel position. Recently, Orr et al. (103) recorded the untwisting of the helical structure as load was applied to cotton fibers. Much of this untwisting was reversible. They indicated that friction between growth layers and between fibrils was a possible cause of permanent set, low intrinsic strength of high-angle cottons and weakness near the reversals (see Fig. 8). A positive relationship between fibril angle of cotton fibers and permanent set has also been found by Rebenfeld (123).

Mechanical properties of sisal fibers have been investigated in relation to their helix angle. Spark et al. (131) developed a micro-extensometer for the automatic recording of load-extension curves of single sisal fibers. Using this instrument he found that fibers with flatter spirals had a higher extensibility, lower Young's modulus and lower breaking strength than had fibers with steeper helix. Preston (117) has also indicated similar results with sisal fibers. In addition, he reported that during the stretching of wet fibers, the helix angle changed in such a way that the helix became steeper just as would a spiral spring. He observed 20 per cent extension to break for a fibrillar angle of about 50°. A great deal of this extension was recoverable in water. Therefore, he antici-

pated a slip between microfibrils to accommodate this large amount of extensibility. Further, Preston (117) has shown that the recovery was possible due to release of strain both in the microfibrils and in the incrusting substance between microfibrils. Assuming good stress communication between the framework (cellulose) and surrounding material (lignin), validity of such an assumption is supported by the work of Chow (13a).

It is reported that the highest strength and Young's modulus, and the lowest breaking extension of native cellulose fibers such as flax, hemp and ramie, and regenerated fibers such as viscose rayon and acetate, coincide with the closest approach to axial orientation of crystallites (20, 41, 57, 84, 85, 129). Intrinsic tenacities (tenacities of zero span length) of ramie, flax, and cotton fibers were shown by DeLuca (20) to be related to the 50 per cent intensity angle of (002) arc. This relationship was independent of their actual DP within the 1,800 to 5,000 DP range investigated.

Ingersoll (57) has indicated a substantial linear tenacity increase with increasing orientation (small angle) whereas elongation decreased hyperbolically. This increase in tenacity with orientation has been explained quantitatively on the basis that highly oriented materials have more crystallites parallel to the fiber axis and hence are in a

position to support load most effectively. In a similar manner, Meredith (84, 85) explained the reason for the lower tensile strength obtained from the poorly oriented cellulose fibers. In these, only a fraction of the chain molecules, those which lie parallel to the fiber axis, are in a position to bear tension stresses whereby they are first to rupture, and the rupture of the others follows directly.

In the case of solid wood specimens, wood tissues and delignified tracheid skeletons tested in tension, analogous high correlation of microfibril angle to strength properties is evident. In 1939, Garland (32) found that 'strength-density' and 'stiffness-density' of shortleaf pine (Pinus echinata Mill.) and loblolly pine (Pinus taeda L.) latewoods were inversely correlated to the sine of the microfibril angle. The breaking load in tension for radiata pine and Douglas-fir fibers, taken from successive growth increments, was stated to increase with increasing orientation (144). Wardrop (144) has also indicated that the longer tracheids, with more cellulose chains favorably oriented parallel to the cell axis, can take a higher applied load in tension.

In a study by Fujisaki (31), an indirect relationship was found between inclination of 'micelles' in the cell wall and modulus of elasticity. This relationship stemmed from the observed increase in modulus of elasticity from pith to bark of sugi (Cryptomeria japonica D. Don.) and the decreased micellar angle in the same cardinal bole direction.

Using the same species, Suzuki (135) obtained a close correlation between the ultimate strain and the micellar angle.

Ifju and Kennedy (55), utilizing Douglas-fir, and Wellwood (158) using Douglas-fir and western hemlock, concluded that micro-tensile strength is related to fibril angle. When earlywood results were analyzed separately from latewood, the tensile strength of the latter only was shown to be highly correlated to fibril angle (158). Cowdrey and Preston (17, 18) have indicated that Young's modulus of Sitka spruce (Picea sitchensis (Bong.) Carr.) increased rapidly across the annual increments from pith to bark. Tracheid length also increased and the helical angle decreased along the same radius. Kellogg and Ifju (62) found that the ratio of longitudinal to transverse thermal conductivity of 20 hardwood and softwood species was the only factor influencing specific strength and specific stiffness. Since all cell wall layers and types would contribute to their thermal conductivity ratios, the authors suggested that this ratio might be an evaluator to what they termed "integral fibril angle'.

Microfibril angle has been related to the rheological properties of wood. Senft (128) attempted to examine the effect of creep-inducing loads, in compression, on ultrastructural anatomy of Sitka spruce but was unable to measure the expected shift in microfibril angle. He attributed this,

however, to the relaxation which presumably had occurred during microfibril angle measurement, and also to the observed high variability of the microfibril angle in the secondary cell wall layers of his samples. Hill (47) and Jentzen (60) have shown, by X-ray analysis, that crystallite orientations of longleaf pine holocellulose tracheid skeletons from earlywood and latewood tissues became steeper as the fibers were dried under various axial tensile loads. The theoretical analysis of extension led Jentzen to conclude that the amount of extension should be relatively independent of the drying load, but related to the initial fibril angle. His experimental results supported these theoretical reasonings.

Frey-Wyssling (29) has speculated that rheological properties are not dependent on the fibril angle, but rather on the relatively poor cohesion between neighbouring microfibrils. He postulated that the microfibrils are 'cemented' together in such a way that they cannot elongate individually unless their reciprocal cohesion is broken. Therefore, he suggested that the magnitude of extensibility and degree of elasticity (elastic portion of the work done as a percentage of the total work) must depend on the forces which hold the microfibrils together in the cell wall. However, the above reported work by Hill (47) and Jentzen (60) on individual tracheids does not support Frey-Wyssling's concept but rather emphasizes the microfibril angle as an important factor responsible for creep.

Based on such strong experimental relationships between mean microfibril angle and elasticity, as well as strength, theoretical models have been suggested to explain the mechanism of deformation in plant fibers (17, 18, 38). Hearle (38), who has proposed a fringed fibril structure for native and regenerated cellulosic fibers, has shown that the deformation of plant fibers can be explained as a combination of two processes: (a) stretching of the fibrils without a volume change and (b) stretching of the fibrils as a spring at constant fibril length, followed by a reduction in volume. According to Hearle's model each fiber is a solid cylinder and the radii of microfibrils are taken to be of the order of 100 Å. This model has been criticized by Cowdrey and Preston (17, 18) and Mark (81), the latter having stated that if larger mechanically effective radii were assumed according to the fringed fibrillar structure, spring-like deformations should not be excluded.

Cowdrey and Preston (17, 18) have suggested two approaches to the deformation mechanism as an alternative to the model of Hearle. The first states that the microfibrils extend like helical springs by bending, twisting and slipping over each other and not by the stretching of individual fibrils which might be either individual microfibrils or aggregates of microfibrils. They had to assume that the microfibril aggregation is 5 μ in radius and that the microfibrillar substance is isotropic in order

to bring experiment and theory into mutual proximity. The second approach is based on the stretching of microfibrils in Hearle's analysis and considers that the cell wall is a single phase system in which the wall substance is regarded as an anisotropic homogeneous medium. According to this analysis, the correlation between the initial compliance and the helical angle takes the form of a quadratic equation in $\sin^2\theta$. Mark (81) considers their first approach not to be an unrealistic concept; and Jentzen's results noted above fit a spring model of inextensible material and of fixed total length.

Similar work has been conducted by Balashov et al. (6). Using X-ray diagrams, they determined the change in the spiral angle as a fiber bundle of sisal was stretched. The extension of the fibers and the change in spiral angle were related to each other experimentally in the linear manner than would be predicted for an extended ideal spring model. The authors were also able to calculate the slip between microfibrils and the change in distance between adjacent microfibrils, in the same cylindrical sheet and between cylindrical sheets, as a fiber underwent a maximum strain of 3 to 5 per cent. Therefore, they concluded that rheological properties of natural fibers are greatly dependent on the microfibrillar organization of the cell wall.

3. Variation in Wood and Tracheids

There has been increasing evidence in the literature that microfibril angle exhibits a high degree of variability between species, within the same species and from pith to bark in the same tree (48, 51, 111). It also differs within annual increments, with a steeper angle in latewood than in earlywood (5, 14, 50, 111, 159). In addition, compression wood is characterized by a flatter angle than that of normal wood.

At the micro level, not only is there variation of microfibril angle from layer to layer in the cell wall, but also within a given layer there may be a difference within the radial and tangential wall (81). Maby (78), Preston (115) and Preston and Wardrop (121) have shown that microfibril angle in the secondary wall is related to cell breadth. Hiller (49) found a similar relationship for latewood of slash pine (Pinus elliottii Engelm.) and of loblolly pine.

It was shown some time ago by Preston (115, 118) and Wardrop and Preston (155) that the microfibril angle (θ) in the S2 layer and the S1 layer is related to cell length (L) by the following equation:

$$L = a + b \cot \theta$$

where: a, b = constants.

Hence, this equation agrees with the general observation

that the microfibril angle in the longer latewood tracheids approaches the axial direction more closely than in shorter earlywood tracheids.

E. Extractives Content

In addition to its major components, cellulose, hemicelluloses and lignin, wood also contains small but in some cases quite appreciable quantities of extraneous components. Many of these substances are extractable with neutral solvents, and are referred to as extractives (12). Wood extractives include a wide variety of organic compounds (mainly polyphenols) of low and high molecular weight.

Distribution of extractives in wood is not well established. They may be infiltrated completely into the amorphous region of the cell walls or they may be deposited mainly in the parenchymatous cell lumen (105). On a macro-level, large differences are reported between species, with age from pith, relative maturity of trees, height in stem, between heartwood and sapwood, and earlywood and latewood (132). Squire (132) has shown that the polyphenolic compounds of Douglas-fir are confined to wood ray tissue. It was shown by Tarkow and Krueger (137) that approximately 75 per cent of the total water soluble extractives of redwood (Sequoia sempervirens (D. Don) endl.) are located in the cell walls. The recent work by Morgen and Orsler (90) has also indicated that appreciable amounts

of extractives may be located in the cell wall.

Although the extractives constitute only a few per cent of the oven-dry weight of wood, their influence on mechanical properties may be considerable. In this respect, knowledge of extractives location in cells should help in understanding their influence on mechanical properties of wood. Brown et al. (11) speculated that the effect of extractives' removal from wood would be greatest on crushing strength parallel to the grain, least on shock resistance. They attributed the difference in behavior to the fact that the former depends on the cumulative resistance of wood and 'infiltration' present in it, whereas the latter is contingent for the most part on the cohesive and adhesive forces acting in the cell wall substance only, forces which are not altered significantly by removal of extractives.

Recently, Arganbright (2) studied the influence of extractives on bending strength of redwood. He found that the green and air-dry modulus of rupture (MOR) was unrelated to the amount of extractives, whereas modulus of elasticity (MOE) decreased with increasing amount of extractives. The author was not able to offer a precise explanation for the observed relationship between extractives and MOE.

Further, on the role of wood extractives on rheological properties of wood, Narayanamurti (94) and co-authors (95, 96) have indicated that removal of extractives caused a

decrease in the rigidity modulus and an increase in the elastic modulus of some hard- and softwood species, one of which was Picea morinda Link. The authors have also found that extractives affected rheological properties of wood at high temperature suggesting that these substances have a plasticizing effect.

Erickson and Sauer (24), following a similar approach, have recently conducted a flexural creep experiment under two drying conditions. According to their results, relative creep, defined as creep deflection divided by the deflection at one minute, was positively correlated with extractives content of redwood boards. The correlation values (r) were given as 0.77 and 0.76 for drying conditions of 106°F and 150°F, respectively. Elimination of one specimen, which developed a collapse streak during the test at 150°F, improved the correlation between relative creep and extractives content ($r = 0.95$).

F. Specific Gravity

Specific gravity is usually defined as a macroscopic measure of the amount of cell wall substance contained in a unit volume. It varies between and within species; radially and axially in the same tree; between earlywood and latewood; and across the growth increment (22). These variations are due to differences in the size of the cells, the thickness of cell wall, and the relative volume of the S2 layer (24, 60, 64, 105).

Strength properties of wood are affected by its specific gravity. Based on extensive tests carried out on many kinds of wood over a wide range of specific gravities, Newlin and Wilson (99) several decades ago established that the mathematical relationship existing between specific gravity and various strength properties can be expressed as a parabolic equation of the nth degree, as follows:

$$S = a\,G^{n}$$

where:

S = any one of the strength properties,

a = constant,

n = constant varying from 1.00 to 2.25 depending on the particular strength property, and

G = specific gravity

Several authors, among them Homoky (54), Ifju and Kennedy (55), Kellogg and Ifju (61), Suzuki (135), Wellwood (158), and Wellwood *et al.* (159) have found strong, positive relationships between specific gravity and the various strength properties of wood. Brown *et al.* (11) have stated that this relationship requires no explanation since the load that a wood specimen will bear is determined to a great extent by the amount of cell-wall substance it contains per unit of volume. However, two anomalies have been reported by Hearmon (39) and Mark (81) which indicate

the lack of correspondence between the elastic constants (rigidity and Young's moduli) and densities of many species.

Little has been reported concerning the effect of specific gravity on creep behavior of wood. In an attempt to account for species differences in creep, Kellogg (61) found a negligable correlation between the species residual (an expression of the differences in amount of creep between the creep curve of each species and the average creep curve for all 9 species studied) and specific gravity. In contrast to Kellogg's results, King (65) has concluded that specific gravity and maximum tensile strain can account for a high percentage of the differences between species with regard to the creep response in and below the region of the threshold of set. He found that species having low specific gravity and high extensibility creep more than those with a high specific gravity but low extensibility.

Erickson and Sauer (24), in the work referred to above, reported a significant inverse relationship between relative creep and specific gravity at low drying temperature (106°F). The same relationship was marked by the combined effect of initial moisture and extractives content at high drying temperature (150°F). The authors had not expected any relationship at the low temperature level since they had standardized the specific gravity effect by expressing creep deflection relative to the initial deflection. They attributed

the obtained relationship to a higher ratio of S2 layer to total cell wall substance as specific gravity increased, and not to the weight contribution of extractives to specific gravity. Using plywood panels, made up from one softwood and six hardwood species, Higgins (46) found that the residual deformation in compression perpendicular to the grain varied from one species to another depending on the density of wood rather than the gross anatomical structure.

III. Creep Phenomenon

Wood is commonly considered to behave elastically, i.e., it obeys Hooke's law, at stresses below the elastic limit. However, it has been indicated that wood also possesses properties dependent on time which are not fully explainable by employing the Hookean elastic theory alone (47, 60, 61, 65, 68, 92, 108, 109, 128). If a load is applied to a wood-fiber or specimen at zero-time, there is an instantaneous elastic deformation (OA, Fig. 9) followed by a continuing deformation (AB) over a period of time (T_0-T_1). This continuing deformation at constant stress is called creep. If the stress is removed at time T_1, an instantaneous elastic recovery (BC_1) takes place, then a retarded partial creep recovery (C_1 to D) occurs at time T_2. The part (DE) of the graph represents the permanent deformation remaining at the end of the loading-unloading cycle.

Many investigators have used rheological models in an attempt to obtain a method by which the reaction of wood to an applied stress, over a long period of time, may be described phenomenologically. These models are composed of springs which deform under stress in a Hookean manner, and dashpots which represent the viscous flow of a Newtonian liquid. They are characterized by linear viscoelastic behavior because the springs are assumed to be Hookean and dashpots have flow rates which are Newtonian.

A number of models, using various combinations of springs and dashpots, have been reported (1). The most common and more satisfactory model used for wood and some high polymer solids, is the four-element model shown in Fig. 10 (70, 108). This model combines a Maxwell body (a spring and dashpot in series) with a Kelvin body (a spring and dashpot in parallel). If the model of Fig. 10 is loaded over a period of time there is an instantaneous elastic deformation represented by the spring constant K_1. This is attributed to an elastic extension of the microfibrils (38) or to their bending, twisting and slipping (17, 18).

The movement of the Kelvin body, represented by the spring constant K_2 and damping coefficient R_2, results in a retarded elastic response, i.e., primary creep (Fig.11). Hearle (38) has indicated that this primary creep is caused by the viscoelastic compression of the non-crystalline

material, and also by the increase in length of the non-crystalline matrix. It is also due to the adjustment between microfibrils in the non-crystalline portion of the cell wall. Alfrey (1) claimed that the observed primary creep is related to the uncurling of molecular chains. The R_3 term (Fig. 10) represents a flow mechanism which is usually associated with the later stages of the secondary creep, and the tertiary creep. This element is said to be responsible for the permanent deformation.

Upon removal of the load, it is assumed that there is an instantaneous recovery of the K_1 spring. The K_2 spring and R_2 dashpot return slowly almost to their original position. Finally, the R_3 dashpot may or may not change its position, depending on the treatment given to the specimen. Even this widely accepted four-element model has been criticized by Pentoney (108) because of its inability to describe the time-dependent behavior over a wide time scale. He then stated that a distribution of retardation times might be proposed. This distribution is assumed to be composed of discrete retardation times through the addition of any number of Kelvin bodies in series to the four element model.

A. Effect of Wood Species

Little has been reported as to differences in creep response among species. Creep behavior of 11 species (9 hardwoods and two softwoods, one of which was Douglas-fir) was compared by King (65) on an initial strain basis and also

on a stress level basis. He found substantial differences between species with respect to the magnitude of creep response, and he attributed a large percentage of this difference, in and below the threshold of set, to specific gravity and maximum strain. Similarly, Kellogg (61) compared the creep response of 9 species, three of which were the same as King's, at the same initial strain. He attempted to account for the variation between species by plotting the average residual from the general curve for each species against parameters such as specific gravity, slope of grain, ultimate strain, modulus of elasticity, and the ratio of modulus of elasticity to specific gravity, which he assumed to represent a measure of fibrillar orientation or crystallinity. The author concluded that the best parameter, which accounted for only 34.6 per cent of the total variation between individual species, was the ratio of modulus of elasticity to specific gravity.

Using individual longleaf pine holocellulose tracheid skeletons from a single growth increment, Jentzen (60) has indicated that those of earlywood underwent much greater elongations than those of latewood under the same applied drying load. He related these elongations to the reduction in the fiber diameter and to the initial fibril orientation.

Contrary to the aforementioned variability between species with regard to creep response, Kingston (66) and Kingston and Clarke (67) reported that mountain ash

(Eucalyptus regnans F.V.M.) and hoop pine (Araucaria cunninghamii Sweet) beams did not, in general, show widely different creep behavior in bending. Unfortunately, the basis on which these species behaved similarly was not given by the authors.

B. Relation to Moisture Content and Temperature

Creep response depends, to a great extent, on the environmental conditions under which the tests are carried out. Thereby moisture content and temperature are considered to be extremely important factors. Their effect was eliminated in this study by carrying out the creep tests under constant temperature (73 ± 3.5°F) and relative humidity (50 ± 2 per cent). The reader is referred to the recent excellent report by Schniewind (125) concerning the effect of temperature and moisture content on the rheological properties of wood.

MATERIALS AND METHODS

I. Wood Samples

The experimental material was carefully chosen from single trees of three western Canadian coniferous species, namely Douglas-fir normal and compression wood, Sitka spruce, and western hemlock, growing on a good site (site index of 120) at the University of British Columbia Research Forest, Haney, B.C. The most important characteristics taken into consideration in the selection of this material were straightness of stem, fairly large diameter,and lack of leaning of the tree except for the one including compression wood. Based on these characteristics, one tree was chosen from each of the abovenoted species in addition to the tree which showed compression wood on one side. The diameters at breast height were 26, 22, 28 and 14 in. outside bark for Douglas-fir normal and compression wood, Sitka spruce and western hemlock, respectively. As determined later by laboratory tests, wood from these four trees furnished a wide range of microfibril angle, specific gravity, crystallinity per cent and extractives content.

After felling the trees one disc was chosen from each at breast height. In order to prevent drying below the fiber saturation point, discs were wrapped in polyethylene sheets and transported to the Faculty of Forestry, where

they were stored in a cold storage room at 35 ± 1°F.

Three adjacent tangential blocks with straight-grained wood were cut from each disc (Fig.12). Each of these tangential blocks included at least one annual increment which exhibited a minimum of curvature. The dimensions of each block were a nominal 2.5 in. longitudinally and 1.5 in. radially whereas width (tangentially) was controlled by curvature of the increments. The selected blocks were aspirated under vacuum in a vacuum/pressure cylinder until waterlogged, prior to sectioning on a sliding microtome.

II. Preparation of Test Specimens

Primary efforts were made to carry out all measurements for the selected features, namely microfibril angle, relative degree of crystallinity, extractives content, and specific gravity, on the same specimens as used for the creep test. But due to physical limitations, such as amount of material required for X-ray diffraction and the necessity of a split surface along the grain for microfibril angle measurement, an appropriate sampling method for securing matched specimens was devised and is described below.

Preliminary experiments have shown that microfibril angle and crystallinity did not differ significantly among three tangentially adjacent blocks for any one disc. Therefore, this number of blocks was chosen to represent each

species. One block was used to provide material for the 3,000 microin.[3] per in. initial strain level, the second for the 6,000 microin. per in. initial strain level, while the third block was used for obtaining the required amount of material for crystallinity determination.

Prior to sectioning tangentially on a sliding microtome, each block was glued to a piece of plywood to ensure the required section quality and to avoid stresses originating from the microtome grips. It has been shown by Kennedy and Chan (63) that slicing angle, i.e., the angle between grain direction and the travel direction of the microtome knife is critical if development of many slip planes is to be avoided. Consequently, a slicing angle of 10° with the grain direction parallel to the knife edge was used.

The increments previous to the selected ones (Numbers 71, 187, 60, and 80 for Douglas-fir normal wood, Sitka spruce, western hemlock and Douglas-fir compression wood, respectively) in each block were sectioned and discarded. At the same time adjustments were made to ensure parallel knife and wood block alignment. Once the tangential surface at the chosen position in either earlywood or latewood region had been satisfactorily prepared, two contiguous microsections were

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[3] One microin. = 10^{-6} in.

taken from each block. One was used for obtaining strips for creep experiments while the other was kept for microfibril angle measurement. Special care was taken to obtain nearly the same section thickness from both earlywood and latewood zones. The thickness ranged from 0.009-0.011 in. as determined by use of a TMI Model 549 micrometer.

Punching out micro-creep test specimens was carried out using the specially machined cutting die fixed to a half-ton arbor press (Fig. 13). It has been indicated by Ifju et al. (56) that rectangular specimens have certain advantages as far as stress behavior is concerned. Accordingly, rectangular specimens with 0.098 in. width and 2.5 in. length were used in this study. Care was taken to ensure a cut parallel to the grain, either by tearing a strip near the edge of the section, or by noting the direction of ink flow along the grain. The test specimens (four to six) obtained were then transferred to the controlled temperature and humidity (CTH) room maintained at 73 ± 3.5°F and 50 ± 2 per cent relative humidity, where they were placed between layers of absorbent paper to prevent curling. At the end of a storage period of eight days no change in specimen weight could be observed to an accuracy of ±0.0005 g. The specimens were then checked for dimensions with the micrometer.

III. Micro-creep Test

A. Testing Machine

A table model Instron testing machine (TM-M-L) was modified for carrying out creep tests in tension parallel to the grain. A strain gauge extensometer with 0.5 in. gauge length and 10 per cent strain limit was connected to a five-pin adapter feeding directly to the load cell amplifier. A record of the strain behavior was automatically recorded by the Instron strip chart recorder. This instrument is on the left hand side of the tensile testing machine (Fig. 14A). Calibration of the chart was carried out using an extensometer calibrator. With the extensometer in place, the calibrator was extended a known amount and corresponding chart motion was adjusted to the desired magnification. It was also possible to increase the sensitivity by using a lower range, namely Range One on the Instron panel, which provided a sensitivity of ±5 microin. per in. To achieve this level of sensitivity, it was necessary to unbalance the strain gauge by a known amount of strain. The zero strain point was suppressed off the scale of the chart by either 2,600 or 5,600 microin. per in. The procedure followed was that indicated in the Operating Instructions manual of the testing machine.

The chart was also calibrated electronically using the coarse balance. One notch clockwise of the balance was equal to 1.05 per cent strain on the 10X range. Therefore, it was convenient to check the calibration before and after each test by simply turning the coarse balance one notch clockwise.

B. Loading System

Test specimens were glued at both ends between two aluminum sheets, using Eastman 910 adhesive. This was done to facilitate loading the specimens. The assembly was carefully aligned in the upper grip of the Instron testing machine. A hooked end wire was put through a hole in the bottom of a bucket, used for loading the assembly, the bucket resting on two supports on the lower crosshead of the machine. A 100-gram weight was hung at the lower end of the wire to straighten out the specimen and also to facilitate mounting the strain gauge extensometer on it. The gauge was made almost weightless by hanging it from the fixed upper crosshead of the Instron. Some lead shot, depending on the tensile strength of each species, was then put into the bucket without loading the specimen. After about 15 minutes, during which the zero strain point was suppressed to either 2,600 or 5,600 microin. per in., the lower crosshead was automatically moved down. More lead shot was immediately poured into the

bucket, so that the initial required strain value of either 3,000 or 6,000 microin. per in. was reached in about 30 seconds. This system provided an accurate and rapid means of loading the assembly. The loading system is shown in Fig. 14B.

It should be indicated that the number of replications from earlywood and latewood used in the statistical analyses is not the same for each group of samples due to the following: (a) unavoidable curvature of some of the annual increments; (b) specimen rupture during conduct of creep test at the higher strain level and (c) the inability to apply the required initial strain level to some test specimens.

Measurements of total creep were taken over a 60-minute period of time at a chart speed of 0.2 in. per minute. The Instron testing machine was carefully checked for drift every fourth test. Two examples of test records are shown in Figures 15 and 16.

C. Selection of a Constant Initial Strain Level

Exploratory tests had shown that loading four to five test specimens, taken from the same section of wood (replications), to a specific percentage of the ultimate tensile strength, resulted in a high variability in total creep response. However, loading similar specimens to a constant initial strain indicated a high degree of similarity among

them in total creep values. This suggested that, in a case where an explanation for the differences in total creep is required, one should fix the elastic portion (initial strain) of the creep behavior.

Previous work done by Kellogg (61) had also indicated a similar trend. He proposed that strains measured at the gross level remain comparable at the molecular level. Therefore, he considered this single factor as the best indication of an equivalent stress condition at the molecular level. Two initial strain levels,[3a] namely 3,000 and 6,000 microin. per in., were chosen to be applied for all specimens used. Selection of these strain levels was based on the work done by King (65) who had found that the threshold of set occurred at almost 4,000 microin. per in. initial strain for all specimens of softwoods and hardwoods tested. He also indicated, by his graphs, that the variability among species below the threshold of set was less than that above it. Therefore, selection of the aforementioned strain levels was useful in obtaining the creep behavior below and above one of the critical regions, i.e., threshold of set.

IV. Micro-specific Gravity

After carrying out creep tests, specimen thickness, width and length was measured to the nearest 0.001 in. These specimens were used for micro-specific gravity determinations.

[3a] The equivalent stress levels are filed in Room 388, Faculty of Forestry.

The specimens were extracted in sequence with ethyl ether, ethyl alcohol, and hot (about 80°C) distilled water. Eight hours were allowed for ether and alcohol extractions while hot water extractions were carried out for four hours, changing water every hour. This extraction process was necessary in order to base specific gravity calculations on extractive-free cell wall substance.

After extraction, specimens were put in weighing dishes. Each tissue group (earlywood or latewood) was placed in one dish. They were then oven-dried at 100 ± 2°C for three hours. Upon completion of drying, specimens were transferred to a desiccator then immediately to a plexiglass glove box where they were cooled for 30 minutes inside the desiccator.

Weights were determined using a Cahn Electro-balance contained in the plexiglass glove box (Fig. 17). To maintain desiccated atmosphere for oven-dry specimens, freshly reconditioned silica-gel drying medium was used. The box was tightly sealed providing the accurate moisture free atmosphere. Weighing was carried out using the 20 mg range on the balance, which gave a sensitivity of 0.001 mg. Calibrations and zeroing of the balance were conducted after every fifth measurement.

Specific gravity based on volume at test and extractive-free oven-dry weight was obtained by dividing weight in mg by volume in mm^3. This method of determining

specific gravity has been used by other workers (54, 56), and has shown high accuracy and efficiency.

V. Extractives Content

Extractives content was determined on the specific gravity samples in the following manner. The specimens were oven-dried (100 ± 2°C) for three hours prior to extraction and weighed as previously described on the Cahn Electrobalance. Following extraction the oven-dry weight was obtained again. Extractives content was calculated based on extractive-free oven-dry weight as follows:

$$\text{Extractives content} = \frac{W_{od} - W_{ef}}{W_{ef}} \times 100$$

where:

W_{od} = oven-dry weight, and

W_{ef} = extractive-free over-dry weight.

Since most of the volatile extractives have boiling points of over 100°C (12), very little loss would be expected in these constituents during oven-drying.

VI. Microfibril Angle Determination

It is well known that the middle layer (S2) of the cell wall constitutes as much as 80 to 95 per cent of the tracheid wall volume. Therefore, microfibril angle is defined, in this work, as the mean helical angle between the direction of the microfibrils in the S2 layer and the cell axis (88). During the course of the exploratory work several methods for microfibril angle determination were tried including the Bailey and Vestal (5) classical iodine staining method. Difficulties were experienced in obtaining consistent development of iodine crystals parallel to the microfibrils of the S2 layer. Consequently, this technique was rejected as an appropriate method for determining microfibril angle in the species used. Two alternate techniques were chosen as indicated below.

A. X-ray Diffraction Method

The X-ray diffraction technique based on the theoretical model developed by Cave (13) was applied by Meylan (87) for measurement of microfibril angle in radiata pine. The technique involves exposing a 1.5 mm thick specimen to X-rays for two hours to obtain a photographic diagram of the (002) arc. Meylan measured the width of this arc by means of the angular separation of the two tangents at the points of inflection of the intensity curve obtained from a photometer

recording. He drew tangents to the sides of the diagram and defined the width of the arc (2T) as the distance between the points of intersection of these tangents with the zero intensity axis. This technique is relatively time consuming and tedious due to the numerous necessary steps involved in obtaining the intensity curve of the (002) arc.

A new technique was therefore developed which gives the X-ray diffraction pattern directly in 20 minutes with no need of the photometer scanning. A Texture Goniometer machine available in the Department of Metallurgy was used for this purpose. The angle (2θ) was set at 21° and the four to six strips of wood taken from the tangential section used for crystallinity determination were stacked together longitudinally and placed perpendicular to the X-ray beam in the middle of a special disc. This disc was set to rotate from the zero degree position to 360°, giving two peaks for the (002) plane, one at 90° and the other at 270°. The angle 'T' was defined as half the angular distance between the points of intersection of these tangents with the zero intensity axis (Fig. 18).[4]

It should be noted that these 'T' values are affected by the mean microfibril angle and the distribution of microfibrils in the S2 layer of the cell wall (13). Because the

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[4]The remaining diagrams are filed in Room 388, Faculty of Forestry.

'T' value does not give the average microfibril angle directly but has to be calibrated with one of the microscopic methods for measuring the microfibril angle, the method described below was used.

B. Optical Method

Since microfibrils in the S2 layer are arranged helically with the longitudinal axis of the tracheids, the angle on the opposite walls between the microfibrils and the tracheid axis is in the opposite direction. Thus the birefringences of the two opposite walls tend to compensate one another. Under this condition, the tracheid functions approximately as an uniaxial crystal with the maximum reflective index along its axis and independent of the microfibril angle (104). In the case of the thick-walled fiber, the optical theory of the system is complex (114). Therefore, a single wall rather than a double wall is required to determine the microfibril angle. Early in 1934, Preston (112) overcame this difficulty by proposing the method of longitudinal sectioning of macerated fibers. In doing so the opposite walls of the fibers were removed and light was passed only through the remaining wall.

A similar approach was used recently by Page (104), where mercury was introduced into the lumens (radial surfaces) of wood samples or delignified tracheid skeletons. When an impregnated sample is examined in a polarizing microscope

using incident (epi-)illumination, the light passes through the tracheid wall, suffers reflection at the mercury surface and exits through the same wall. Under epi-illumination with the polarizer and analyzer (polars) crossed, a tracheid with a mercury-filled lumen has an extinction position when the fibrils in the S2 layer are parallel to the plane of one of the polars. The microfibril angle is measured as the angle between this plane and the axis of the tracheid in the extinction position. The only prerequisite in the case of wood samples is that the surface to be examined has been split along the middle lamella. Consequently, Page used mild delignification prior to wood splitting.

Page's method with some modifications was used in the present study. The samples of which the tangential surfaces were matched with that used for creep tests, were subjected to a mild delignification (solution contains equal parts of acetic acid and hydrogen peroxide). This facilitated splitting the section surface manually along the middle lamella, as required for obtaining single walls.

The samples were then dehydrated using acetone, following which they were put into a small pressure cylinder. Mercury was introduced and a pressure of 1,000 psi was applied through a piston in the cylinder for a few minutes. The pressure was then released and the specimens were examined in a polarizing microscope using incident (epi-)

illumination to determine microfibril angle. Thirty measurements[5] were taken on each matched section.

VII. Determination of Crystallinity by X-ray Technique

A. Specimen Preparation

A section matched with that used for creep tests was available from the third block, as noted above, for crystallinity determination.

The preliminary experiments during this investigation showed that thickness as well as tracheid orientation are extremely important for cell-wall crystallinity determination. Since two different kinds of tissues, namely earlywood and latewood, are to be used as experimental materials and their specific gravities differ, it was necessary to use the same mass per unit area from both earlywood and latewood. This was done by grinding the material to be used in a Wiley mill and collecting the particles which passed through a 20 mesh screen but were retained on the 40 mesh screen. By using wood meal, the same relative thickness was maintained and fiber orientation was also minimized. Grinding the material to this size would not affect the diffraction pattern (97).

During the preliminary stages of this work it was found that 400 mg and 500 mg of material produced identical diffractograms. Therefore, a standard sample of 400 mg in a 1/2 x 1 1/2 in. rectangular specimen holder was used.

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[5]The original experimental data are filed in Room 388, Faculty of Forestry.

The ground wood was shaped into a thin rectangular pellet (1/2 x 1 1/2 in.) by compression at about 1,000 psi in a specially designed die. As a prior step, a drop of dilute glue solution (10 ml Duco cement plus 100 ml amyl acetate) was placed on 400 mg of ground wood after it had been lightly compacted in the die, then the full specified pressure was applied in one to two minutes. It was indicated by Nelson and Schultz (98) that the amount of cement used would not affect the diffractogram.

B. X-ray Diffraction

A Philips X-ray Diffractometer was used for crystallinity determination. X-rays were generated from a water-cooled copper target passed through a 0.5° divergence slit and then to a nickel filter in order to eliminate the Kβ radiation of the X-ray tube. Test were made at 40 Kv and 15 ma current. The goniometer consisted of a proportional counter with a preamplifier circuit mounted on a moveable arm, which was moved by a constant speed motor and a system of gears. This motor was operated at one degree (2θ) per minute. The sample surface, at the center of the circle surrounded by the goniometer, moved at half the speed of the goniometer.

Diffracted X-rays entering the proportional counter through the 0.1° receiving slit and 0.5° scatter slit were amplified by the preamplifier circuit and fed to a recording panel.

The time constant, which controls the use and fluctuations of the rate meter, was maintained at eight seconds. A scale factor of either 2 x 10^2 or 4 x 10^2, depending on the intensity of the peaks, was used. Chart speed was maintained at 0.39 in. per minute. Test for air scatter indicated that correction for such effect was of a minor order and need not be made.

Samples were mounted on a specially designed aluminum holder, then placed appropriately in the middle of the circle surrounded by the goniometer. It was necessary to make sure that the specimen was at the same level as the holder. To achieve that, an aluminium sheet having the same thickness as that of the specimen was glued to the top of the holder.

Previous work has shown that the diffraction pattern developed over the range of 6 to 30° (2θ) angle is sufficient for crystallinity determination (73, 92). Three peaks were obtained over this range, namely (002) and (101 + $10\bar{1}$) (Fig. 20).[6] The same range was employed in this work and two diffractograms were taken for each specimen except for Sitka spruce latewood where one measurement was taken due to some experimental difficulties which did not allow taking the second measurement.

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[6]The rest of diffractograms are filed in Room 388, Faculty of Forestry.

C. Selection of an Equation for Crystallinity Determination

The per cent of crystalline material in a partially crystalline polymer such as wood can be determined theoretically with high precision by comparing the intensity of X-rays diffracted by the crystalline portion with that intensity diffracted by the non-crystalline portion in the same samples (142). However, many difficulties have been faced by other workers. Assigning values to the crystalline, amorphous, and background scattering, as well as the unsymmetrical peaks, have been among the major difficulties (33, 41). For the purpose of his work, Murphey (92)used the total integrated intensities minus the background (the pattern developed when the specimen was excluded from the apparatus). This included the X-ray diffracted by crystallites, the coherent amorphous scatter, and the incoherent and thermal agitation scatter. The disadvantage of this method is that one is not able to separate the part diffracted by the crystalline region of the cell wall from that of the amorphous region.

Turley (142) has stated the advantage of using the relative per cent crystallinity value because of the difficulties noted above. He drew the background line between two points which were chosen so that all diffraction patterns for the material had minima at these points. Then he

sketched the non-crystalline peak in an arbitrary manner based on a set of minimum points. He calculated the relative per cent crystallinity as the ratio of the area under the crystalline peaks and above the non-crystalline peak to the total area above the background line, multiplied by 100. This method is useful in ranking a series of samples of the same polymer according to their crystallinity. However, it is not an accurate method due to the difficulties in separating crystalline from non-crystalline peaks.

An empirical method for estimating the degree of crystallinity proposed by Segal et al. (126) was chosen to determine the Crystallinity Index (CrI), which expresses the relative degree of crystallinity. The equation used was as follows:

$$CrI = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

where:

I_{002} = the maximum intensity (in arbitrary units) of the (002) lattice diffraction, and

I_{am} = the intensity of diffraction in the same units at $2\theta = 18°$.

This equation was considered the best and was used for the purpose of the present work since the two parameters in the above equation are determined at two fixed points. The

background was designated as the line drawn between two points which were chosen so that all diffraction patterns had minima here (see Fig. 20).

RESULTS AND DISCUSSION

Experimental values for the three coniferous woods, Douglas-fir (normal and compression wood), Sitka spruce, and western hemlock tested at 3,000 microin. per in. initial strain (initial strain No. 1) and at 6,000 microin. per in. initial strain (initial strain No. 2) are summarized in Tables 1, 2 and 3. Four independent variables, microfibril angle (X_1), (Table 1, Column 2) relative degree of crystallinity (X_2) (Table 2) specific gravity (X_3), extractives content (X_4), and the dependent variable, total creep over a period of 60 minutes (Y) (Table 3) are used in the statistical analyses.

I. Statistical Analyses and Interpretation of Results

Multiple regression analysis was employed to find a mathematical function that could be used to describe the relationship which was assumed to exist between total creep and the independent variables studied. The following linear function

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4$$

was found to be the best one to represent the anticipated relationship for both levels of initial strain used.

Accordingly, this function was used for regression analyses to choose the most important variables which should be included in the equation. The general relationship between total creep in all species and the independent variables was found to be as follows:

$$Y_1 = -111.9230 + 13.6294X_1 + 00.9093X_2 + 144.0210X_3 - 11.1135X_4 \quad \ldots\ldots [1]$$

$$SEE^7 = 34.0773$$
$$R^2 = 0.7681^{**}$$
$$N = 34$$

and, $$Y_2 = -1685.2100 + 31.5733X_1 + 20.1994X_2 + 451.2920X_3 + 29.6591X_4 \quad \ldots\ldots [2]$$

$$SEE = 64.4748$$
$$R^2 = 0.8647^{**}$$
$$N = 34$$

for initial strains Numbers 1 and 2, respectively.

Computation of all possible combination was carried out to select the most significant subset of the independent

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[7] Standard error of estimate.

** Significant at the one per cent level.

variables which contribute significantly in accounting for the variation in total creep. Results of this procedure are summarized in Tables 4 and 5. The following two regression equations were chosen to best represent the relationship between total creep and the independent variables for initial strain levels Numbers 1 and 2, respectively:

$$Y_1 = -50.6658 + 13.2377X_1 + 143.2880X_3 - 10.7829X_4 \quad . . [3]$$

SEE = 33.5114

$R^2 = 0.7680^{**}$

N = 34

and, $Y_2 = -297.2390 + 23.0248X_1 + 407.8860X_3 + 32.7107X_4$

. . . . . [4]

SEE = 65.6095

$R^2 = 0.8550^{**}$

N = 34

These two regression equations were selected for three reasons. Firstly, each of the variables (microfibril angle (X_1), specific gravity (X_3) and extractives content (X_4) individually contributed significantly to the variation in total creep (Y). In other words, the partial coefficients of determination are significant, as indicated in Tables 4 and 5. Secondly, the combination of X_1, X_3 and X_4 gives the highest coefficient of determination (R^2) (Tables 4 and 5, Column 2). Thirdly, the standard error of estimate in

total creep using these three variables together is the lowest among the other combinations given in Tables 4 and 5, Column 3.

From the linear multiple regression analyses presented above, two conclusions can be drawn up to this point. Firstly, for all coniferous wood tissues used, including both earlywood and latewood, the total creep response can be best explained by the mathematical functions [3] and [4] for initial strains Numbers 1 and 2, respectively. Secondly, 76.80 and 85.50 per cent of the variability in total creep response is accounted for by the variation in microfibril angle, specific gravity and extractives content, under both strain conditions Numbers 1 and 2, respectively.

The first conclusion raises the question of whether separate prediction equations should be used for earlywood and latewood or whether these two groups should be represented by a single equation. Therefore, covariance analyses were carried out using the variables X_1, X_3 and X_4. Equations [5] and [6] given with Table 6 represent earlywood and latewood regression model at 3,000 microin. per in. strain level, respectively. Examination of the results given in Table 6 indicates that the differences for testing slopes (creep response) and levels (magnitude of creep response) of Equations [5] and [6] are not significant at the 5 per cent level. Consequently, total creep response of earlywood and latewood specimens tested under a 3,000 microin. per

in. initial strain can be explained by the same prediction regression Equation [3]. On the other hand, creep response of earlywood (Table 7, Equation [7]) and latewood (Table 7, Equation [8]) specimens tested under a 6,000 microin. per in. could be represented by two separate functions. Since the objective of this investigation was to explain creep response in relation to the abovenoted independent variables, it becomes evident that creep response, as a function of microfibril angle, specific gravity, and extractives content under the higher strain level (No. 2), could be represented approximately by a single function (Equation [4]). This facilitated establishment of the phenomenological model for creep response as a function of the structural features and other properties of the tracheid walls, as will be discussed below.

Examination of creep results given in Table 3, reveals that total creep increases as the initial applied strain (constant stress at the micro-level (61)) increases. It is confirmed in Table 8, where differences for testing levels of Equations [3] and [4] are shown to be significantly different at the 0.5 per cent level. This implies that the higher initial strain level (6,000 microin. per in.) would seem to bring into play the effect of the structural features, especially microfibrils, forcing them to react to the applied strain in a pronounced manner. In addition, creep behavior at the lower strain level is shown

in Table 8 to be different from that at the higher strain level. In other words, slopes of the two Equations [3] and [4] are significantly different at the 0.5 per cent level. The implication is that the 6,000 microin.per in. applied to the test specimens exceeded the well established strain level (about 4,000 microin. per in. (65)) at which threshold of set could have actually begun. After the development of set, creep is reported to increase 'more than linearly' with increasing stress level (10, 47, 65, 66). This suggests that above the threshold of set some form of yielding begins (65, 69). Yielding is probably due to structural changes such as formation of secondary bonds in the amorphous region of the fiber (47, 92). These new bonds would tend to 'lock' the molecular chain segments in the deformed region resulting in a permanent deformation after removal of the applied stress.

Based on the above results, it can be seen that the anticipated correlation of microfibril angle of the S2 layer, cell wall crystallinity, specific gravity and extractives content with total creep response has been established. The role which these variables play in creep response is presented below.

II. Relationship of Microfibril Angle to Total Creep

Before investigating the microfibril angle - total creep relationship, it is important to discuss the results given in Table 1. The Table includes 'T' values obtained by X-ray diffraction technique, microfibril angle obtained by mercury impregnation method and θ angle obtained from Meylan's equation (87). It was of interest to calibrate the angle 'T' with the mercury angle. Regression analysis was carried out using all eight samples from the species used in this experiment. The correlation coefficient (r) was 0.7990 which is significant at the 2.5 per cent level. The regression equation is given with Fig. 19. Examination of the results given in Table 1 reveals that the available data are not sufficient to establish a calibration curve for the species used; in addition, the correlation coefficient is not good enough in comparison with that obtained by other workers (Meylan (87)).

A further trial was conducted to calibrate the 'T' angle with Meylan's equation for radiata pine. From the results given in Table 1, it is clear that there is a difference between the mercury angle and that obtained from Meylan's equation. A large difference is noted in the case of earlywoods of Douglas-fir compression and normal wood, Sitka spruce and western hemlock, and also for Douglas-fir latewood (normal wood). On the other hand, it is small in the latewoods of Douglas-fir compression wood,

Sitka spruce and western hemlock. For this reason Meylan's equation was, in general, considered not accurate enough to be used for calibrating the angle 'T' obtained by X-ray technique for species other than radiata pine. Accordingly, the results suggest that a calibration curve is required for each species. This is a serious disadvantage which detracts from the practical application of X-ray technique.

Since the mercury angle is obtained by a direct method, it is considered to be a more representative and accurate value than the one obtained from the Meylan's equation. Consequently, microfibril angles used in the analysis of results were those obtained by the mercury impregnation method. It should be indicated that the averages shown in Table 1, Column 2, are based upon 33 to 73 observations.[8] They are considered to represent the mean microfibril angle for each of the individual strips used for creep tests. This was necessary because it was extremely difficult to assign a precise value for each strip individually.

Microfibrils are composed of aggregations of cellulose chain molecules which represent the main structural component of the framework of tracheid walls. It should be noted that only cellulose is considered herein to contribute to creep response of wood tissues in this study, even though other components such as lignin and hemicelluloses, and also the forces (primary and secondary bonds) which hold the microfibrils

[8]The original experimental data are filed in Room 388, Faculty of Forestry.

together, may affect creep response (29, 117); but this remains to be proven experimentally. Indications from related experiments on molecular stress relaxation by Chow (13a) show that all these major wood components (cellulose, lignin and hemicelluloses) contribute to stress distribution in wood. However, they contribute at different levels and their response to stress is highly time-dependent.

It has been shown that microfibrils control the mechanical behavior of wood and other natural cellulosic fibers to a great extent by acting as elements for supporting the applied stress and the resulting strain (9, 18, 47, 60, 103, 117, 128). If the microfibrils were arranged parallel to the longitudinal axes of the fibers or tracheids, they would be in a position to resist deformation originating from tensile loads with maximum efficiency. Unfortunately, these structual elements are oriented at an angle to the longitudinal axis of the tracheid in the thick S2 layer. This angle differs between species as well as across the annual increment from earlywood to latewood (48, 50, 51, 111). It can be seen from the experimental values presented in Table 1 that Sitka spruce is characterized by the lowest microfibril angle, (9.22°) whereas the earlywood of Douglas-fir compression wood is characterized by the highest angle (28.22°) among the wood tissues used. In general, earlywood has a larger microfibril angle than latewood. This variability is directly related to tracheid

length, where, within one annual increment, earlywood has always shorter tracheids then latewood (115, 118, 155). Accordingly, species or wood tissues (earlywood or latewood) are expected to react differently to the applied constant stress, depending largely on the microfibril angle.

According to the statistical analyses, microfibril angle is the most important structural feature affecting creep response, whether as a single variable or combined with specific gravity and extractives content in the multiple regression Equations [3] and [4]. Based on the 34 specimens from earlywood and latewood tissues of the abovenoted species, the relationships between microfibril angle and total creep response under strain level No. 1 (Fig. 21) and strain level No. 2 (Fig. 22) are established herein.

It can be seen from the simple regression equation given in Fig. 21 that 66.90 per cent of the variation in creep response is attributable to microfibril angle at 3,000 microin. per in. strain level. At the higher initial strain level, 6,000 microin. per in., 68.19 per cent of the variation in creep response is attributable to microfibril angle (Fig. 22). This suggests that microfibril angle must play an important and consistent part in creep response. In general, total creep increases as microfibril angle increases. A specimen having a large microfibril angle prior to application of a constant tensile strain could logically be expected to react to a greater degree than a specimen having a relatively

small microfibril angle. This reaction would result in a larger deformation in the case of the former.

During the course of this work microfibril angle was not measured after applying strain due to some experimental limitations. However, close examination of the graphs presented in Figures 15 and 16, for example,[9] reveals that the rate of creep at both strain levels used is larger in the case of Douglas-fir earlywood (an angle of 21.63°) than in the case of spruce latewood (an angle of 9.22). This larger rate of change could be considered a result of the expected large movement of microfibrils to orient themselves with the applied constant strain. This argument is supported by the work of Jentzen (60) on longleaf pine earlywood and latewood pulps. Jentzen has stated that earlywood tracheid skeletons underwent a greater change in their 'crystallite orientation' than did the delignified latewood tracheid skeletons under the same load in tension. Similar results were also reported by Hill (47) for the latewood pulp of the same species. Working on sisal fibers, Balashov et al. (6) were able to record a decrease in microfibril angle under tensile strains that increased from 5 to 20 per cent.

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[9]The rest of creep diagrams are filed in Room 388, Faculty of Forestry.

Accepting the fact that microfibril angle becomes smaller under a constant strain for a relatively long period of time (60 minutes) it follows that the change towards a smaller angle can be expected to be large for a specimen having a large initial angle. This will in turn lead to higher creep deformation due to the large expected movement associated with adjustment of the stiff inextensible microfibrils, to accommodate the applied strain without failure. The possibility also exists that the microfibrils might slip past each other if the applied strain is large enough, as in the case of initial strain of 6,000 microin. per in., giving rise to the high observed creep (6, 117).

These results indicate that the microfibril angle of the S2 layer of the tracheid wall is one of the fundamental characteristics of the cell wall and exerts a profound effect on the time-dependent behavior of wood. Therefore, microfibril angle should be considered as one important basic measurement for selecting material of the required strength for specific purposes.

III. Relationship of Cell Wall Crystallinity to Total Creep

Microfibril angle is not the only important structural feature affecting the creep response of wood. According to the generally accepted fringed fibrillar model for the supermolecular arrangement of the cellulose chains, the micro-

fibrils are considered to be large, imperfect crystals, separated along their peripheries by non-crystalline regions (35, 37). Consequently, the relative degree of cell wall crystallinity should logically affect creep behavior of wood. According to the results of this investigation, total creep response decreases as the relative degree of crystallinity in tracheid walls increases (Figures 23 and 24). As a single structural feature, cell wall crystallinity was found to contribute up to 64.27 and 54.11 per cent of the total variability in creep response under initial strain conditions Numbers 1 and 2, respectively.

Examination of Table 2 indicates that within one annual increment of the species used, except western hemlock, latewood tissue has higher relative degree of crystallinity than earlywood. The averages of this characteristic are shown below.

RELATIVE DEGREE OF CRYSTALLINITY (PER CENT)

	Earlywood	Latewood
Douglas-fir		
Normal wood	59.59	63.80
Compression wood	58.16	60.84
Sitka spruce	61.75	65.02
Western hemlock	61.14	58.50

These results confirm the general trend observed by Holzer and Lewis (53) on Douglas-fir, and also by Lindgren (77) on Swedish spruce, that within an annual increment, earlywood is lower in crystallinity than latewood.

The variability between earlywood and latewood with regard to the abovenoted characteristic could possibly be due to two causes:

1. It seems that the presence of lignin reduces the degree of cellulose lattice perfection. This is confirmed by two indications in the literature:
   (a) Within one annual increment, except for western hemlock, earlywood is two to three per cent higher in lignin content than latewood (160).
   (b) Removal of lignin from slash pine samples increased the proportion of crystalline material (98a).
2. Earlywood has larger microfibril angle than latewood (Table 1), within the same annual increment. In the present study, microfibril angle is shown to be strongly related (inversely) to the relative degree of crystallinity (Fig. 25). This relationship will be discussed below.

With regard to compression wood, the above results reveal that the relative degree of crystallinity is consider-

ably lower than that of Douglas-fir normal wood. The difference is more pronounced in the case of latewood tissues. Numerical values of this characteristic are higher than those obtained by Lee (73). In the present study, averages are 61.7 and 59.5 per cent for Douglas-fir normal and compression wood, whereas those obtained by Lee were 54.3 and 46.4 per cent. This is probably due to the fact that Lee used samples from different annual increments. In addition, he determined crystallinity on wood pulp prepared by the peracetic acid method. Recently, similar differences were also reported between normal and compression wood of loblolly pine by Parham (105a).

The higher lignin content (138), larger microfibril angle, and possibly the absence of the S3 layer of the compression wood tracheids appear to be responsible for its lower relative degree of crystallinity.

The role which crystallinity plays in influencing creep response is possible since the crystallites form the rigid reinforcing part of the tracheid wall structure. If this well ordered portion is predominant, wood exhibits a high degree of resistance to the stress which is applied for a long period of time, thereby minimizing the development of excessive creep.

A controversial issue has been reported in the literature concerning the effect of applied load on crystallinity of the cell wall. Murphey (92) has reported

that crystallinity of yellow birch and sugar maple increased as the tensile load was increased. This change was constant over a 24-hour period, but the crystallinity did not return to the original level after a recovery period. On the other hand, Hill (47) and Jentzen (60), using longleaf pine holocellulose tracheid skeletons from earlywood and latewood tissues, did not observe any significant change in crystallinity due to tensile load imposed during drying. In spite of this controversy, it is reasonable to believe that, under stretching conditions, there may be some molecular movement in the amorphous portion of the cell wall which would result in improving the degree of structural perfection. This would happen through increase in the degree of crystallinity. Whether the degree of crystallinity changes or not requires further investigation, but it is at least clear from the results of this experiment that the initial degree of crystallinity is an important factor in predicting total creep response.

Examination of the multiple regression analyses (Tables 4 and 5) reveals that, after adjusting for the linear relationship between total creep and each of the independent variables, microfibril angle, specific gravity, and extractives content, the relative degree of crystallinity is no longer a significant variable. This behavior was observed for both initial strain levels used and, accordingly, the relative amount of crystallinity was the first independent

variable to be eliminated from the regression equations. The reason for this may be found in the fact that crystallinity is highly correlated with microfibril angle (r = 0.9310 for the lower strain level and 0.9238 for the higher strain level) (Table 9). Therefore, if microfibril angle is included in the regression equation it also takes care of the crystallinity effect. The relationship between microfibril angle and crystallinity is shown in Fig. 25. It can be seen from this figure that the small microfibril angle is associated with a high relative degree of crystallinity. Based on eight measurements only, the degree of correlation was found for the combined data to be very high (r = 0.9284), as shown in Fig. 25.

Crystallinity of cellulose fibers has been under investigation for a relatively long period of time. The most difficult problem has been and still is the inability of scientists to separate the effect of crystallinity on strength properties of natural and regenerated cellulose fibers from that of crystallites orientation (57, 143). With a heterogenous material such as wood, the problem is greater.

The relationship between crystallinity and microfibril angle was reported by Lindgren (77) for Swedish spruce. A positive relationship between the crystallinity of the paper fibers and fiber length was reported by Ohta et al. (101). Since the microfibril angle decreases as the fiber length increases (115, 118, 155), the microfibril

angle should be inversely related to the cell wall crystallinity.

To explain this relationship one could consider the theoretical and experimental work by Cave (13), who has shown that microfibril direction scatter increases with increasing mean microfibril angle about the longitudinal tracheid axis. This, in fact, would mean that in the case of a small (steep) angle, the scatter around the mean microfibril angle is smaller and the microfibrils in the S2 layer probably lie almost parallel to each other. As a result, the relative degree of amorphous material, (amorphous cellulose, hemicelluloses and lignin) required to fill the micro-spaces between the microfibrils would be smaller. Considering the case of a large angle, the microfibrils are probably not parallel to each other; consequently, relatively large micro-spaces between the microfibrils would be occupied by the amorphous material. This would then result in a relatively lower crystallinity of the cell wall as shown by Fig. 25, and Tables 1 and 2.

A second possible reason for this relationship may be that cellulose chain molecules, in the case of a small microfibril angle, will have a better chance for increased frequency of cross links (bonding between neighbouring chains) along their unit length. Consequently, a tendency of improved geometric order should be observed with better chain coherence in the resulting cellulose as com-

pared to situations associated with tracheids characterized by larger microfibril angle. It should be indicated that the abovenoted reasons are attempts to explain the relationship, however, the exact nature of it still requires further investigation.

It should be re-emphasized that, with regard to creep response of wood in tension parallel to the grain, microfibril angle should logically be used in the multiple regression model (Equations [3] and [4]). In doing so, the effect of crystallinity content is included primarily due to its high correlation with microfibril angle. On the other hand, because of this same correlation, relative degree of crystallinity could be used in the abovenoted models instead of microfibril angle but with about 10 per cent reduction in the R^2 values (Tables 4 and 5). Reasons for this high degree of correlation, as noted above, remain conjectural.

IV. Relationship of Specific Gravity to Total Creep

Specific gravity, i.e., the amount of cell wall substance contained in a unit volume, did not contribute significantly to creep response as a single variable (r^2 = 0.0848 and 0.0148 for the lower (Fig. 26) and higher (Fig. 27) initial strains, respectively). On the other hand when specific gravity was combined with microfibril angle and extractives content, it contributed significantly to creep behavior. In other words, specific gravity became

an important variable after adjusting for the linear relationship between creep and each of microfibril angle and extractives content. This is due to the fact that specific gravity cannot act alone, but must be combined with the other variables.

Examination of the results given in Table 3, Columns 2 and 5, reveals that specific gravity values of Douglas-fir earlywood (normal wood) samples used for the lower initial strain level are different from those used for the higher initial strain level. This is probably due to the morphological variation such as cell wall thickness and cell diameter between the two groups of samples. In addition, it is supported by the unpublished results of Parker (106a) who found substantial differences in specific gravity along the circumference of the same annual increment.

The interpretation given to the effect of specific gravity is the usual one. Besides representing the amount of cell wall material per unit volume, woods with high specific gravity also contain a high ratio of S2 layer to total tracheid wall thickness (24, 60, 64). The microfibrils in this layer make a smaller angle with the longitudinal tracheid axis than do the other cell wall layers. Consequently, a large portion of the cell wall would have a small microfibril angle, which in turn limits the deformation to a large extent. It is confirmed by the results given in Table 9 that specific gravity is inversely correlated with

the microfibril angle. Therefore, total creep response decreases as specific gravity increases. The result is in agreement with the work done by Erickson and Sauer (24) and also confirms King's findings (65). The latter has concluded that species of low density and high extensibility exhibit greater creep than species with a high specific gravity but low extensibility, in and below the region of the threshold of set. This reported inverse relationship between extensibility and specific gravity could also be explained by utilizing an approach similar to that used to explain the relationship between creep and specific gravity.

Another point of interest is that the relationship between the total creep response and specific gravity is more pronounced under the 3,000 than under the 6,000 microin. per in. strain level. Keeping in mind King's work, in which he stated that the creep - initial strain relationship was linear up to the threshold of set and curvilinear thereafter, it is possible that below the threshold of set, specific gravity plays a relatively important part. Once set occurs, other variables, especially microfibril angle, interact together and overshadow the importance of specific gravity in relation to creep response. This actually confirms Kellogg's results (61), wherein little or no correlation was found between the species residual and specific gravity above the threshold of set.

It is shown in Table 9A that specific gravity is positively correlated with the cell wall crystallinity. The relationship does not require further explanation, since the crystalline cellulose is supposed to have higher specific gravity than the amorphous one. This is due to the fact that the amount of cell wall material per unit volume of the former is greater than that of the latter. However, the same relationship did not reach significant level for the samples tested at the higher strain level (Table 9B). This probably is due to the original differences in specific gravity of groups of Douglas-fir earlywood (normal wood) samples tested at each strain level (Table 3). Nevertheless, it is unlikely that the above relationship is applicable at the macro-level.

V. Relationship of Extractives Content to Total Creep

Extractives content for each group of samples is given in Table 3. The averages are 6.14, 7.05, 4.16 and 4.89 per cent for Douglas-fir normal and compression wood, Sitka spruce and western hemlock, respectively. These averages are obtained by simple calculation of the values given in Table 3 for each growth increment. Examining the published data of the extractives content for the same species revealed that the average extractives content differs from one authority to the other, depending on the method used for determination and also on the basis for calculation (58, 74). However, the above values are not unreasonable in comparison with those reported by Lewis (74).[10]

[10] 5.92, 4.90 and 5.30 per cent for Douglas-fir, black spruce and western hemlock, respectively.

Determination of the extractives content was done on samples after they were used for creep tests; the amount of extractives was then correlated with the total creep response. The ideal experimental approach to the creep response- extractives content relationship would be to run creep tests on control specimens with extractives removed. But it was realized that, during extraction, some changes might have taken place in the samples due to the effect of drying and also due to swelling changes that occur during extraction. Therefore, a comparison between control and extracted samples is not valid and would be misleading, under this condition.

The importance of extractives effect on creep response has not been clearly determined by the few studies done in the last decade on species entirely different from the ones used in this investigation. The reason lies in the fact that the exact location of extractives in wood tissues is not well defined. If extractives are located on the lumen surfaces of tracheids and of wood rays, the major repository in wood structure, they would not be expected to influence creep response and other mechanical properties; if they are located within the cell wall structure, i.e., in the amorphous region, they would probably affect creep and other mechanical properties. A few studies have shown that appreciable amount of extractives is located in the cell wall structure (90, 105, 137). Tarkow and Krueger (137), for example, have

found that approximately 75 per cent of the total water soluble extractives of redwood is located within the cell wall structure, probably in the amorphous region. But it must be realized that redwood contains a large amount of water soluble extractives (over 20 per cent of the oven-dry weight); whereas the total amount of extractives, in any one of the species used in the present investigation, did not even reach 10 per cent of the extractive-free oven-dry weight.

The result of this author's work indicated that extractives content is an important variable in governing creep response whether as a single parameter (Figures 28 and 29) or combined with others. It is even more important than specific gravity (Figures 26, 27, 28 and 29). As the extractives content increases, more creep response is expected. This relationship between creep response and extractives content is not only possible under high temperature (24, 94, 96) but also under ambient temperature as used in this work (73 ± 3.5°F).

It is noted in this study that the r^2 value for total creep-extractives content relationship is 16.98 per cent at the 3,000 microin. per in. strain level (Fig. 28) and 47.88 per cent at the 6,000 microin. per in. strain level (Fig. 29). This is due to the fact that the samples used for the latter contain larger percentages of extractives than those used for the former, except for western hemlock earlywood (Table 3). Differences in the extractive content

between the two cases are given in percentage below.

	Earlywood	Latewood
Douglas-fir		
Normal wood	19.8[11]	28.0
Compression wood	10.7	3.5
Sitka spruce	15.3	11.1
Western hemlock	14.3	21.3

Accordingly, if the amount of extractives increases, as pointed out above, higher correlation would be expected with total creep.

Extractives are not expected to affect creep response of wood unless they are partly located in the cell wall structure. Since it was found in this study that creep response depends partly on extractives content, it is reasonable to believe that part of these extractives was located within the cell wall structure, especially in the amorphous region. This indirect evidence is, in general, in agreement with the results obtained by Tarkow and Krueger (139) and also by Morgen and Orsler (90) on very different species, as indicated earlier. However, a more detailed study of the absolute creep values of extracted and unextracted wood sections might be indicative of the real action by which extractives exert their effects.

[11]This means that the average extractives content of Douglas-fir earlywood samples used for the higher strain level is 19.8 per cent higher than that of the samples used for the lower strain level.

The role which extractives play in controlling creep response of wood in tension parallel to the grain is probably due to their induced plasticization effect. Since crystalline regions of the cell wall are believed to be impermeable to these compounds, it is not unreasonable to propose that part of them is located in the amorphous regions, between microfibrils, as indicated earlier. It follows that their presence in these particular micro-spaces reduces the bonding (primary and secondary) between structural elements and hence minimizes their ability to resist applied stresses. As a result, the movement of microfibrils under the applied stress is facilitated by the existence of extractives.

Some interesting correlations were found in this study between extractives and the other independent variables used. Extractives content was shown in Table 9 to be related positively to the microfibril angle. This means that as the latter becomes larger there is the possibility for more of the former to exist in the amorphous regions. It is confirmed by the inverse correlation found between the relative degree of crystallinity and extractives content. Extractives content was also inversely related to specific gravity in the case of the samples tested at the lower strain level (Table 9A). Since it is proposed that extractives reduce bonding between microfibrils, therefore, their existence would result in less wood substance per unit volume. The same two independent variables were not significantly correlated for samples tested at the higher strain level

(Table 9B). The probable reason for this anomaly is that Douglas-fir earlywood samples (normal wood) tested at the 6,000 microin. per in. strain level have higher specific gravities than those tested at the 3,000 microin. per in. strain level.

In summarizing the above results, it is evident that structural features, such as microfibril angle and specific gravity, in addition to extractives content, play an important role in governing the total creep response of wood (Equations [3] and [4]). This response is enhanced in earlywood mainly by the large microfibril angle; in addition, low specific gravity (Figures 26 and 27) and higher extractives content (Figures 29 and 29), help in making creep response more pronounced.

VI. Significance of Results

The results of this study appear to provide new and worthwhile information concerning creep response as a function of some structural features of wood. The information obtained is of fundamental scientific interest because of the relationships revealed between creep response in tension parallel to the grain and each of microfibril angle of the S2 layer, relative degree of crystallinity of cell wall cellulose, specific gravity and extractives content. This increases knowledge about the rheological behavior of wood. Such knowledge may also give guidance to tree breeders and forest managers who, through their combined efforts, aim at faster tree growth and decreased rotations. The value of

such efforts can be measured only through maintenance or improvement of important wood characteristics. However, of particular importance was the determination of the most important structural feature controlling the magnitude of creep response, namely microfibril angle of the S2 layer. This supports the earlier evidence that microfibril angle should be considered as one of the important basic measurements which influence physical and mechanical properties of wood.

The relationships revealed in this study, between total creep and the abovenoted characteristics, could also help in formulating an appropriate rheological model for a complete description of the creep behavior of wood, i.e., phenomenological rheology.

The knowledge obtained from this study could also be combined with other parameters, which may be revealed at the micro-level (for example, lignin content, hemicelluloses content and primary and secondary bonds which hold the microfibrils together), and at the macro-level such as grain deviation, latewood per cent and wood defects, for a full understanding of creep behavior of wood in tension parallel to the grain. For practical purposes, structural members from samples with small microfibril angle, high degree of crystallinity, high specific gravity and low extractives content would show a low tendency for creep under permanent load. Accordingly, their working stresses could

perhaps be higher due to the lower anticipated reduction in their basic stresses.

RECOMMENDATIONS FOR FURTHER RESEARCH

The information derived from this study would be useful in determining the most important microscopic, submicroscopic and other features of wood which control creep response in tension parallel to the grain. Microfibril angle was found to be the most important and most logical structural feature governing the magnitude of creep response. It is suggested that, with an appropriate experimental technique, further research could be conducted to determine the change in the microfibril angle associated with the deformation which results from a stress applied over a long period of time. A part of this deformation is said to be non-recoverable, which could be explained by the expected permanent decrease in the microfibril angle. This, if proven, would increase fundamental knowledge concerning the relationship between submicroscopic properties and time-dependent behavior of wood, the latter of which is relatively new.

It has been observed in this study that cell wall crystallinity is highly correlated with the microfibril angle. It is recommended that a study be initiated to re-examine this relationship to determine whether it is valid for other coniferous woods and for hardwoods. This would increase fundamental knowledge with regard to correlations existing among wood physical properties.

Another aspect which requires intensive investigation is the development of an appropriate phenomenological rheological model, which could characterize wood by a complete description of its mechanical behavior under long term loading. Since lignin and hemicelluloses are expected to influence the rheological properties of wood, it is recommended that a study be conducted to determine the role which these components play in governing creep response.

CONCLUSION

From the results of this study on creep response as a function of some structural features, specific gravity and extractives content of Douglas-fir normal and compression wood, and normal wood of Sitka spruce and western hemlock, the following conclusions are drawn:

I. Under constant temperature and relative humidity (73 ± 3.5°F and 50 ± 2 per cent) conditions, total creep response of Douglas-fir, normal and compression wood, Sitka spruce and western hemlock, is controlled by the microfibril angle of the S2 layer, and the specific gravity and extractives content of the wood. These variables contribute up to 76.8 and 85.5 per cent of the total variability in creep response in the case of 3,000 and 6,000 microin. per in. initial strain level, respectively.

II. Microfibril angle is the most important and most logical structural feature of the tracheid walls governing creep response. The total creep response increased as the microfibril angle increased. It is proposed that the creep-inducing stresses cause reorientation of the microfibrils by forcing them to align themselves more nearly parallel to the longitudinal tracheid axis. As a result, the microfibril angle in the S2 layer of the tracheid wall becomes steeper

(smaller). This change in the angle appears to be larger for a specimen having a larger angle prior to the application of a constant strain.

III. Cell wall crystallinity, expressed as relative degree of crystallinity, i.e., crystallinity index, affects total creep response. A relatively high percentage of crystallinity increases the rigidity of the cell wall which thereby resists excessive creep.

IV. Cell wall crystallinity is inversely correlated with the microfibril angle of the S2 layer.

V. Specific gravity affects creep response only when it is combined with microfibril angle and extractives content. It is suggested that if the specific gravity of a wood tissue is high, the ratio of S2 layer to total tracheid wall thickness is also high, hence there is a large volume of the cell wall in which the microfibrils are at a small angle to the long tracheid axis. This would decrease the total creep response.

VI. Extractives content plays a relatively important role in controlling creep behavior of wood under a constant temperature of 73 ± 3.5°F and 50 ± 2 per cent relative humidity. The extractives-total creep relationship suggests that a part of these extractives is in the amorphous region of the cell wall structure.

VII. Extractives in the cell wall probably act as plasticizers causing a reduction in the primary and secondary bonding between microfibrils. This would facilitate the movement of the stiff inextensible microfibrils to accommodate the creep-inducing stresses.

VIII. Total creep response is affected by the initial applied strain, increasing as the initial strain increases.

IX. Results obtained throughout the thesis are compatible with the hypothesis that short-term creep response of earlywood and latewood tissues of some coniferous species, stressed in tension parallel to the grain, is a function of microfibril angle of the S2 layer of tracheid wall and relative degree of crystallinity in the cell wall, along with specific gravity of that wood tissue and its extractives content.

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TABLE 1

SUMMARY OF MICROFIBRIL ANGLE DETERMINATIONS

Species	Mercury Angle (Degree)	'T' Angle (Degree)	θ Angle (From Meylan's Equation (87)) (Degree)
Douglas-fir			
Normal wood			
Earlywood	21.63(4.98)[a]	20.70	13.44
		21.38	
		19.69	
Average		20.59	
Latewood	12.95(2.48)	23.51	15.16
		22.73	
		23.96	
Average		23.40	
Compression Wood			
Earlywood	28.22(4.03)	38.70	25.35
		39.94	
		41.51	
Average		40.05	
Latewood	17.37(2.74)	26.55	16.93
		27.11	
		25.20	
Average		26.29	
Sitka spruce			
Earlywood	14.30(2.74)	15.64	10.65
		15.98	
		16.43	
Average		16.02	
Latewood	9.22(1.80)	15.53	10.28
		15.08	
		15.64	
Average		15.42	
Western hemlock			
Earlywood	20.74(4.00)	22.28	14.96
		24.19	
		22.73	
Average		23.07	
Latewood	21.26(2.01)	36.00	21.93
		33.08	
		34.31	
Average		34.46	

[a] Values in parentheses are the standard deviations of the averages reported, and are based upon 33 to 73 measurements.

TABLE 2

RELATIVE DEGREE OF CRYSTALLINITY (CRYSTALLINITY INDEX)

Species		Relative Degree of Crystallinity (Crystallinity Index) Per cent
Douglas-fir		
Normal Wood		
Earlywood	1	60.99
	2	58.18
Average		59.59
Latewood	1	64.22
	2	63.38
Average		63.80
Compression Wood		
Earlywood	1	57.33
	2	58.99
Average		58.16
Latewood	1	59.66
	2	62.01
Average		60.84
Sitka spruce		
Earlywood	1	61.54
	2	61.95
Average		61.75
Latewood	1	65.02
Western hemlock		
Earlywood	1	60.73
	2	61.54
Average		61.14
Latewood	1	60.82
	2	56.18
Average		58.50

TABLE 3

SPECIFIC GRAVITY, EXTRACTIVES CONTENT AND TOTAL CREEP FOR THE SAMPLES TESTED AT 3,000 AND 6,000 MICROIN. PER IN. INITIAL STRAIN

Species		Samples Tested at 3,000 Microin. per in. Initial Strain			Samples Tested at 6,000 Microin. per in. Initial Strain		
		Specific[a] Gravity	Extractives Content Based on Extractive-free Oven-dry Weight (Per cent)	Total Creep (Microin. per in.)	Specific Gravity	Extractives Content Based on Extractive-free Oven-dry Weight (Per cent)	Total Creep (Microin. per in.)
Douglas-fir							
Normal wood							
Earlywood	1	0.281	4.81	324	0.376	4.34	674
	2	0.273	6.57	207	0.342	4.38	607
	3	0.287	5.99	197	0.329	9.38	724
	4	0.284	5.46	180	0.332	9.59	610
	5	0.282	5.20	234	0.297	7.38	596
	6	0.295	6.28	214	0.300	7.68	616
Average		0.284	5.72	226.0	0.335	7.13	637.8
Latewood	1	0.773	3.72	200	0.700	5.17	485
Compression Wood							
Earlywood	1	0.303	7.05	370	0.278	8.87	714
	2	0.290	7.57	285	0.308	7.79	787
	3	0.271	7.38	255	0.304	7.86	720
	4	0.311	6.33	254	0.292	7.00	767
	5	0.354	6.88	274			
Average		0.306	7.04	287.6	0.296	7.88	747.0
Latewood	1	0.647	6.36	230	0.738	6.22	636
	2	0.677	6.83	190	0.653	6.99	590
	3	0.691	6.00	220	0.700	6.69	610
Average		0.672	6.40	213.3	0.697	6.63	612.0

TABLE 3 (continued)

Species		Samples Tested at 3,000 Microin. per in. Initial Strain			Samples Tested at 6,000 Microin. per in. Initial Strain		
		Specific Gravity[a]	Extractives Content Based on Extractive-free Oven-dry Weight (Per cent)	Total Creep (Microin. per in.)	Specific Gravity	Extractives Content Based on Extractive-free Oven-dry Weight (Per cent)	Total Creep (Microin. per in.)
Sitka spruce							
Earlywood	1	0.381	3.98	140	0.314	6.21	314
	2	0.312	4.37	145	0.300	6.03	327
	3	0.382	4.92	130	0.358	4.47	307
	4	0.391	4.30	164	0.341	3.44	293
	5	0.362	3.15	160	0.368	4.09	323
	6	0.402	3.92	110			
Average		0.372	4.11	141.5	0.336	4.85	312.8
Latewood	1	0.604	4.01	84	0.557	4.73	270
	2	0.575	4.01	110	0.493	3.45	250
	3	0.552	3.01	107	0.488	4.52	320
	4	0.570	3.53	167	0.511	4.08	234
	5	0.545	3.53	104	0.526	3.57	290
Average		0.569	3.62	114.4	0.515	4.07	272.8
Western hemlock							
Earlywood	1	0.262	5.16	217	0.262	5.83	427
	2	0.248	8.60	182	0.259	5.26	394
	3	0.246	5.87	177	0.261	4.52	454
	4	0.234	5.76	167	0.250	6.16	407
Average		0.248	6.35	185.8	0.258	5.44	420.5
Latewood	1	0.432	3.00	298	0.458	4.38	494
	2	0.413	3.02	254	0.481	3.44	393
	3	0.450	4.35	220	0.467	6.05	440
	4	0.496	3.69	230	0.478	4.75	480
	5				0.488	4.74	527
	6				0.511	3.47	510
Average		0.448	3.52	250.5	0.481	4.47	474.0

[a]Based on oven-dry weight and volume at test.

TABLE 4

MULTIPLE COEFFICIENTS OF DETERMINATION (R^2) AND STANDARD ERRORS OF ESTIMATE (SEE) BASED ON 34 WOOD SPECIMENS TESTED AT 3,000 MICROIN. PER IN. INITIAL STRAIN

Correlation	R^2	SEE	Level of Significance
Total creep vs. microfibril angle and relative degree of crystalinity	0.6812	38.6467	0.5 per cent
Total creep vs. microfibril angle and specific gravity	0.7344	35.2744	0.5 per cent*
Total creep vs. microfibril angle and extractives content	0.7007	37.4409	0.5 per cent
Total creep vs. relative degree of crystallinity and specific gravity	0.6683	39.4198	0.5 per cent
Total creep vs. relative degree of crystallinity and extractives content	0.6434	40.8708	0.5 per cent
Total creep vs. specific gravity and extractives content	0.1898	61.6040	5.0 per cent
Total creep vs. microfibril angle, relative degree of crystallinity and specific gravity	0.7413	35.3869	0.5 per cent
Total creep vs. microfibril angle, specific gravity and extractives content	0.7680	33.5114	0.5 per cent*
Total creep vs. microfibril angle, relative degree of crystallinity and extractives content	0.7014	38.0191	0.5 per cent

TABLE 4 (continued)

Correlation	R^2	SEE	Level of Significance
Total creep vs. relative degree of crystallinity, specific gravity and extractives content	0.6714	39.8842	0.5 per cent
Total creep vs. microfibril angle, relative degree of crystallinity, specific gravity and extractives content	0.7681	34.0773	0.5 per cent

*Partial correlation coefficients are significant.

TABLE 5

MULTIPLE COEFFICIENTS OF DETERMINATION (R^2) AND STANDARD ERRORS OF ESTIMATE (SEE) BASED ON 34 WOOD SPECIMENS TESTED AT 6,000 MICROIN. PER IN. INITIAL STRAIN

Correlation	R^2	SEE	Level of Significance
Total creep vs. microfibril angle and relative degree of crystallinity	0.6869	94.8457	0.5 per cent
Total creep vs. microfibril angle and specific gravity	0.7698	81.3324	0.5 per cent*
Total creep vs. microfibril angle and extractives content	0.7610	82.8748	0.5 per cent*
Total creep vs. relative degree of crystallinity and specific gravity	0.5563	112.9190	0.5 per cent
Total creep vs. relative degree of crystallinity and extractives content	0.7153	90.4467	0.5 per cent*
Total creep vs. specific gravity and extractives content	0.4850	121.6500	0.5 per cent
Total creep vs. microfibril angle, relative degree of crystallinity and specific gravity	0.7996	77.1387	0.5 per cent*
Total creep vs. microfibril angle, specific gravity and extractives content	0.8550	65.6095	0.5 per cent*
Total creep vs. microfibril angle, relative degree of crystallinity and extractives content	0.7610	84.2425	0.5 per cent

TABLE 5 (continued)

Correlation	R^2	SEE	Level of Significance
Total creep vs. relative degree of crystallinity, specific gravity and extractives content	0.7541	85.4505	0.5 per cent*
Total creep vs. micro-fibril angle, relative degree of crystallinity, specific gravity and extractives content	0.8647	64.4748	0.5 per cent

*Partial correlation coefficients are significant.

TABLE 6

COVARIANCE ANALYSIS FOR TESTING THE DIFFERENCE IN TOTAL CREEP BETWEEN EARLYWOOD (EQUATION [5])[a] AND LATEWOOD (EQUATION [6])[b] AT 3,000 MICROIN. PER IN. INITIAL STRAIN

Group	DF	SS	MS	F	Level of Significance
Earlywood	17	24931.4			
Latewood	9	7884.3			
Total	26	32815.7	1262.1		
Difference for testing slopes	3	859.9	286.6	0.227	N.S. [+]
Sums	29	33675.6	1161.2		
Difference for testing levels	1	15.2	15.2	0.013	N.S.
Combined regression	30	33690.8			

[a] $Y = 1.6255 + 13.7204X_1 + 11.0443X_3 - 14.6503X_4$ . . .[5]

[b] $Y = -66.4666 + 13.3015X_1 + 189.6270X_3 - 13.4366X_4$ . . .[6]

[+] Not significant at the 5 per cent level.

TABLE 7

COVARIANCE ANALYSIS FOR TESTING THE DIFFERENCE IN TOTAL CREEP BETWEEN EARLYWOOD (EQUATION [7])[a] AND LATEWOOD (EQUATION [8])[b] AT 6,000 MICROIN. PER IN. INITIAL STRAIN

Group	DF	SS	M.S	F	Level of Significance
Earlywood	15	33106.3			
Latewood	11	18684.8			
Total	26	51791.1	1992.0		
Difference for testing slopes	3	44158.1	14719.4	7.389	0.5 per cent
Sums	29	95949.2	3308.6		
Difference for testing levels	1	33217.8	33217.8	10.040	0.5 per cent
Combined regression	30	129167			

[a] $Y = -783.620 + 32.294X_1 + 1660.890X_3 + 19.769X_4$ . . .[7]

[b] $Y = -354.748 + 18.544X_1 + 756.033X_3 + 16.424X_4$ . . .[8]

TABLE 8

COVARIANCE ANALYSIS FOR TESTING THE DIFFERENCE IN TOTAL CREEP AT 3,000 MICROIN. PER IN. INITIAL STRAIN (EQUATION [3])[a] AND AT 6,000 MICROIN. PER IN. INITIAL STRAIN (EQUATION [4])[b]

Group	DF	SS	MS	F	Level of Significance
3,000 microin. per in. initial strain	30	33697.7			
6,000 microin. per in. initial strain	30	129208.3			
Total	60	162906.0	2715.1		
Difference for testing slopes	3	169149.0	56382.9	20.766	0.5 per cent
Sums	63	332055.0	5270.7		
Difference for testing levels	1	1252030.0	1252030.0	237.545	0.5 per cent
Combined regression	64	1584085.0			

[a] $Y = -50.6658 + 13.2377X_1 + 143.2880X_3 - 10.7829X_4$ . . .[3]

[b] $Y = -297.2390 + 23.0248X_1 + 407.8860X_3 + 32.7107X_4$ . . .[4]

TABLE 9

CORRELATION COEFFICIENTS FOR THE VARIABLES WHICH WERE DETERMINED ON (A) SPECIMENS TESTED AT 3,000 MICROIN. PER IN. AND (B) SPECIMENS TESTED AT 6,000 MICROIN. PER IN. INITIAL STRAIN

A

Variables	Microfibril angle (X_1)	Relative degree of crystallinity (X_2)	Specific Gravity (X_3)	Extractives Content (X_4)	Total Creep (Y)
Microfibril angle (X_1)	1	-0.9310*	-0.6048*	0.6662*	0.8179*
Relative degree of crystallinity (X_2)		1	0.5320*	-0.4856*	-0.8017*
Specific gravity (X_3)			1	-0.3907†	-0.2911 N.S.+
Extractives content (X_4)				1	0.4121†
Total creep (Y)					1

TABLE 9 (continued)

B

Variables	Microfibril angle (X_1)	Relative degree of crystallinity (X_2)	Specific Gravity (X_3)	Extractives Content (X_4)	Total Creep (Y)
Microfibril angle (X_1)	1	-0.9238*	-0.4650*	0.5546*	0.8258*
Relative degree of crystallinity (X_2)		1	0.3233 N.S.	-0.4279†	-0.7356*
Specific gravity (X_3)			1	-0.2841 N.S.	-0.1215 N.S.
Extractives content (X_4)				1	0.6920*
Total creep (Y)					1

*Significant at the one per cent level.

†Significant at the five per cent level.

+Not significant at the five per cent level.

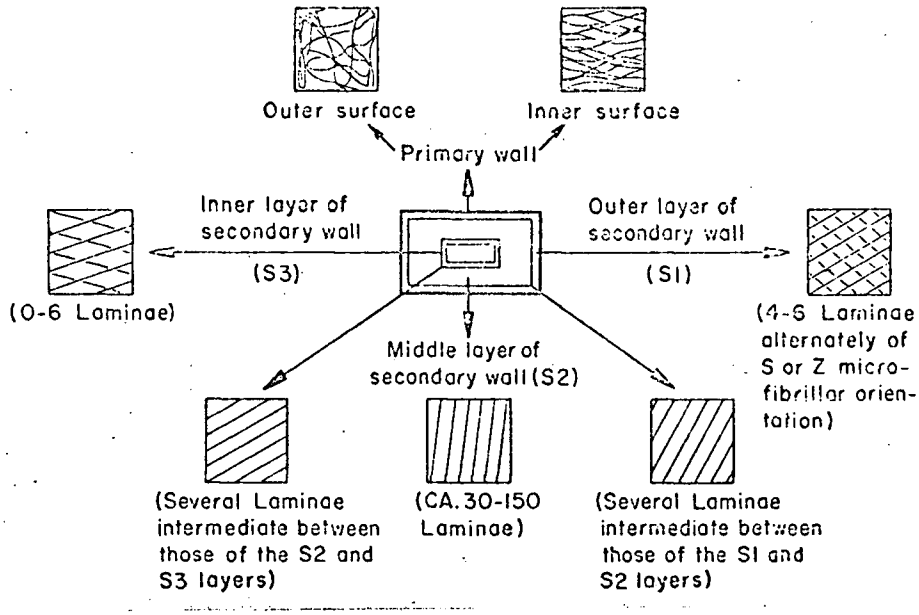


FIG. 1. DIAGRAMMATIC REPRESENTATION OF CELL WALL ORGANIZATION OF A TYPICAL FIBER OR TRACHEID SHOWING THE TEXTURE OF THE DIFFERENT CELL WALL LAYERS, AFTER WARDROP (147).

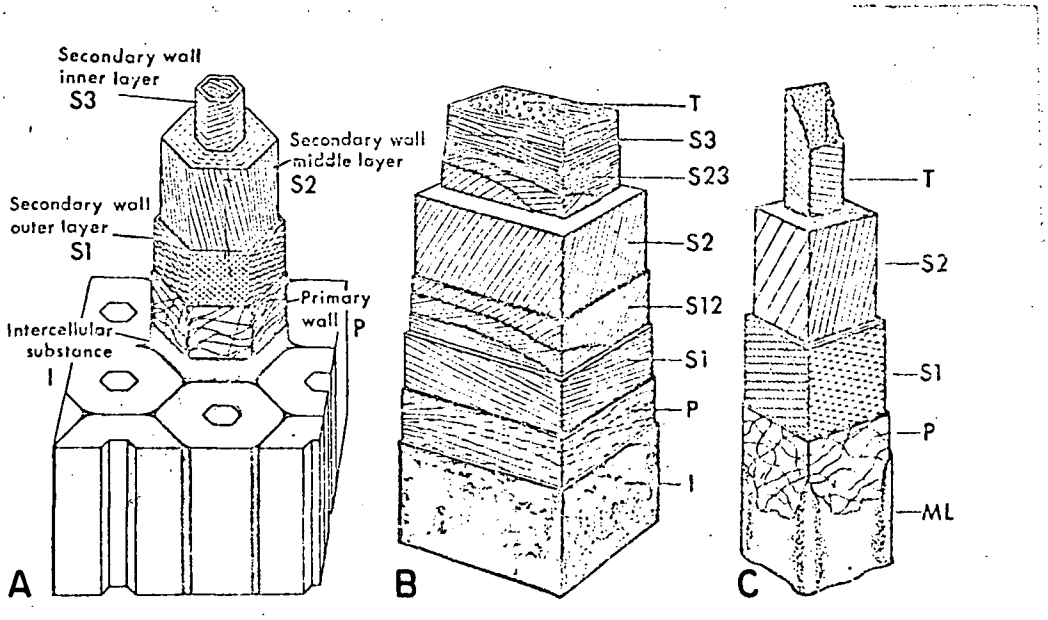


FIG. 2. DIFFERENT CONCEPTS OF CELL WALL ORGANIZATION OF A TYPICAL FIBER OR TRACHEID, SHOWING FIBRILLAR AND/OR MICROFIBRILLAR DIRECTIONS. (A) FROM WARDROP AND BLAND (149); (B) HARADA *ET AL.* (34); (C) FROM FORGACS (26). SEE TEXT FOR LEGEND.

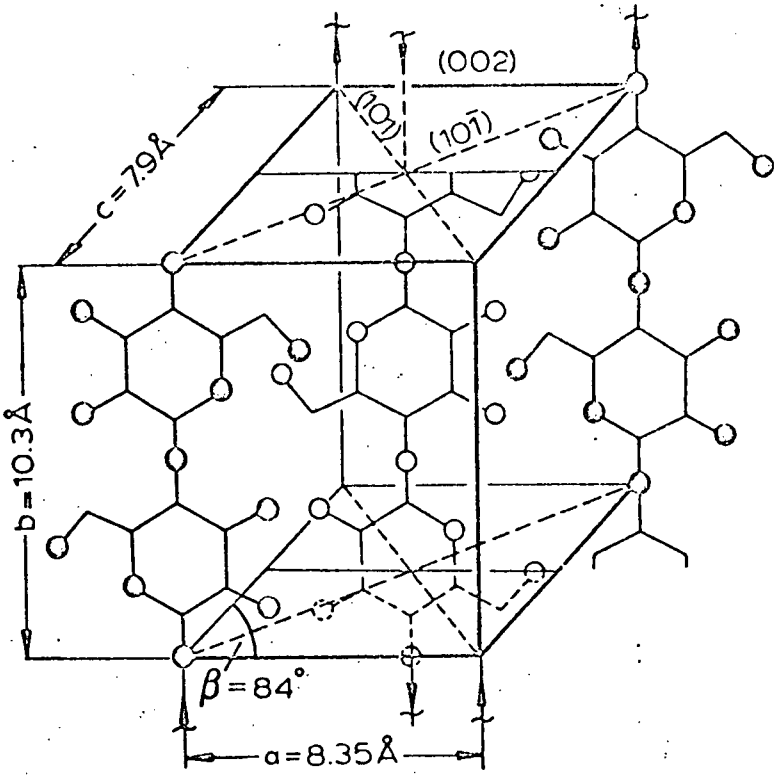


FIG. 3. THE CLASSICAL MODEL OF MEYER AND MISCH (86) FOR THE UNIT CELL OF CRYSTAL LATTICE OF NATIVE CELLULOSE (CELLULOSE 1).

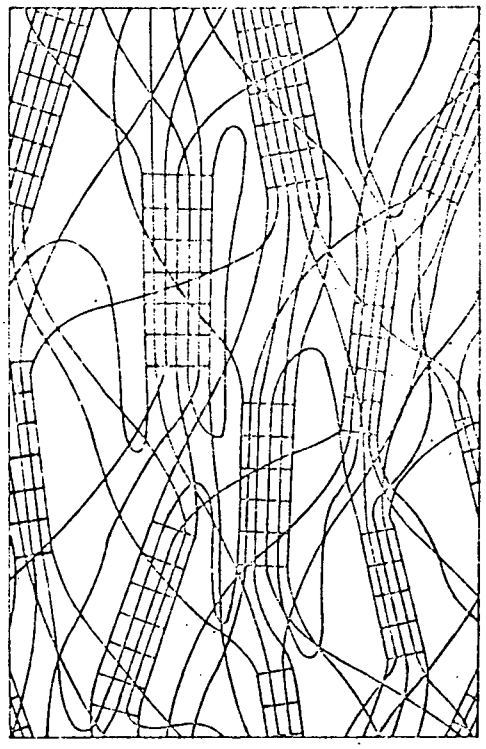


FIG. 4. FRINGED MICELLAR MODEL, AFTER HEARLE (36).

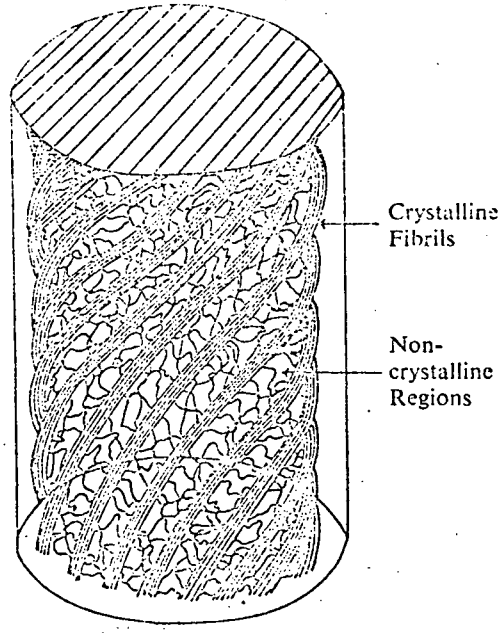


FIG. 5. FRINGED FIBRILLAR MODEL, AFTER HEARLE (36).

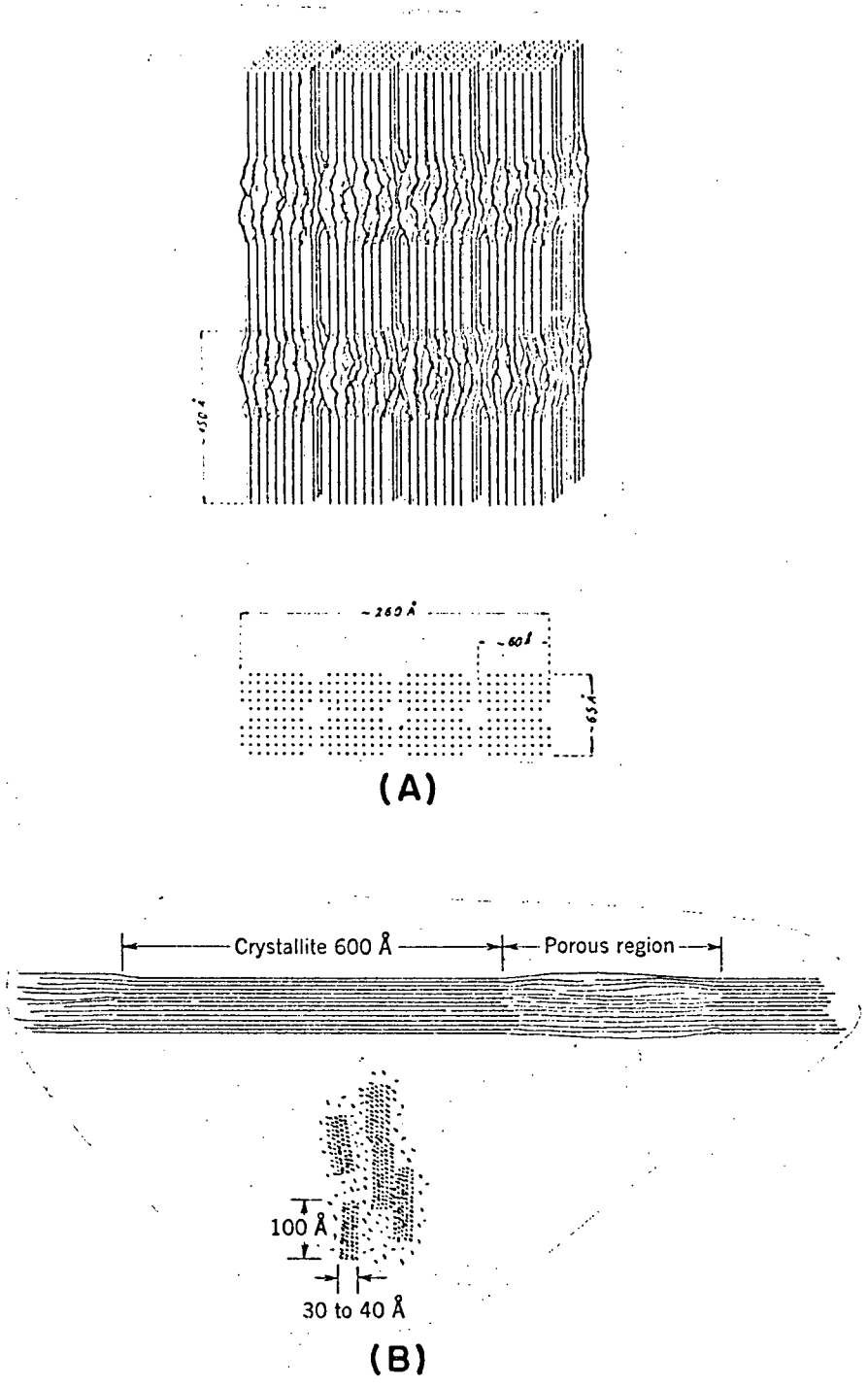


FIG. 6. DIAGRAMMATIC REPRESENTATION OF CELLULOSIC MICROFIBRILS ACCORDING TO CONCEPTS OF (A) HESS *ET AL.* (43) AND (B) RÅNBY (122).

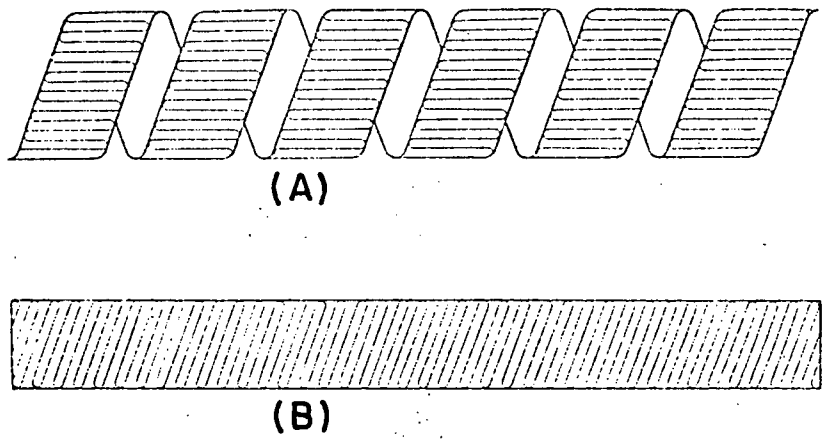


FIG. 7. FOLDED CHAIN MODEL, AFTER MANLEY (79). THE MICROFIBRIL CONSISTS OF A TIGHTLY WOUND HELIX (A) FROM A RIBBON (B) ABOUT 8 Å THICK IN WHICH A SINGLE CELLULOSE CHAIN IS FOLDED REPEATEDLY.

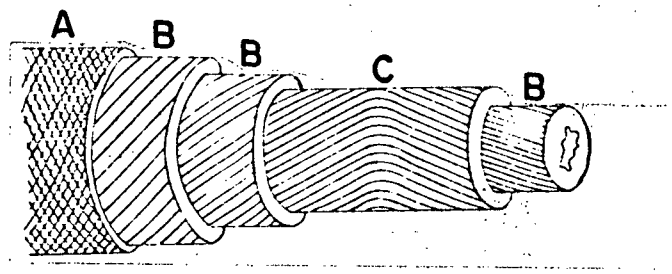


FIG. 8. A MODEL OF THE COTTON FIBER SHOWING THE SPIRAL STRUCTURE AND REVERSALS IN THE GROWTH LAYER: (A) PRIMARY WALL; (B) GROWTH LAYERS; (C) REVERSALS, AFTER ORR *ET AL.* (103).

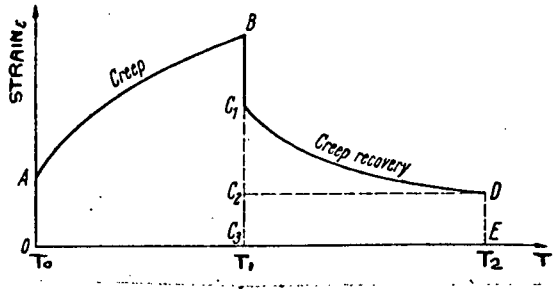


FIG. 9. DEFORMATION OF AN ELASTIC-PLASTIC BODY AS A FUNCTION OF TIME. LOADING DURING TIME T_0 TO T_1, FOLLOWED BY UNLOADING TIME T_1 TO T_2, AFTER KOLLMANN AND CÔTÉ (70).

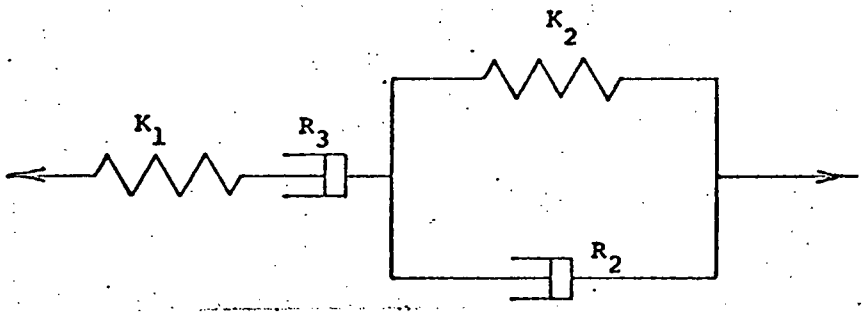


FIG. 10. FOUR-ELEMENT SPRING AND DASHPOT MODEL, AFTER PENTONEY (108). SEE TEXT FOR LEGEND.

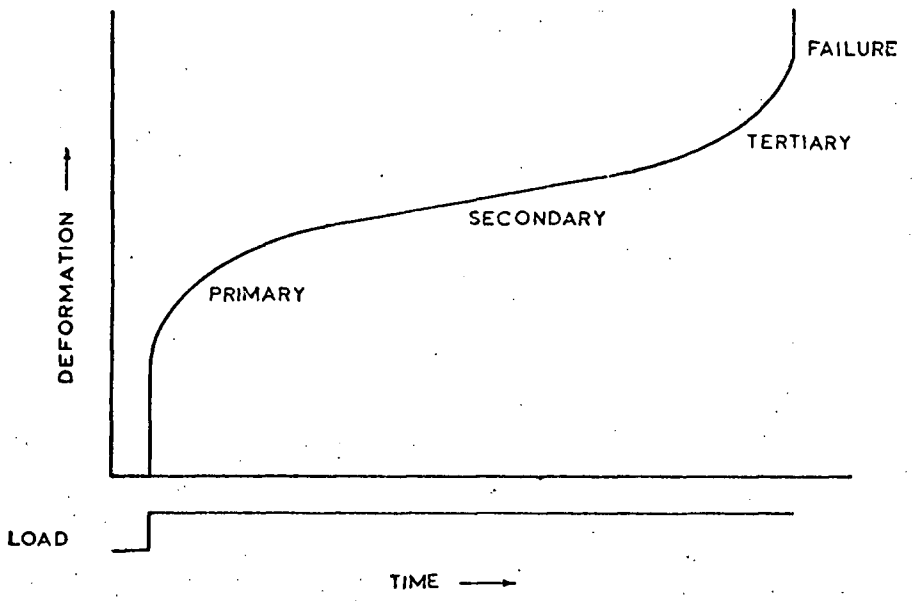


FIG. 11. IDEALIZED LONG-TIME CREEP, AFTER PENTONEY (108).

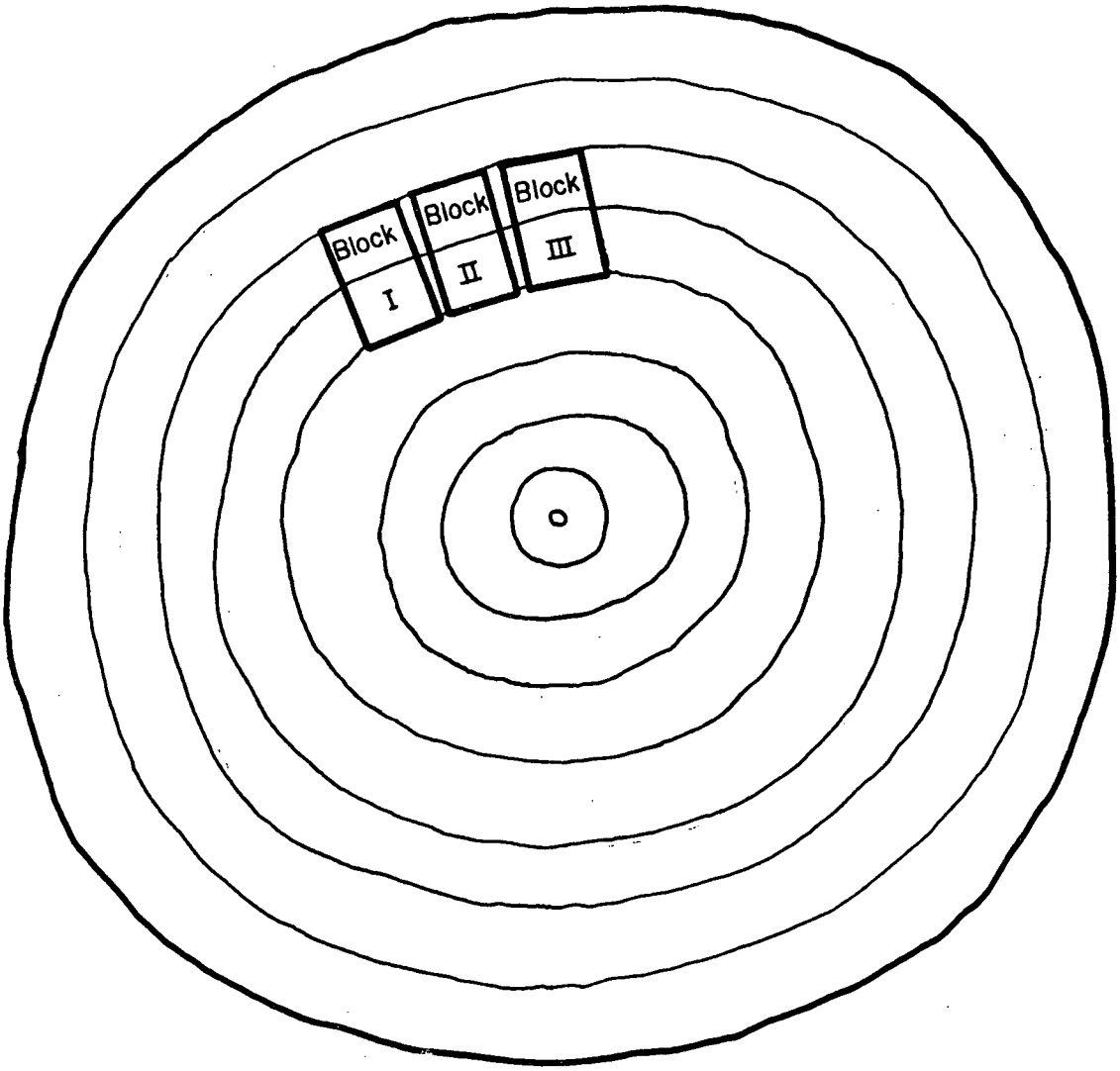


Fig. 12. SCHEMATIC REPRESENTATION FOR SAMPLING PROCEDURE.

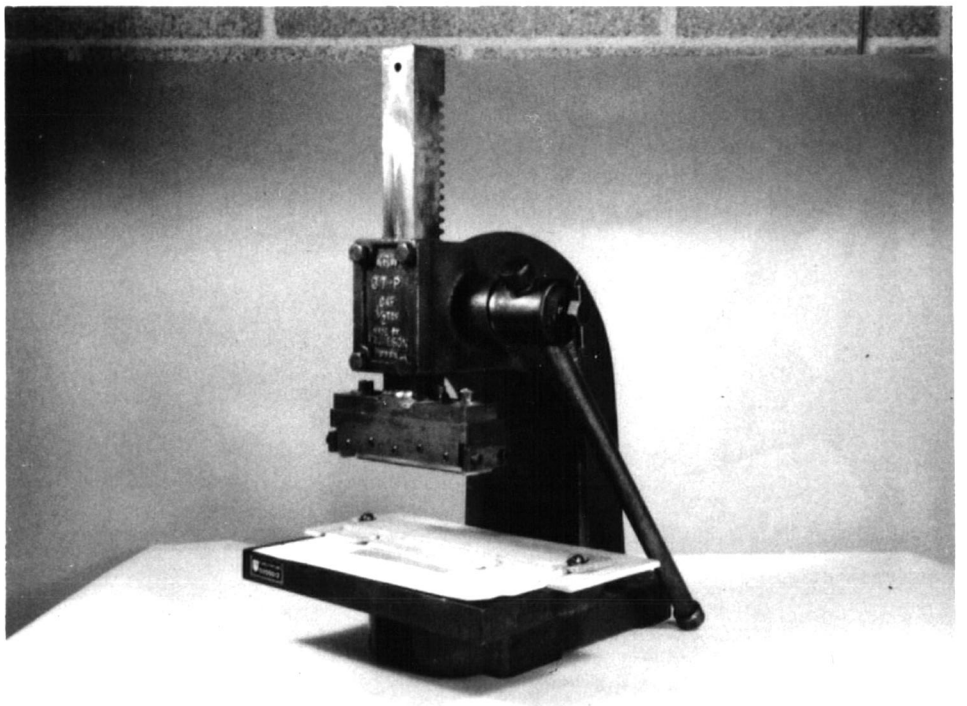
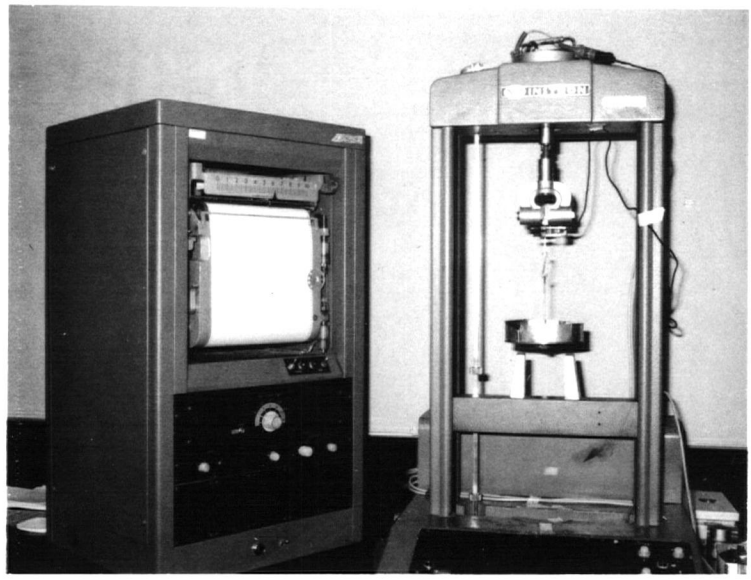
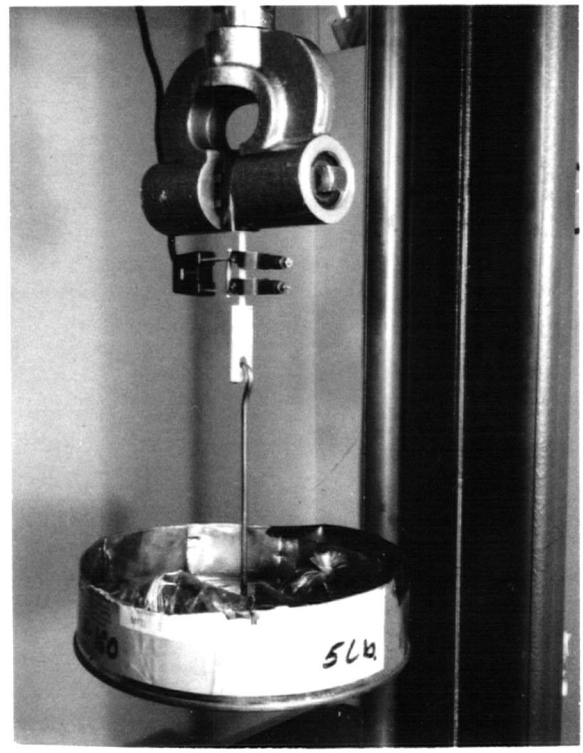


FIG. 13. ARBOR PRESS WITH ADJUSTABLE CUTTING DIE USED FOR CREEP TEST SPECIMEN PREPARATION.



A



B

FIG. 14. CREEP PARALLEL TO THE GRAIN SET-UP. (A) TABLE MODEL INSTRON TESTING MACHINE AND (B) LOADING SYSTEM.

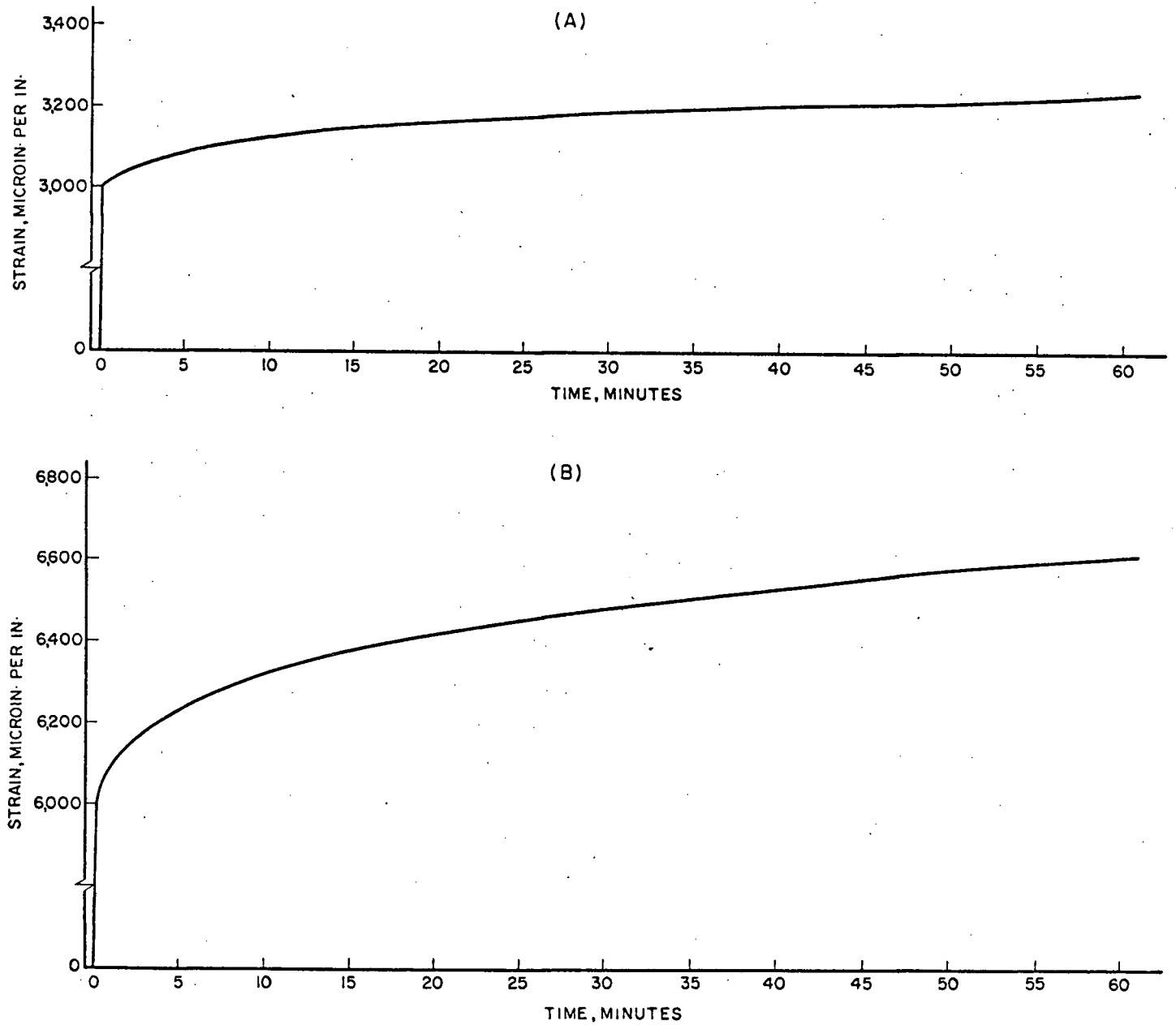


FIG. 15. STRAIN-TIME RELATIONSHIP FOR DOUGLAS-FIR EARLYWOOD (NORMAL WOOD) AT (A) 3,000 MICROIN. PER IN. (SPECIMEN NO. 5) AND (B) 6,000 MICROIN. PER IN. (SPECIMEN NO. 4) INITIAL STRAIN.

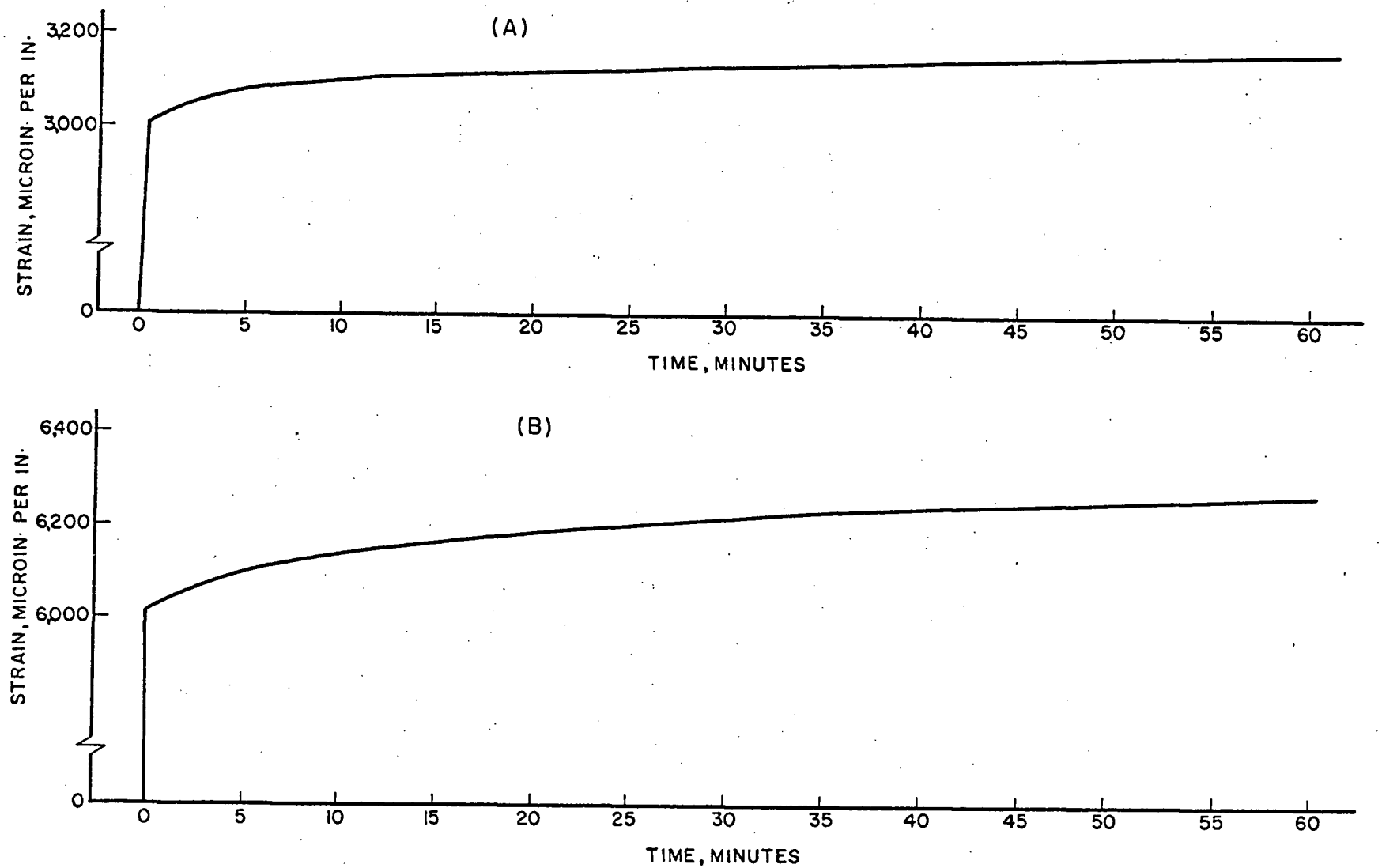


FIG. 16. STRAIN–TIME RELATIONSHIP FOR SITKA SPRUCE LATEWOOD AT (A) 3,000 MICROIN. PER IN. (SPECIMEN NO. 4) AND (B) 6,000 MICROIN. PER IN. (SPECIMEN NO. 1) INITIAL STRAIN.

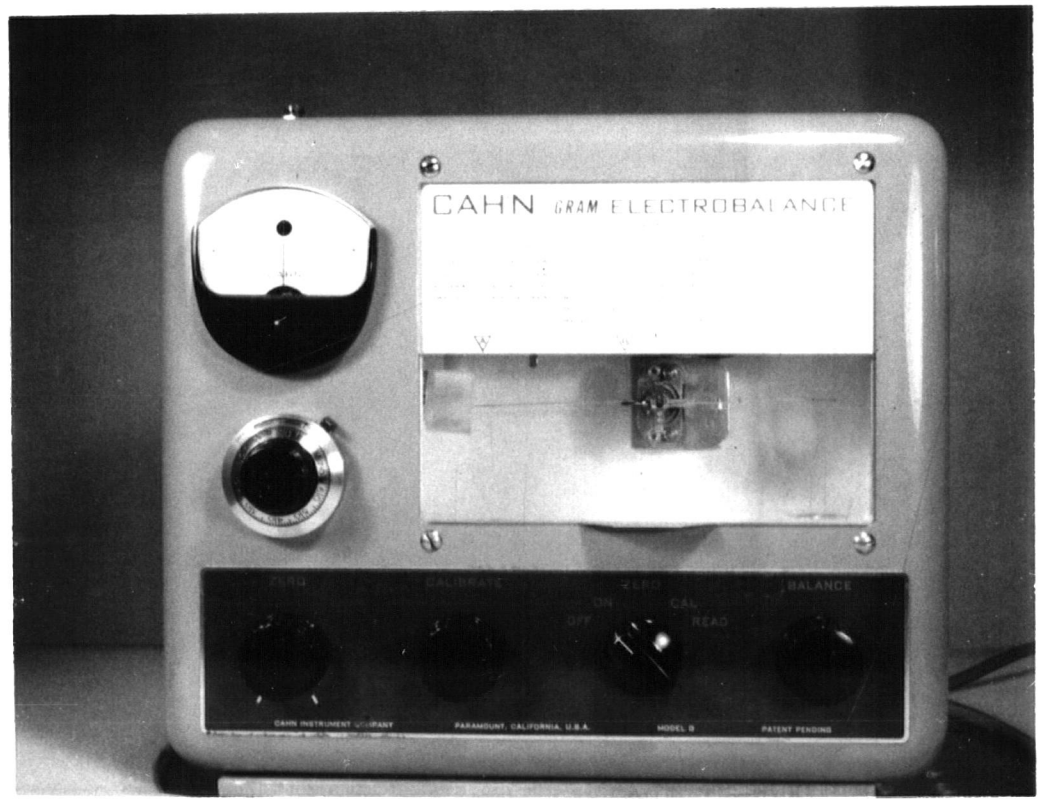
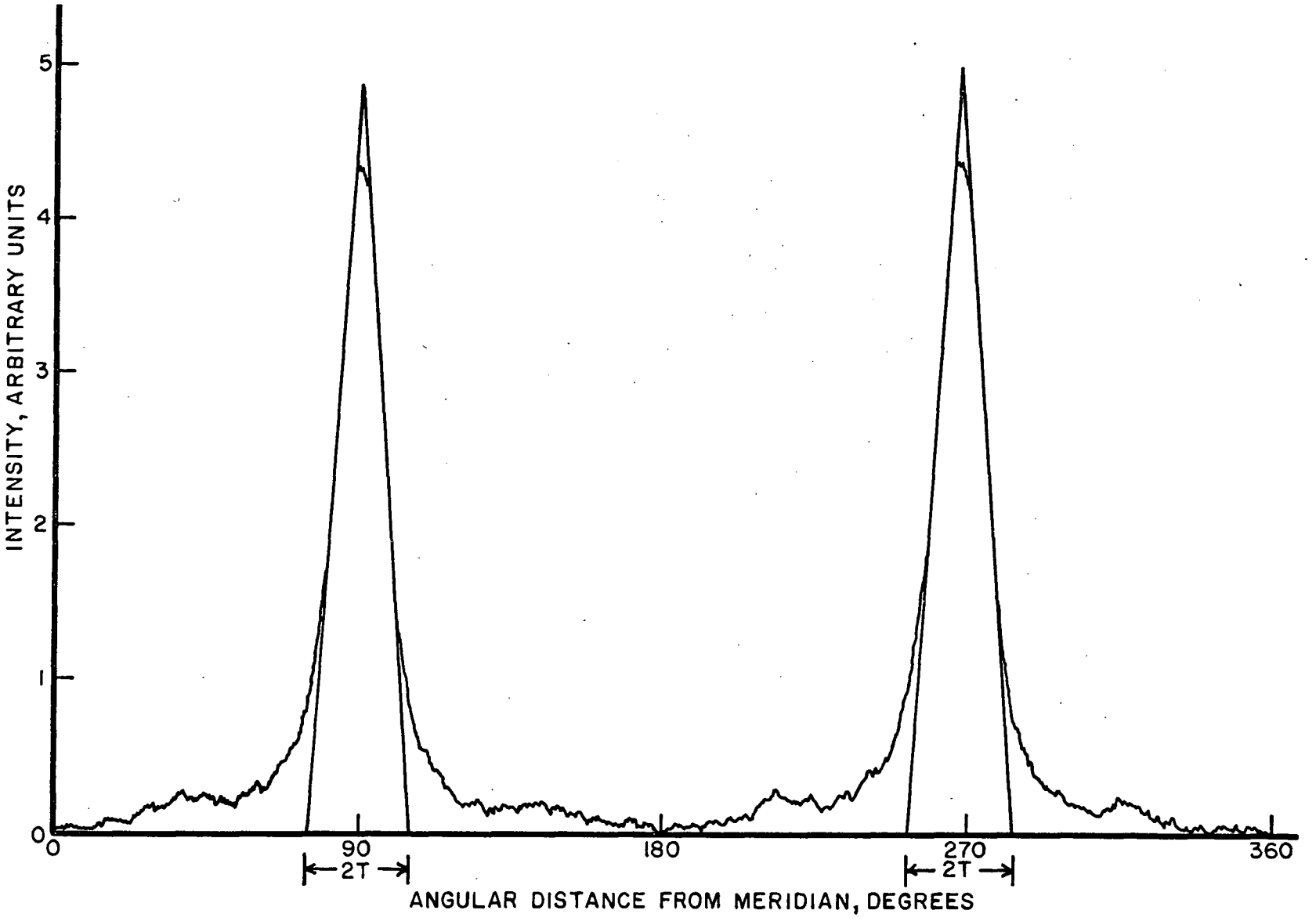


FIG. 17. CAHN ELECTRO-BALANCE USED FOR SPECIMEN WEIGHT DETERMINATION.



FIG·18· X-RAY INTENSITY AROUND THE (002) ARC FOR SITKA SPRUCE LATEWOOD USING TEXTURE GONIOMETER MACHINE·

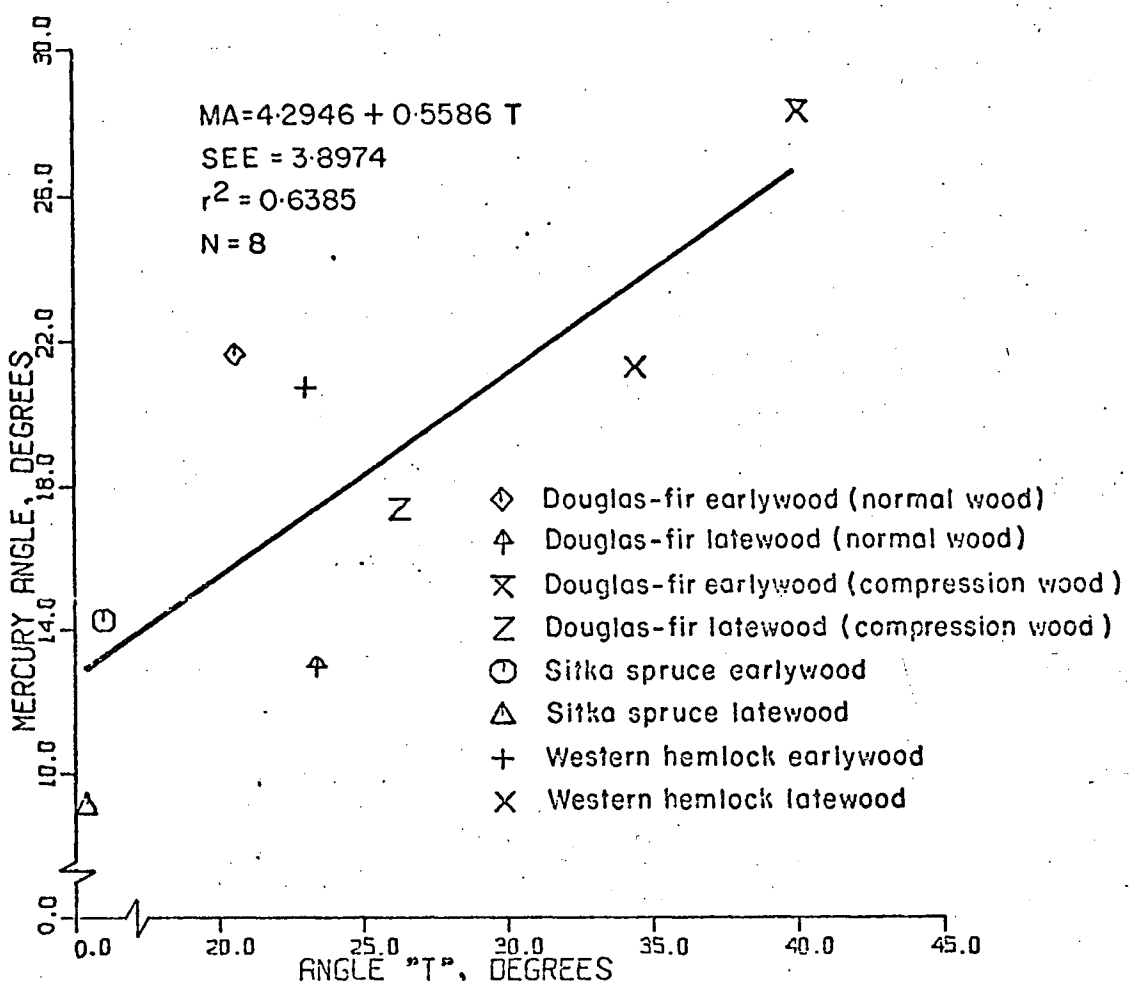


FIG. 19. RELATIONSHIP BETWEEN ANGLE "T" DERIVED FROM X-RAY AND MEAN MICROFIBRIL ANGLE MEASURED BY MERCURY IMPREGNATION (MA).

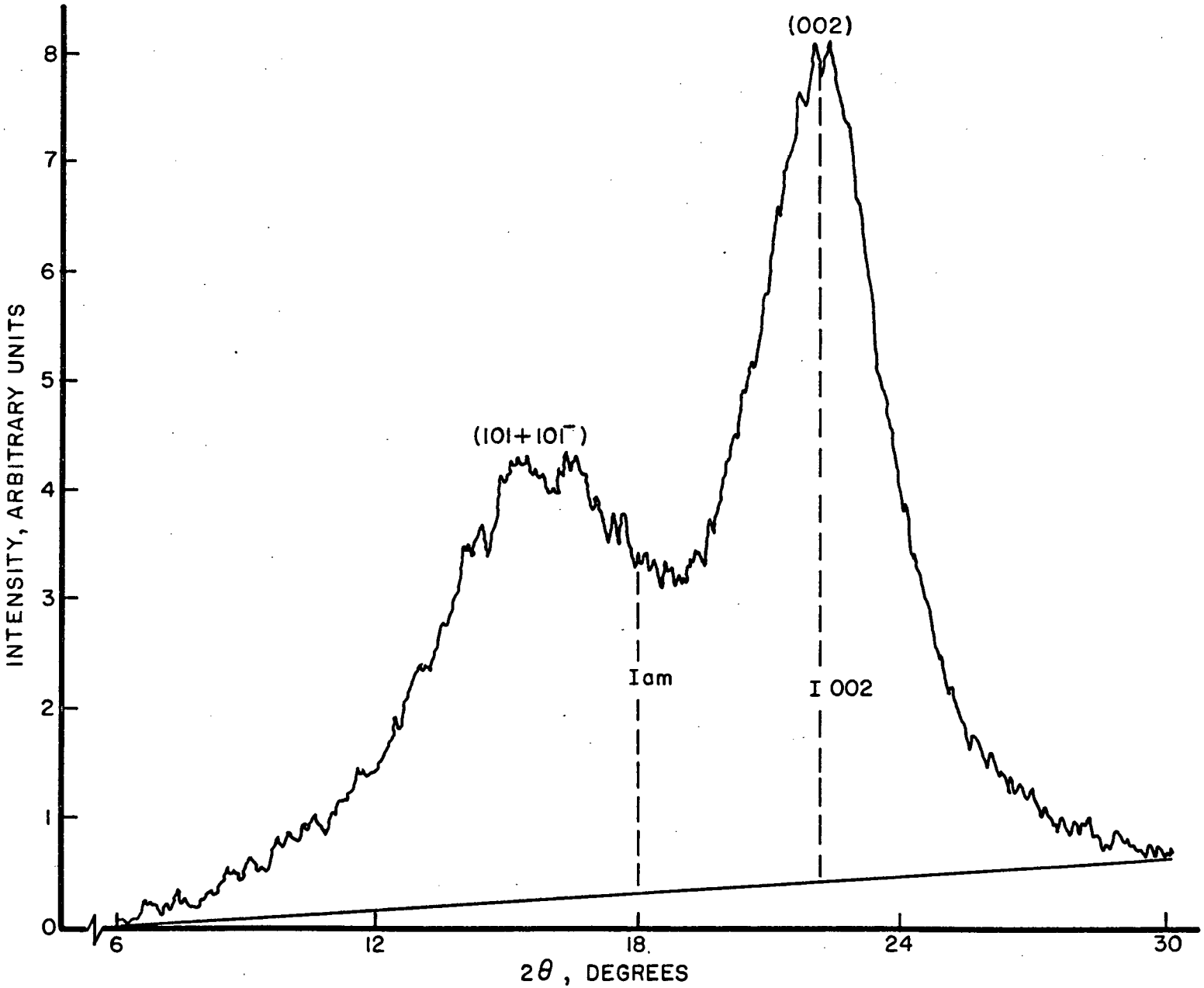
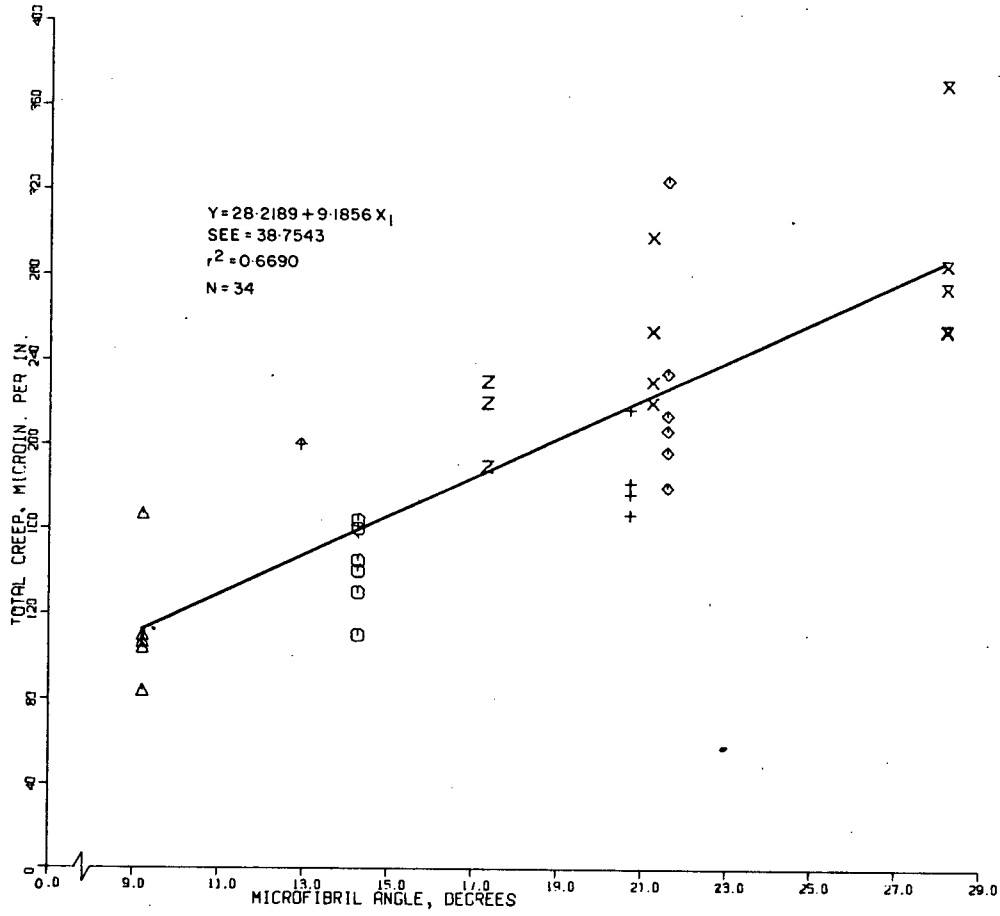


FIG. 20. X-RAY DIFFRACTION PATTERN OF DOUGLAS-FIR LATEWOOD (COMPRESSION WOOD).

FIG. 21. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND MICROFIBRIL ANGLE (X_1) AT 3,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.

FIG. 22. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND MICROFIBRIL ANGLE (X_1) AT 6,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.


Y=28·2189+9·1856 X1
SEE=38·7543
r2=0·6690
N=34
TOTAL CREEP, MICROIN. PER IN.
MICROFIBRIL ANGLE, DEGREES

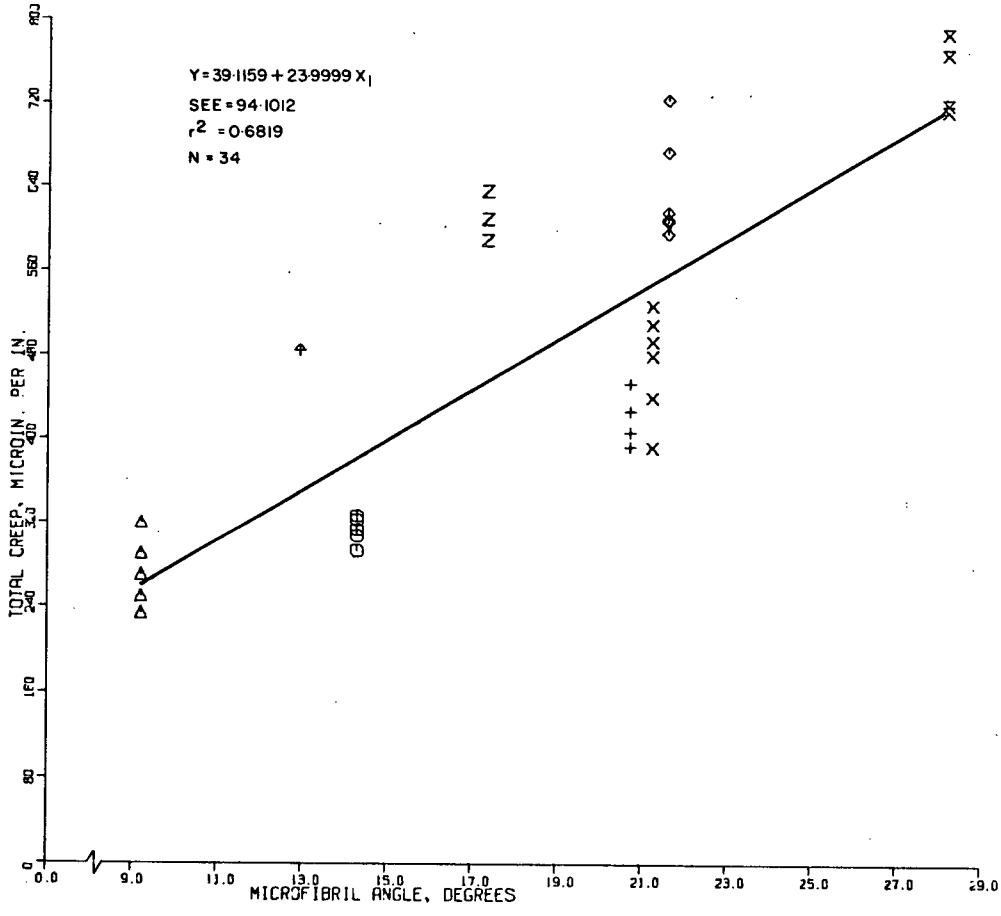
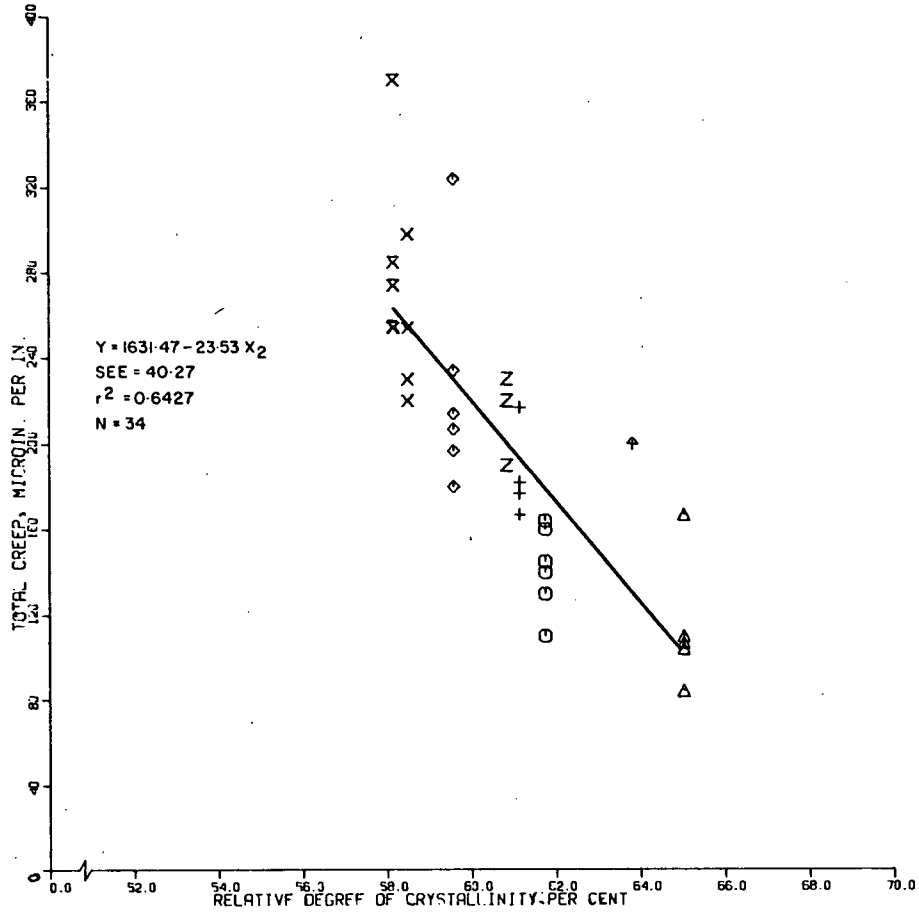
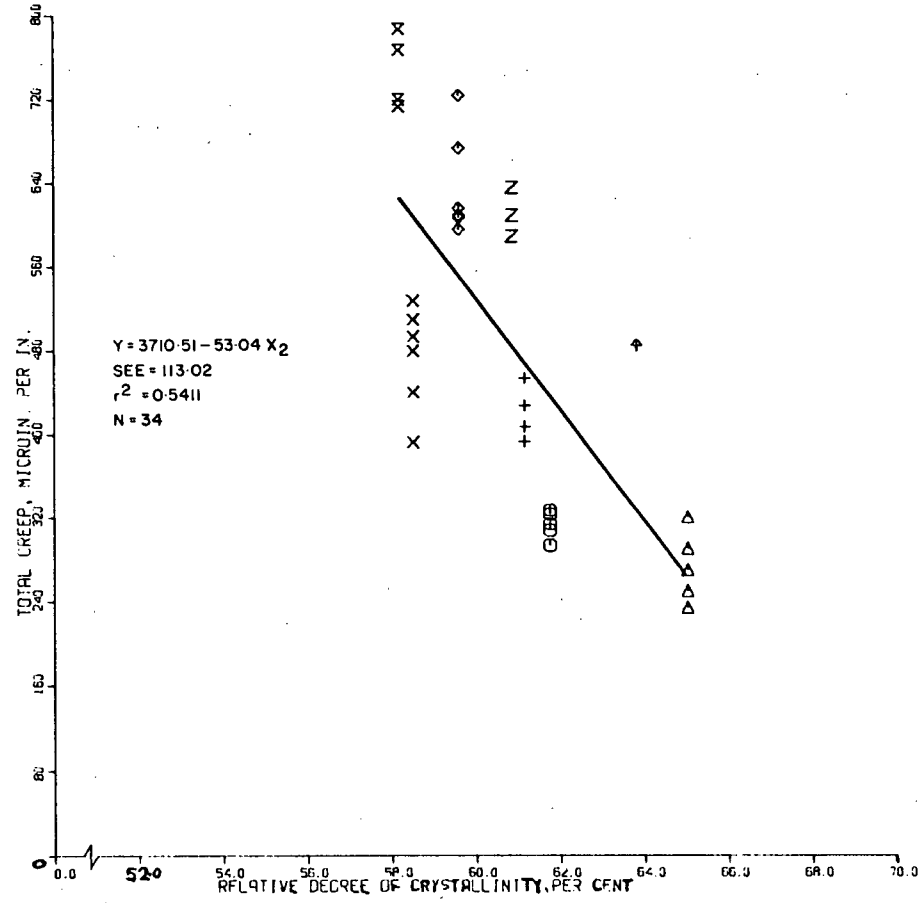

Y=39·1159+23·9999 X1
SEE=94·1012
r2=0·6819
N=34
TOTAL CREEP, MICROIN. PER IN.
MICROFIBRIL ANGLE, DEGREES

FIG. 23. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND RELATIVE DEGREE OF CRYSTALLINITY (X_2) AT 3,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.

FIG. 24. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND RELATIVE DEGREE OF CRYSTALLINITY (X_2) AT 6,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.


Y = 1631·47 - 23·53 X2
SEE = 40·27
r2 = 0·6427
N = 34
TOTAL CREEP, MICROIN. PER IN.
RELATIVE DEGREE OF CRYSTALLINITY, PER CENT


Y = 3710·51 - 53·04 X2
SEE = 113·02
r2 = 0·5411
N = 34
TOTAL CREEP, MICRUIN. PER IN.
RELATIVE DEGREE OF CRYSTALLINITY, PER CENT

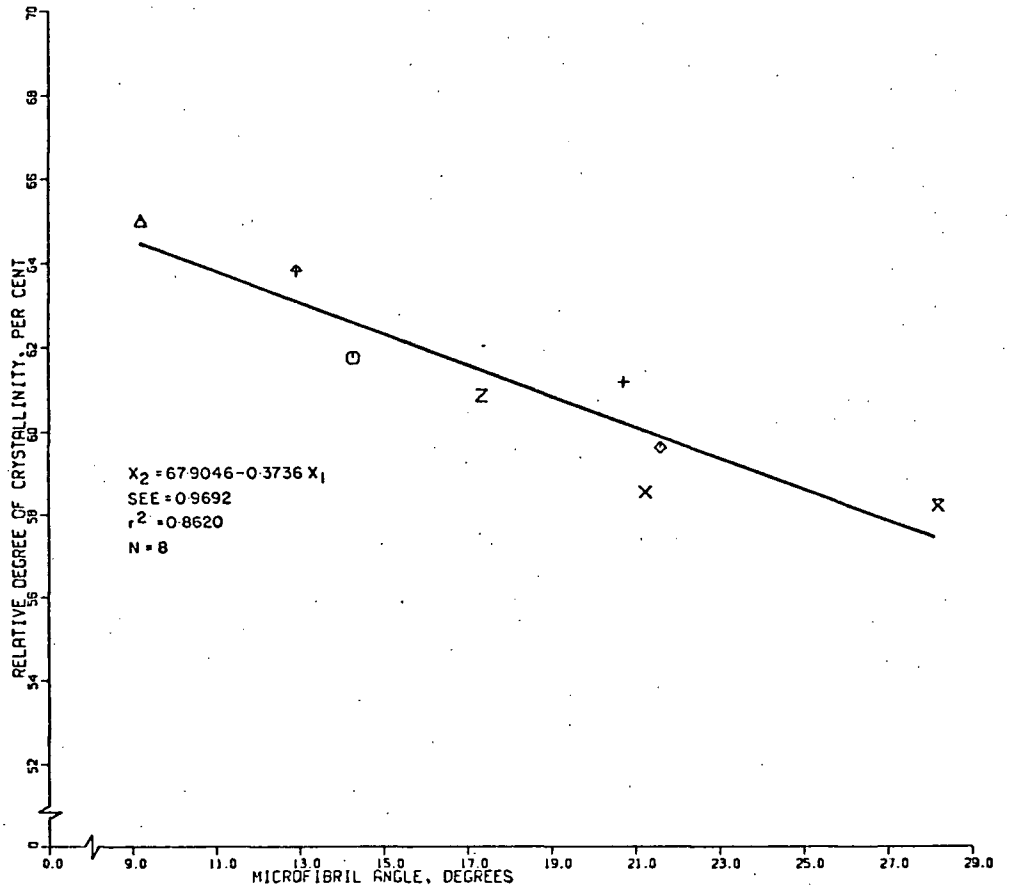
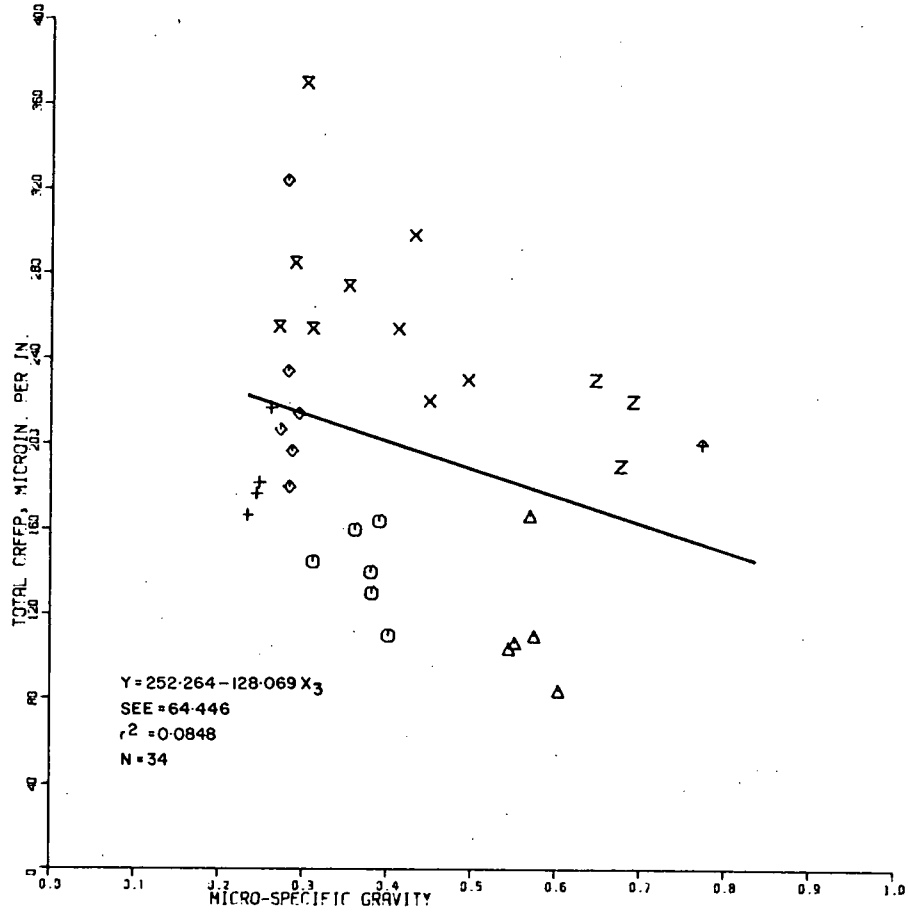


FIG. 25. RELATIONSHIP BETWEEN MICROFIBRIL ANGLE (X_1) AND RELATIVE DEGREE OF CRYSTALLINITY (X_2). SEE FIG. 19 FOR LEGEND.

FIG. 26. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND SPECIFIC GRAVITY (X_3) AT 3,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.

FIG. 27. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND SPECIFIC GRAVITY (X_3) AT 6,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.


TOTAL CREEP, MICROIN. PER IN.
MICRO-SPECIFIC GRAVITY
Y = 252·264 − 128·069 X3
SEE = 64·446
r2 = 0·0848
N = 34

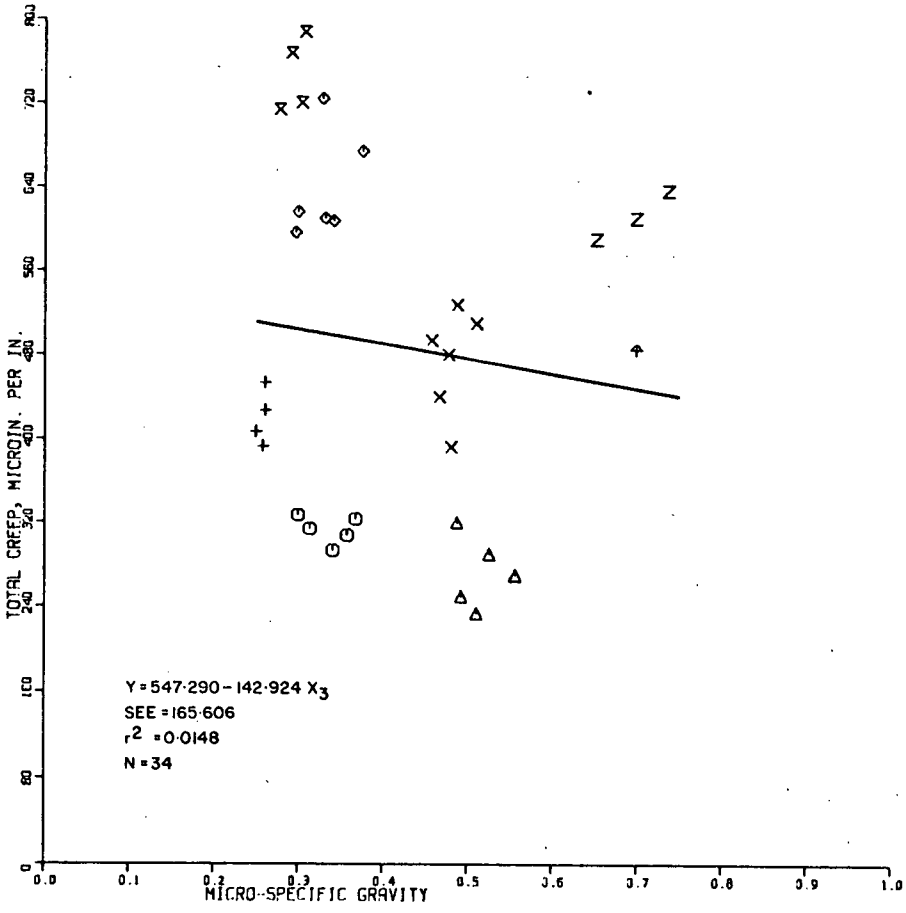
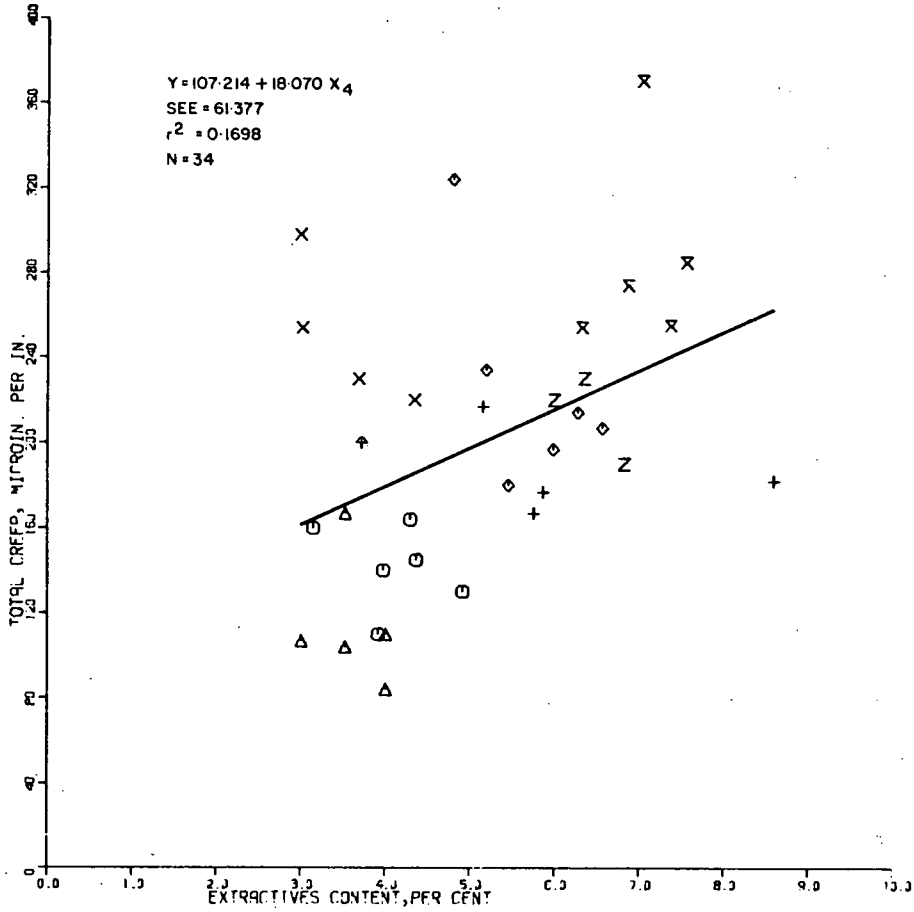
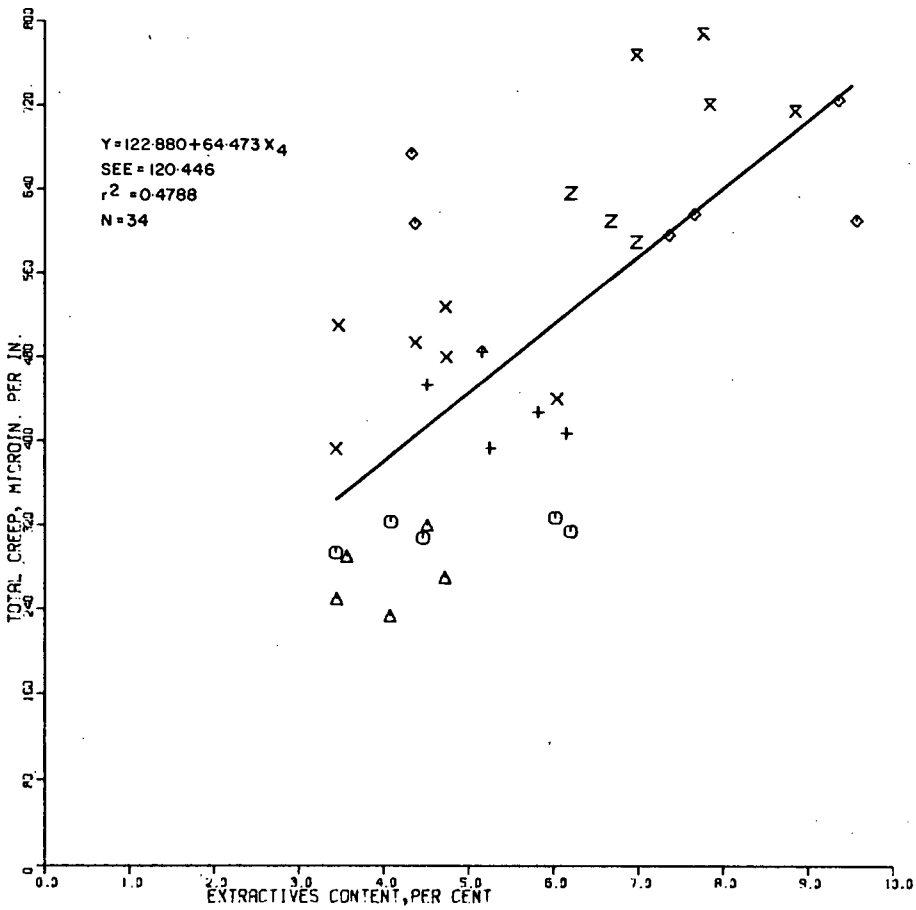

TOTAL CREEP, MICROIN. PER IN.
MICRO-SPECIFIC GRAVITY
Y = 547·290 − 142·924 X3
SEE = 165·606
r2 = 0·0148
N = 34

FIG. 28. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND EXTRACTIVES CONTENT (X_4) AT 3,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.

FIG. 29. RELATIONSHIP BETWEEN TOTAL CREEP (Y) AND EXTRACTIVES CONTENT (X_4) AT 6,000 MICROIN. PER IN. INITIAL STRAIN. SEE FIG. 19 FOR LEGEND.


Y = 107·214 + 18·070 X4
SEE = 61·377
r2 = 0·1698
N = 34
TOTAL CREEP, MICROIN. PER IN.
EXTRACTIVES CONTENT, PER CENT


Y = 122·880 + 64·473 X4
SEE = 120·446
r2 = 0·4788
N = 34
TOTAL CREEP, MICROIN. PER IN.
EXTRACTIVES CONTENT, PER CENT