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## RHEOLOGY OF PROTEINS

A SYMPOSIUM on the "Rheology of Proteins" was held in London on January 20 by the British Society of Rheology. Four papers were presented, each of which aroused considerable discussion. The chairman, Mr. A. G. Ward (British Gelatine and Glue Research Association), in his opening remarks, outlined the wide scope covered by the title of the symposium. Proteins, the mechanical properties of which play a vital part in their functioning in living organisms, include serum proteins, epidermal and connective tissue proteins, muscle proteins, many cellular proteins, mucoproteins and mucopolysaccharides, etc. The rheological properties of certain of these also contribute to their subsequent use in meat, egg and other protein foods, in protein fibres, in gelatin and animal glue, and in other products. In the background, for all these substances, rest the two common problems of the covalently bonded structure of the protein molecules and the secondary forces—ionic, hydrogen bonding, and van der Waals interactions—which link the molecules together to form macroscopic aggregates. Only by isolating the simplest systems will it be possible to solve the many complex problems.

Dr. E. J. Ambrose (Chester Beatty Research Institute), in his paper on the formation of structure in protein solutions, was concerned with the conditions in which a two-phase system can be formed, the protein or related component being organized or ordered in the concentrated phase. The properties of a macromolecule determining its tendency to form such structures are particle size and flexibility, the latter influencing the disturbance by Brownian movement, shape, the interaction forces, and the degree of reversibility of the changes. If the interaction forces are strong, there is a greater tendency to a disordered separation of the second phase, whereas weak forces suffice to make the necessary links only when a degree of order is attained. These considerations were illustrated by the structures obtainable with *poly-γ-benzyl-L-glutamate* in chloroform solution, and with insulin. Birefringent structures show orientation to be occurring when phase separation occurs.

Primary interest is on the behaviour of the cell nucleus and its component nucleo-proteins. Whereas nucleic acid alone fails to give phase separation, nucleoprotein forms fibrous structures, showing

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birefringence and dichroism. The existence of different structures at different concentrations for nucleo-proteins is shown by a discontinuity in the relation between concentration and diffusion constants. No discontinuity is observed for the nucleic acid alone. The techniques and theories have been applied to the structure of chromosomes and also to the formation of the tail of the sperm during meiotic division.

In the second paper, Dr. A. Wassermann (University College, London) described his work on a viscosimetric investigation into the influence of pH and ionic strength on molecular shape of a structural muscle protein. Two views have been put forward concerning the actomyosin molecule, the first regarding it as a stiff rod or elongated ellipsoid of revolution and the second treating it as a flexible random coil. Use of the mixed-bed ion-exchange resin column enables actomyosin solution of very low salt content to be prepared. Alkali additions give solutions, the 'intrinsic viscosities' of which extrapolated to zero rate of shear, and to zero concentration, are a maximum in the region of pH 8-9. At higher pH, lower values are obtained. No peak is observed on the acid side. The absence of change below pH 4 is assumed to arise from anion binding neutralizing the charges developed. The peak is considered to arise from the stretching out of the molecule by the repulsion of negative charges present at the pH's concerned. Beyond a certain pH the concentration of counter ions becomes sufficient to reduce the forces between the charges, so that a reduction in 'intrinsic viscosity' occurs; similarly, the addition of neutral salt in sufficient amount reduces the changes in intrinsic viscosity to zero. All these observations are fully compatible with the random coil hypothesis but are inconsistent with the stiff rod theory.

In the third paper at the symposium, on some rubber-like and relaxation properties of gelatin, Mr. D. W. Jopling (Kodak, Ltd.) demonstrated that gelatin films maintained at a water content of 25-30 per cent by immersion in 85/15 alcohol/water or by exposure to high humidity show rubber-like extensibilities of up to several hundred per cent. This is best shown when the ordered arrangement, brought about by drying, has been destroyed by immersion in boiling methanol.

By cross-linking gelatin molecules with formaldehyde, measurements on the resultant film can be carried out at temperatures at which normal gelling forces are absent, for example, 60° C. Stress relaxation then follows the Maxwell relation  $\log(\text{stress}) = A - B(\text{time})$ , whereas at room temperature stress is

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proportional to log (time). It is assumed that at 25° C. the mechanism is primarily the release of secondary bonds, with a wide spectrum of relaxation times, whereas at 60° C. only primary bonds are broken. At 40° C. both processes can be observed to occur in succession. The form of the extension-tensile stress relation for gelatin cross-linked with formaldehyde at 60° C. follows the theoretical curve for an ideal rubber-like system up to extension ratios of 4:1. The values for the chain molecular weight between cross-links deduced from the theory appear high; the reasons for this are not clear.

The final paper, by Dr. J. Brooks and Mr. H. P. Hale (Low Temperature Research Station), on the mechanical properties of the ovomucin gel of the egg, was given by Dr. Brooks. The thick white fraction of the hen's egg consists of a gel in which a transparent phase is separated by a series of parallel bands of microscopic fibres. After the egg is laid, the volume of thick white decreases at a rate depending on the temperature. The mechanical properties of the gel have been investigated by a method originally devised by Freundlich, in which the displacement of a minute nickel particle inserted in the gel is measured microscopically. The force is applied by means of a magnetic field. For stresses of short duration, the displacements are elastic in character in both the transparent phase and the bands. The bands are anisotropic. The relative values of the rigidities are converted into absolute units by means of a series of measurements on gelatin gels. Measurements have been made on fresh eggs and on eggs stored at 25° and 0° C., and it has been found that there is a close relation between the rigidity of the thick white fraction and its volume, irrespective of the reason for the volume difference.

A. G. WARD

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# THE BRITISH SOCIETY OF RHEOLOGY

Bulletin No. 48

September, 1956

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## Future Meetings of the Society.

*September Conference and Annual General Meeting at Sheffield University.* The date is September 27th and 28th and the subject will be Extrusion and Rolling Processes. Some summaries of papers to be read are given in this bulletin.

There will be a visit to the B.I.S.R.A. laboratories. The Annual General Meeting will be held during the Conference. Further information will be given separately.

*Rheological Control of Industrial Processes.* This Meeting will be held in London, December 7th.

## News of Members.

Mr. D. Fitzgerald (Nat. Coal Board) has been awarded the eighth "Arthur Duckham" fellowship by the Institution of Gas Engineers. The fellowship is tenable at the Central Laboratory of the Dutch State Mines at Geleen, and his work will be under the guidance of Prof. van Krevelen, Director of the laboratory.

Mr. G. H. Tattersall has recently joined the British Iron and Steel Research Association as a leader of the Wire Drawing Section.

## New Members.

Mr. C. C. Mell, c/o Blundell Spence and Co. Ltd., Sculcoates Lane, Hull. Rheological measurements on paints and thixotrometers.

Mr. R. H. Thomas, 34 Wern Road, Margam, Port Talbot, Glam. Viscoelastic liquids and general theory.

Dr. M. Winogradoff, Bexford Ltd., Research Labs., Brantham, Nr. Manningtree, Essex. Viscoelastic materials and their properties, photoelastic relaxation and flow birefringence in relation to structure, dopes, etc.

## News of Meetings.

*Nederlandse Reologische Vereniging.* The next meeting will be held on 8th November when some technical applications of rheological knowledge will be considered. The proposal on rheological nomenclature and definitions, discussed on May 3rd, will shortly be published. On the afternoon of the May 3rd meeting, Prof. Schultz-Grunow from Aachen (Germany) treated the rheology of concentrated solutions and plastic solids, with the help of dimensional analysis.

*A Course of Lectures on Rheological Techniques*, by R. W. Whorlow, B.SC., A.INST.P., at the Battersea Polytechnic, Physics Dept.

A course of about ten lectures, suitable for physicists and chemists, will be given on Wednesdays, 6.30 to 8.30 p.m., commencing on 10th October.

The emphasis throughout the course will be on experimental methods leading to absolute measurements of stress and strain. Examples of instruments and results will be drawn from a wide range of industries. The syllabus includes rheological behaviour and terminology, experimental methods for evaluating stress-strain-time relations for non-Newtonian flow, extension and compression for non-Hookean solids, factors influencing instrument design, empirical formulae of wide application for rubber, plastics, paints, etc.

Fee for the course, £1 0 0.

*A Course of Lectures on Rheology in the Surface Coating Industries*, by specialists from industry at the Borough Polytechnic.

The opening speaker is Dr. Bowles on Friday, 2nd November.

*A Course of 24-30 Lectures on Introduction to the Mathematical Theory of Elasticity*, at the Acton Technical College, by G. D. Smith, M.Sc.

*A Course of 18 Lectures on Mathematical Theory of Elasticity and Plasticity*, at the Battersea Polytechnic, by D. E. R. Godfrey, M.Sc., Ph.D.

*A Course of 11 Lectures on Theory of Elasticity*, at the Northern Polytechnic, by M. Rothman, M.Sc., Ph.D., A.F.R.Ae.S., M.I.Ae.S.

The editor will be glad to receive from members particulars of any future meetings or courses of lectures of rheological interest.

**Liaison between National Societies of Rheology.** Early in 1956 the Committee discussed the question of liaison between the various National Societies. It was decided that apart from any other arrangements, the British Society would in future send, without charge, copies of all notices and literature published to the Secretaries of all other National Societies of Rheology. This would include the Bulletin of the Society, notices of meetings, reprints of interest to members, and when possible, early information regarding future programmes.

It was suggested that if this procedure could be adopted generally by other societies, it would be a convenient method of keeping all societies in touch with each other's activities. It would also avoid the currency difficulties which arise if each Secretary joins the other National Societies to obtain their literature.

These suggestions were forwarded to the six national societies viz., U.S.A., Japan, Germany (two societies), France and Holland. The first four societies have replied welcoming the scheme and agreeing to include all other National Societies in their mailing list. Circulars regarding future meetings have also been received from Holland.

Any members of the British Society who would like information regarding forthcoming rheological meetings in other countries may apply to the Secretary for such information.

### **Books on Rheology.**

Following a suggestion by a member, a selected list of books of general interest has been compiled by the committee. It is hoped that the list, mainly of publications in the English language, may be of help to new students and for reference purposes.

Acad. Sci., Amsterdam.

*First Report on Viscosity and Plasticity*. North Holland Pub. Co., Amsterdam, 2nd Editions, 1939.

Acad. Sci., Amsterdam.

*Second Report on Viscosity and Plasticity*, North Holland Pub. Co., Amsterdam, 1938.

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*High Polymers—Vol. VI, Mechanical Behaviour*, Interscience, N.Y., 1948.

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*Viscosity and Plasticity*, Heffer and Sons, 1947.

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*The Mechanical Properties of Cheese and Butter*, Dairy Inds. Ltd., London, 1952.

Bingham, E. C.

*Fluidity and Plasticity*, McGraw-Hill Book Co., 1922.

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*An Introduction to Industrial Rheology*, J. and A. Churchill Ltd., 1938.
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*Rheology in Relation to Pharmacy and Medicine*, Pharmaceutical Press, London, 1948.
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*Essays in Rheology (Oxford Conference, 1944)*, Sir Isaac Pitman and Sons Ltd., London, 1947.
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*The Principles of Rheological Measurement (London Conference, 1946)*, Thomas Nelson and Sons Ltd., 1949.
- British Rheologists' Club.  
*Some Recent Developments in Rheology*, United Trade Press, 1950.
- British Rheologist's Club and Textile Institute.  
*Leeds Conference, 1948, Journal of the Textile Institute* **39**, No. 7, July 1948.
- British Rheologists' Club and Institute of Petroleum.  
*London Conference, 1945, Journal of the Institute of Petroleum* **31**, August, 1945.
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*Kinetic Theory of Liquids*, Clarendon Press, Oxford, 1946.
- Frey-Wyssling, A. (Ed.)  
*Deformation and Flow in Biological Systems*, North Holland Publishing Co., Amsterdam, 1952.
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*Linear Polymers*, Longmans Green and Co., 1951
- Green, A. E. and Zerna, W.  
*Theoretical Elasticity*, Clarendon Press, Oxford, 1954.
- Green, H. S.  
*The Molecular Theory of Liquids*, North Holland Pub. Co., Amsterdam, 1952.
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*Industrial Rheology and Rheological Structures*, John Wiley and Sons, Inc., N.Y., Chapman and Hall Ltd., London, 1949.
- Gross, B.  
*Mathematical Structure of the Theories of Viscoelasticity*, Hermann et Cie, Paris, 1953.
- Haward, R. N.  
*Strength of Plastics and Glass*, Cleaver-Hume Press, 1949.
- Hermans, J. J. (Ed.).  
*Flow Properties of Disperse Systems*, North Holland Pub. Co., Amsterdam, 1953.
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*The Physics of Rubber Elasticity*, Clarendon Press, Oxford, 1949.
- Proceedings of the First International Congress on Rheology*, Schwenningen, Holland, 1948, North Holland Publishing Co.
- Proceedings of the Second International Congress on Rheology* (Ed. Harrison), Oxford, 1953, Butterworths Scientific Publications.

## EXTRUSION AND ROLLING PROCESSES—SUMMARIES OF PAPERS

to be read at Sheffield University, September 27th and 28th

### Temperatures Developed in Extrusion.

By M. T. WATKINS

The importance of temperature has been stressed but little attention has been given to the measurement of the temperature attained within the material during the process.

In considering the factors governing the quality of extrusions Smith and Swindells<sup>1</sup> advocated the lowest temperature consistent with the type of alloy, the extrusion ratio and the press capacity. The significant extrusion temperature was that of the section as it emerged from the die and this was frequently higher than the temperature of the billet placed in the container. Pearson<sup>2</sup> stressed the importance of limiting the higher extrusion temperatures, which depending on the metal, the nature of the deforming process and the rate of deformation, could lead to intergranular disintegration or hot shortness of the product.

Several methods have been suggested for measuring the temperature of the extruded product, the commonest being based on the liquid or air calorimeter<sup>3</sup> and on radiation pyrometer methods. An approximate method for calculating the temperatures reached in the cold extrusion of metals has been given by Bishop<sup>4</sup> but this is applicable to plane strain conditions only. The present paper describes an apparatus and instrumentation for the measurement of the extrusion pressure and of the temperature distribution within the metal being extruded and to determine the effect on these quantities of the method of extrusion, slug dimensions, punch speeds, reduction in cross-sectional area, tool profile and lubrication.

Suitable sub presses were developed for the direct and inverted extrusion of rod and Hooker tube and for the inverted extrusion of tubular containers. Solid cylindrical slugs were used for rod and tubular products and axially pierced slugs for Hooker tubes. The sub press was set up for the appropriate extrusion and mounted on a 150 ton up-stroking hydraulic press. The extrusion load was measured by a parallel plate variable capacity type gauge and the punch stroke by a concentric cylinder variable capacity type gauge mounted on the press.

The load and displacement gauges were designed to function with appropriate frequency modulated pre-amplifiers of a six-channel cathode ray recording equipment. The temperature within the slug during the extrusion was measured by insulated thermocouple wires protected by a high tensile steel sheath. The emfs developed in up to four thermocouples were fed to high gain direct coupled amplifiers. The outputs from the various first stage amplifiers were fed through normal driven amplifiers to the cathode ray tubes. A record was then obtained on 70 mm. film giving the complete load, displacement, temperature time characteristic of the particular extrusion.

Rods, Hooker tubes and tubular containers of Chemical Lead Grade A, Aluminium 99.5% purity, Copper E.R.H.C. and Brass 70/30 were extruded at room temperature at punch speeds varying from 1 inch to 30 inches per minute. All slugs excepting those used in the study of slug dimensions were of diameters 1 and 2 in. and of heights 1 and 2 in. respectively.

The major factors contributing to the rise in temperature inside the slug during extrusion were the reduction and punch speed, the latter presumably governing the amount of heat lost by conduction through the die, the chamber and the punch.

The highest recorded temperatures for the extrusion of lead and aluminium rod at 99.4% reduction have been 180°C and 280°C respectively, the thermocouples being located  $\frac{1}{8}$  inch inside the slug and remaining unbroken over 0.95 inch stroke. In the extrusion of lead rod at 99.4% reduction an increase in punch speed from 1 inch to

5.5 inches per minute resulted in an increase in the measured temperature from 90°C to 170°C. In the extrusion of aluminium rod at 94% reduction a similar increase in speed resulted in an increase in temperature from 65°C to 160°C. The thermocouples were located  $\frac{1}{8}$  inch inside the slugs as before.

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## Some Applications of Rheology in the Investigation and Practice of Polymer Extrusion.

By W. F. O. POLLETT (Henley's Research Labs.).

The screw extruder is extensively used in the electric cable industry for the application of dielectrics and protective coverings, composed essentially of high polymers. The theory of the operation of the screw in conjunction with viscous materials is well established qualitatively, but quantitative relationships have generally been based on Newtonian behaviour, which does not correspond even approximately to the rheological properties of the rubbers and thermoplastics used for cable covering. The Ostwald de Waele relationship provides a somewhat better approximation within certain limits of screw design.

Much can be learnt about the qualitative aspects of flow in an extruder by the familiar method in which materials of different colours are used to map out the flow paths. The information obtained in this way has been supplemented by two complementary techniques. It is well known that strains in many high polymers may be "frozen in" by a reduction in temperature and elastic recovery can subsequently occur if the temperature is again raised. The recoverable strains produced in plasticised polyvinyl chloride during its passage through an extruder can be stabilised in this way after the machine has been shut down, and their distribution studied by the reheating of suitable sections taken from various positions. The results provide corroborative evidence of the nature of the deformations taking place in the extruder. Further, it has been shown that the temperature at which recovery occurs depends upon that at which the p.v.c. was deformed. This observation can be turned to advantage when it is desired to investigate the existence of temperature gradients in the polymer. It also affords a method of removing strains, introduced accidentally when the partially cooled p.v.c. is removed from the extruder, without serious interference with those it is desired to measure. Again, the phenomenon may sometimes be used to distinguish between deformations produced at different stages of the process. For example it is possible to decide whether an eccentric coating originated at the die, or whether it was caused by pressure exerted at the haul-off capstan. The preceding methods have greatly assisted the correct design of the T-heads normally used for the application of extruded wire and cable coverings. They have shown that when the material emerges from the screw channel it converges towards the axis of the barrel, in consequence of the Weissenberg effect caused by the shearing action of the end of the screw. Unless the boundaries are suitably shaped, this will result in stagnant zones being left, which may give rise to decomposition of the polymer, or to premature curing in the case of vulcanisable compositions. Similarly the shearing in the die produces a longitudinal tension, which can be reduced by a high working temperature, but cannot be entirely eliminated.

When the conditions at the die are such as to favour the formation of high recoverable strains, there is a tendency for the extrudate to exhibit a characteristic type of surface roughness. This is probably caused by the stretching, which the surface layers suffer, as the velocity gradient existing in the die is dissipated in the vicinity of the die mouth. Often this effect can be avoided by an increase in temperature, but this is not always practicable. Rheometer tests have shown that the recoverable strain in polymer melts, at a given rate of shear, can be materially reduced by applied shears in the order of  $10^3$  to  $10^4$  and that the effect increases with the rate of shear. Under factory extrusion conditions the rate of shear at the die surface may amount to  $10^4$  or even  $10^5 \text{ sec.}^{-1}$  and a die designed so that this is maintained for a sufficient interval, sometimes permits surface markings to be avoided when a simple increase in temperature fails to provide a solution.

## The Extrusion of Metals.

By PAUL FELTHAM (The University, Leeds).

To arrive at optimum conditions in the extrusion of metals, particularly on the industrial scale, it is desirable to base the determination of the extrusion variables and the principal features of the design of presses on a theoretical basis, which should indicate the relative significance and the mutual relation between such factors as ram-pressure, extrusion-temperature, ram-velocity, the purity of the metal, and the geometry of the billet and the die. Although relations between some of these variables have been studied by a number of workers, and discussed in the standard works on the subject, the theoretical evaluation of extrusion pressures is still at best semi-empirical and generally unreliable.

The principal object of the present work was therefore to develop from first principles simple relations for determining extrusion pressures from the given variables, i.e. the extrusion-temperature, the ram-velocity, the extrusion-ratio, and the relevant rheological properties of the metal without the introduction of empirical relations, yet with sufficient accuracy to render them useful in practice.

In the direct extrusion process work done by the ram is expended on "useful" plastic work in reducing the dimension of the metal from  $A_0$  to  $A$ , where  $A_0$  is the cross sectional area of the billet and  $A$  that of the extruded bar. Work is "wasted" due to friction at the billet/container interface, as well as on such plastic work which does not yield a final plastic deformation, i.e. a deformation is first introduced and subsequently reversed, so that work is done but no deformation results from it. Examination of the slip surfaces of metals which are undergoing extrusion shows the flow to be essentially streamlined, and effects due to "useless" plastic work of the latter type are neglected in the theory developed.

If then the metal is regarded as being fully plastic, and fully work-hardened on approaching the die orifice, on account of the large strains to which it is subject in the zone close to the die, then the maximum extrusion pressure is shown to be

$$\sigma_{\max} = Y_d [(\ln(A/A_0)) \cdot \exp. (4\mu L'/D)]$$

where  $Y_d$  is the tensile stress yield of the fully work-hardened material at the extrusion temperature,  $\mu$  is the effective coefficient of friction at the billet/container interface (equal to approximately 0.05 with unlubricated surfaces),  $L'$  is a length slightly smaller than the billet length, and  $D$  is the billet diameter. The actual die is generally a simple hole in the container, but a virtual die is formed by the metal itself during extrusion. Generally this takes the form of a frustum of a right cone the generators of which are tangents to the actual die (here assumed circular for simplicity). By the use of this fact it is possible to show that the strain rate in the "virtual die" is approximately equal to

$$d\varepsilon/dt \sim (3v/D) \ln (A_0/A),$$

where  $v$  is the ram-velocity. Since, at the high temperatures at which extrusion generally takes place,  $Y_d$  depends significantly upon the strain-rate and the temperature, it is possible to obtain this important variable from high-temperature rheological data for metals subject to rapid flow. Such data were available for pure copper, aluminium, iron, as

well as for mild steel. For metals of commercial purity (assumed to be 99.5% or somewhat less) the values of  $Y_d$  were taken to be twice those given by the data for the respective pure metals under similar extrusion conditions, to comply with general experience. Extrusion forces (i.e. the maximum required total force acting on the ram) were calculated from the theory for aluminium (99.5%), copper (99.97%, as well as of technical purity), and lead (99.9%). A wide range of extrusion-temperatures, extrusion-ratios, ram-velocities, and billet dimensions were used; the experimental data being taken from measurements reported in the literature. Very good agreement was obtained in all cases.

The maximum extrusion force  $F_{\max} = A_0 \sigma_{\max}$  has a minimum for a given value of  $A$ , and a fixed volume  $V$  of the billet. From the condition  $dF_{\max}/dD = 0$  an optimum value of the billet diameter  $D$ , can be determined. Such a determination may be particularly useful when using a press near its maximum capacity.

At very high temperatures (e.g. above  $0.85T$  melt on the absolute scale), if a high extrusion-ratio is used, the back shear stress at the billet surface due to friction may exceed the shear stress of the metal of which the billet is made. In that case, if  $Y_b$  is the tensile yield stress of the billet in the surface region, and  $m = Y_b/Y_d$ , one obtains for the maximum extrusion stress

$$\sigma_{\max} = Y_d [\ln(A_0/A) + (2mL'/D)].$$

A "skin" of metal is then generally found to adhere to the container wall after extrusion. To find  $Y_b$  the strain-rate at the billet/container interface must be evaluated. This is given, approximately, by  $\ln[(h+v)/h]$ , where  $h$  is the thickness of the "skin." In most cases occurring in practice this is of the same order of magnitude as the strain-rate in the die. It is obvious that of the two expressions for  $\sigma_{\max}$  the one giving the lower value will apply in any particular application.

## The Use of Optical Methods in the Manufacture of Plastic Sheet

By H. H. ALLEN (British Celanese Ltd.).

Optical methods using polarised light are well established in polymer structure and flow research as well as in stress analysis, but are less well known in the field of plastic sheet or film production, whether extruded or cast. Yet optical methods are a powerful tool for obtaining quickly information about rheological conditions of both product and process.

In research, the microscope is the proper instrument but for technical applications the projection method is more suitable (easier handling, larger area to view and easier photographing).

Polymer flow cannot be directly inspected in the die but only in the solidified product. To make deductions from this as to its flow history it must be assumed that the previous flow history is "frozen" in the solid sheet. This is true for most extruded polymers as their relaxation time is considerably longer than the solidification time. Experimental evidence for this will be produced.

*Optical Methods (a) Surface Inspection.* Surface inspection can give useful information on flow history. Visual inspection of the highly reflecting surface of a transparent plastic is difficult. A recently developed method which produces a magnified image of the surface will be demonstrated showing normal and abnormal flow lines, "cold flow" patterns, and the detection of impurities. *(b) Birefringence.* Birefringence is a measure of orientation (anisotropy) the orientation of the sheet being of practical importance as it affects the physical properties of a plastic sheet as well as reflecting flow conditions in the machine. The main cause of orientation are the stresses due to flow in the extruder die or casting box. Birefringence, however, is small for thin films because (a) flow stresses are generally small. (b) birefringence is proportional to thickness and, therefore, is small for thin films and foils. The phase difference is seldom more than one wavelength, and there is therefore no ambiguity about fringe order.

Under these circumstances the best method is to measure light transmission of the specimen between crossed polaroids. The method is briefly described, and results

discussed by means of slides showing graphs of optical anisotropy vs. (a) breaking strength, (b) heat shrinkage, (c) die pressure, (d) die temperature. These demonstrate the correctness of the "frozen flow" theory as well as the effect of orientation on the product. (No attempt is made in this paper to explain correlation between optical anisotropy and physical properties of plastics.) The practical value of the method is the immediate detection of change in operating conditions and insuring a uniform product.

The method also determines the direction of orientation. Divergence between extrusion or casting direction and direction of optical anisotropy indicates flow disturbance. A demonstration is given of a specimen where flow direction in the centre is not parallel to the extrusion direction.

The difficulties arising in some materials such as polyamides due to superposition of crystal orientation on the photoelastic effect are discussed.



# RHEOLOGY ABSTRACTS

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## 3335. Effect of Temperature on the Flow Stress of Work-hardened Copper Crystals.

M. A. ADAMS and A. H. COTTRELL, *Phil. Mag.*, 1955, **46**, 1187.

After correction for the effect of temperature on elastic constants the flow stress proves to be insensitive to temperature from 90°K to 180°K, falls rapidly with increasing temperature from 180°K to 250°K, and is virtually constant from 250°K to 473°K. The change of flow stress with temperature is closely proportional to the flow stress itself. A.

## 3336. The Tensile Properties of Single Crystals of High-purity Iron at Temperatures from 100 to -253°C.

N. P. ALLEN, B. E. HOPKINS and J. E. MCLENNAN. *Proc. Roy. Soc. A*, 1955, **234**, 221.

At temperatures down to -124°C, the crystals were fully ductile, giving chisel-edge fractures and 100% reduction in area. At -196°C, depending on the orientation of the stress axis, the behaviour covered the whole range from fully ductile with 100% reduction in area to completely brittle with cleavage fractures and no apparent deformation. Between these limits, mixtures of slip, twinning and cleavage were obtained. At -253°C, the crystals gave cleavage fractures, and, over most of the orientation range studied, this occurred without prior deformation. It is shown that the resolved shear stresses required to produce slip or twinning at -196°C vary with the orientation. A.

## 3337. Relationship between Small-angle Dislocation Boundaries and Creep.

B. ANCKER, T. H. HAZLETT and E. R. PARKER. *J. appl. Physics*, 1956, **27**, 333.

Small-angle dislocation boundaries of controlled nature (sharp or diffuse) and density were introduced (by prestraining at room temperature and annealing at 800°C) into high-purity polycrystalline nickel prior to creep at 700°C. The initial density of small-angle boundaries ranged from an average of nearly zero per grain, to a large number which was limited by the occurrence of crystallisation. X-ray microscopic examination showed that the dislocation networks developed during creep were nearly identical and were independent of the initial density and nature. A.

## 3338. On the Molecular Nature of Rubber Friction.

G. M. BARTENEV. *Kolloidyn Zh.*, 1956, **18**, 249 ("Discussion").

Coulomb's friction law modified to introduce a "factor of contact" so that friction force  $\propto FS^\phi$ . Curves of  $F$  vs rate of sliding show a Bingham-shaped curve for rubber. For metals, after critical  $F$  is reached,  $F$  falls; and at high velocities, becomes asymptotic to a lower critical value. Various equations are discussed which relate stickiness and coefficient of friction to friction force and loads. G.W.S.B.

## 3339. Plastic Extension Tests on Aluminium and Aldrey Wires for Electrical Conductors.

L. BIONDI and G. PARISIO. *R.C. 55 Riun. Assoc. Elettrotec. Ital., Bellagio*, 1955, **42**, Paper 153.

Numerous tests have been carried out to determine the permanent set under steady loads of wires produced by different processes. The plastic strain  $\epsilon$  follows the empirical law  $\epsilon \propto f^n t^y$  where  $f$  is the stress and  $t$  time. A list of test results is included. A.

**3340. Theory of Propagation of Elastic Waves in a Fluid-saturated Porous Solid.**

M. A. BOLT, *J. Acoust. Soc. Amer.*, 1956, **28**, 168 and 179.

A theory is developed for the propagation of stress waves in a porous elastic solid containing a compressible viscous fluid. The emphasis of the present treatment is on materials where fluid and solid are of comparable densities, as for instance in the case of water-saturated rock. It is found that the material may be described by four non-dimensional parameters and a characteristic frequency. There are two dilatational waves and one rotational wave.

The breakdown of Poiseuille flow beyond the critical frequency is discussed for pores of flat and circular shapes. A.

**3341. The Tensile Strengths of Liquids under Dynamic Loading.**

T. H. BULL, *Phil. Mag.*, 1956, **1**, 153.

A method of measuring the dynamic tensile strengths of liquids, involving the use of an electrical pressure bar, is described. Cavitation phenomena in water subjected to large transient tensile stresses have been observed using a spark shadowgraph technique. Under similar conditions of transient loading the cavitation thresholds of water and of glycerol are approximately  $17 \times 10^6$  dynes/cm<sup>2</sup> and  $63 \times 10^6$  dynes/cm<sup>2</sup> respectively. A.

**3342. On the Internal Viscosity of Chain-like Macromolecules.**

R. CERF, *C.R. Acad. Sci.*, 1955, **241**, 496.

The arguments of a previous paper are carried further by distinguishing between rotation and deformation of a large molecule. Two limiting cases are arrived at, corresponding to completely rigid and non-rigid molecules respectively. A.

**3343. Deformation Characteristics of Hafnium.**

D. S. EPPELSHEIMER and D. S. GOULD, *Nature*, 1956, **177**, 241.

A total deformation by cold rolling of about 30% appears to be the maximum obtainable before cracking occurs, although at elevated temperatures (500°C) no such limit was observed. The compression texture was found to be (0001) rotated 15°–30° from the compression axis. The rolling texture is very similar. The twinning and slip modes which could account for such a texture are noted. A.

**3344. Stress Distribution in a Tee Junction of Thick Pipes.**

H. FESSLER and B. H. LEWIN, *Brit. J. appl. Phys.*, 1956, **7**, 76.

The stress distribution on the inner surfaces of both pipes near their juncture, as caused by internal pressure, was determined by the "frozen stress" photoelastic technique. An approximate method of calculation (based on the theoretical solution for a hole in an infinite plate under uniform tension) was developed and a measure of agreement between the calculated and measured stresses was found. A.

**3345. Temperature Dependence of Viscosity and its Relation to Vapor Pressure for Associated Liquids.**

L. A. GIRIFALCO, *J. Chem. Phys.*, 1955, **23**, 2446.

A theoretical equation for the relation between temperature and viscosity is derived on the assumption that a molecule possesses two types of force centres one of which involves dipole interactions and the other dispersion forces. Experimental data for water and ethyl alcohol (both associated liquids) are satisfactorily accounted for. A.

**3346. The Tearing of Aluminium Foils.**

J. HENNEPHOF, *J. of Mech. and Physics of Solids*, 1956, **4**, 172.

The energy for tearing a unit length out of the foil consists (a) energy for bending the foil and (b) the energy for the plastic deformation of the borders of the tear. The first quantity is the main part of the total tearing energy. E.W.J.M.

**3347. The Activation Energies of Viscous Flow and Self-diffusion in Molten Metals.**

A. HRBEK, *Hutnické Listy*, 1956, **11**(3), 164. In Czech.

Experimental data and theories relating to self-diffusion and viscosity in liquid metals are surveyed. The activation energies for both processes are evaluated from data giving the temperature dependence of the viscosity in the case of Na, Al, K, Fe, Cu, Zn, Ag, Cd, Sn, Sb, Au, Hg, Pb and Bi. The equation  $Q = C_v \cdot T_m$ , relating the activation energy  $Q$ , identical for both processes, to the specific heat at constant volume and the melting temperature, is derived. This is applicable also to non-metals, e.g. water. P.F.

**3348. Experiments on Viscoelasticity.**

D. J. IVEY. *Amer. J. of Physics*, 1956, **24**, 373.

The propagation of sound waves in rubber strips is used to illustrate viscoelastic behaviour. The experiments introduce time-dependent mechanical properties of solids  
E.W.J.M.

**3349. New Equipment for Measuring the Creep of Refractories at High Temperatures.**

A. JOURDAIN. *Silicates Indust.*, 1956, **21**, 116.

The design and mode of operation of a new creep-testing machine developed by the author are described. A carbon-resistance in the shape of a cylinder is used as furnace; automatic recording of the strain and thermostatic temperature control are used. The tests are carried out in vacuo. Examples of the performance of the equipment are given. P.F.

**3350. Non-Newtonian Viscoelastic Properties of Rod-like Macromolecules in Solution.**

J. G. KIRKWOOD and R. J. PLOCK. *J. Chem. Phys.*, 1956, **24**, 665.

The theory of Kirkwood and Auer is extended to include the dependence of the viscoelastic moduli of solutions of rod-like macromolecules on rate of shear. Power series expansions in the rate of shear of the intrinsic viscosity and intrinsic rigidity are constructed. In the stationary case, the intrinsic viscosity is presented as a function of rate of shear with use of coefficients of the pertinent spherical harmonics. A.

**3351. Regular Laminary Detachment (and Flow of a Viscous Compressible Fluid, traversed by Quasi-Shock Waves, from a Boundary).**

R. LEGENDRE. *C.R.Acad. Sci.*, 1955, **241**, 732.

The flow of a viscous, compressible fluid, traversed by quasi-shock waves, in the neighbourhood of, and its detachment from, an isothermal boundary the specific mass of which depends only upon the pressure in its neighbourhood, is discussed mathematically. The mathematical relation to be satisfied for adherence of a viscous fluid to the boundary of an obstacle in the fluid, is  $\bar{V} = ZF$ , where  $\bar{V}$  denotes the velocity of the fluid,  $Z$  the distance to the boundary or an appropriate regular function of the distance to the boundary, and  $F$  is a regular finite vector or vector function for  $Z=0$ . The gradient of current surface detached from a boundary is three times that of the pressure gradient; in three-dimensional flow this gradient is either very small or nearly thrice that of the plane containing the pressure gradient and tangential to the line of detachment. Detachment cannot be analysed when the pressure gradient is ignored. A.

**3352. Linear Superposition in Viscoelasticity and Theories of Delayed Effects.**

E. R. LOVE. *Australian J. of Physics.*, 1956, **9**, No. 1, 1.

In the formula expressing the stress history as an integral involving the strain history linearly, based directly on experiment, linear superposition is a necessary physical datum but is not sufficient. Necessary and sufficient conditions are established and expressed in physical as well as mathematical terms. E.W.J.M.

**3353. Theory and Experimental Verification of the Oscillating disk Method for Viscosity Measurements in Fluids.**

P. MARIENS and O. VAN PAEMEL. *Appl. Sci. Res.*, 1956, **A5**, 411.

A relation between the damping of a cylinder, suspended from a torsion wire and oscillating around its axis in an infinite space, filled with a viscous fluid, and the co-efficient of viscosity of this fluid has been deduced. The theoretical result has been checked by measurements in water, diethylether and air. P.G.M.

**3354. The Wall Effect and the Viscometry of Suspensions.**

A. D. MAUDE and R. L. WHITMORE. *Brit. J. appl. Phys.*, 1956, **7**, 98.

When the viscosity of a suspension of spherical particles uniformly distributed in a simple liquid is measured in a capillary tube viscometer, a wall effect, which is not found with coaxial rotating cylinder viscometers, is observed. The paper suggests that it may be caused by the redistribution of a portion of the suspension particles on entering the tube of the viscometer, leading to a reduction in both the concentration and viscosity of the suspension near to the walls of the tube. The diminution in concentration is measured experimentally and found to yield a value for the wall effect which is in agreement with that obtained from viscosity measurements. The reason for the redistribution of the particles at the tube inlet is discussed. A.

**3355. Rheology of Wood—Part II. The Swelling and Shrinkage of Wood under Mechanical Restraint.**

D. NARAYANAMURTI and P. C. MAHAJAN. *Appl. Sci. Res.*, **A5**, 1956, 389.

The swelling and shrinkage of wood under restraint have been studied in the case of fourteen species of Indian timbers. By mechanically restraining swelling in the radial direction the tangential swelling will increase. By repeatedly wetting and drying under mechanical restraint of one side the specimens compress. Strength tests done on the specimens at the end of the cycles showed an increase, thus indicating possibilities of improving the properties of wood with simple treatment. P.G.M.

**3356. The Rheological Properties of Cellulose Sheets: Retrospect and Synthesis.**

A. H. NISSAN. *Tappi*, 1956, **39**, 93.

The principal characteristics of paper and board are reviewed and the studies of Steenberg outlined. Objections to a three-element model consisting of two springs and a dashpot with Eyring liquid, which afforded a useful curve-fitting technique, are considered. A new theory is proposed in which energy is imparted to the hydrogen bonds of the amorphous portion of the cellulose when paper is strained. Using an acceptable function for the energy potential of the hydrogen bond, it is deduced that the stress-strain curve will be non-linear. Variables of this curve are shown to consist of two parameters and the significance of these parameters in terms of time effects "frozen-in strain," and abundance of bond formation in the tested sample is discussed. E.W.J.M.

**3357. On the Effect of Speed on the Kinetic Friction of some Plastic Materials on Ice.**

C. D. NIVEN. *Canad. J. Phys.*, 1956, **34**, 362.

Curves are shown illustrating the effect of speed on the friction of Teflon, Bakelite, Nylon, Terylene, Plexiglas, Perspex, cellulose acetate, polystyrene and polyethylene, sliding on ice at  $-5^{\circ}\text{C}$  and  $-15^{\circ}\text{C}$ . Bakelite and Teflon had the lowest kinetic friction of the materials examined. Terylene also slid easily but polyethylene had rather high friction. Departure from Amontons' Law was very evident at high speed. A.

**3358. Delay Time of Plastic Flow in Germanium.**

J. R. PATEL, *Phys. Rev.*, 1956, **101**, 1436.

In recent experiments the effect of delay time was reinvestigated to determine its effects on the stress-strain curves. Using the equation  $\tau = \tau_0 \exp(Q/RT)$  after Seitz, it is found that  $\tau_0 = 3 \times 10^{-10}$  sec and  $Q = 39,000$  cal/mole. The impurity acting as a Cottrell atmosphere is thought to be copper. A.

**3359. The Accelerated Motion of Droplets and Bubbles.**

T. PEARCEY and G. W. HILL, *Australian J. of Physics*, 1956, **9**, 19.

The effect of the finite rate of the diffusion of vorticity in the case of accelerated motion of small droplets and bubbles can be an important factor in determining at any instant their velocity and position. E.W.J.M.

**3360. The Ductile Fracture of Polycrystalline  $\alpha$ -Iron.**

N. J. PETCH, *Phil. Mag. (Ser. 8)*, 1956, **1**, 186.

Evidence that ductile fracture requires a compressive stress on the dislocation arrays close in value to that required for cleavage is presented. It is shown that the ductile fracture stress  $\sigma_f$  is related to the grain diameter  $l$  by the equation  $\sigma_f = \sigma_0 + kl^{-1/2}$ , where  $\sigma_0$  and  $k$  are constants. This is the same form of relationship as already found for cleavage and the slope  $k$  is practically the same for both types of fracture. A.

**3361. An Investigation of the Power Requirements of Ball Mills by the use of Small Models.**

H. E. ROSE, *Tonindustrie-Zeitung*, 1956, **80**, 143.

In this paper the relationships between the power required to drive a ball or tube mill and the physical dimensions of the mill shell, the grinding media and the powder charge have been studied. The rate of grinding has not been investigated, but it is shown that, in general, the introduction of a powder to a mill increases the coefficient of friction to such an extent that the performance of a smooth mill does not differ significantly from that of a mill fitted with lifters. It is also shown that, for a given mill and ball charge, the power requirements increase with a reduction of the mean size of particle of the powder, but the powder demand becomes sensibly independent of the particle size when the ratio of ball diameter to particle diameter exceeds about 400. H.E.R.

**3362. Viscosity and Internal Friction in Metal Wires.**

C. SĂLCEANU, *C.R. Acad. Sci.*, 1955, **241**, 1554.

Derives relation between viscosity ( $\eta$ ) of metal in solid state and logarithmic decrement ( $\lambda$ ) as measured in determinations of internal friction by oscillations in wire specimen. Agreement is found between trend of  $\eta$  calc. and  $\lambda$  obs. as frequency of oscillation varies. It is suggested that there must be a connection between these physical constants. A.

**3363. Relation between Viscosity and Surface Tension of Liquids.**

N. K. SANYAL and S. S. MITRA, *J. Chem. Phys.*, 1956, **24**, 473.

It is shown that  $T \log \eta - a\delta = b$  where  $T$  = abs. temperature,  $\eta$  = viscosity,  $\delta$  = surface tension and  $a$ ,  $b$  are constants, fits the properties of water and alcohol between 273°K and the boiling points. For water  $a$  is 66.56 and  $b$  is  $\sim 29.6$ , for alcohol  $a$  is 10.68 and  $b$  is  $\sim 19.8$ . A.

### 3364. Finite Displacements in Rheological Bodies.

A. E. SCHEIDEGGER. *Can. J. of Physics*, 1956, **34**, 498.

The displacement within the continuous medium is described by three "co-ordinate" functions as functions of three "parameters" and of time, "co-ordinates" and "parameters" being equivalent. Formulae are deduced which enable one to express the boundary conditions, the equations of motion and any chosen rheological condition in either parameter, space or co-ordinate space. The notion of finite strain is scrutinized. The scheme presented is not simple and is strictly phenomenological in that there is no intrinsic motivation to assume one rheological condition after another. E.W.J.M.

### 3365. Intrinsic Viscosity and Kinematic Viscosity.

C. TANFORD. *J. Phys. Chem.*, 1955, **59**, 798.

A simple relation between these is derived which permits an evaluation of the former without the need of accurate density measurements. A.

### 3366. The Hagenbach—Couette correction in Capillary Viscometry.

P. TUOMIKOSKI. *Ann. Acad. Sci. Fennicae AI*, 1955, No. 189, 13 pp.

Describes an experimental method of finding the correction to be applied to capillary viscometers in which the outlet is funnel shaped. The requisite theory and some typical results are given. A.

### 3367. In Memoriam: Fedor Nikoforovich Shvedov. On the 50th anniversary of his death.

M. P. VOLAROVICH and S. M. LEVI, *Kolloidnyi Zhurnal*, 1956, **18**, 129.

A brief biography: claims that S's paper in 1900 anticipated Bingham's (1922) equation  $\tau = \theta + \eta_{pl} \cdot \frac{du}{dz}$  where  $\theta$  is critical shear stress and  $du/dz$  velocity gradient. Further claims that B.P. Veinberg (1912, *J. Soc. phys.-chim. russe*, **44**, 201) applied the same principle to concentric cylinder viscometers, thus anticipating Reiner and Rivlin (1927).

V's equation is written:  $M(\text{rotation moment}) = (-\eta_{pl} \cdot r \frac{dw}{dr} + \theta) 2 \pi r^2 H$ . (H is cylinder height). This gives, for angular velocity  $\Omega$  of cylinder:

$$\Omega = \frac{\theta}{\eta_{pl}} \ln \frac{r_1}{r_0} + \frac{PR}{2\pi H \eta_{pl}} \frac{1}{r_1^2} - \frac{1}{r_0^2}$$

Quotes V's table for yield-values of an asphalt at 15°, 20° and 30° to show that "Bingham systems" were known in Russia at this date. G.W.S.B.

### 3368. Streaming Birefringence as a Hydrodynamic Research Tool—applied to a Rotating Cylinder Apparatus above the Transition Velocity.

H. WAYLAND. *J. appl. Phys.*, 1955, **26**, 1197.

Streaming birefringence was studied in the annular space between a fixed outer and a rotating inner cylinder at velocities on both sides of transition to Taylor vortex flow. With a well collimated light beam, quantitative measurements were possible as close as 0.1 mm. from a wall. For an aqueous colloidal solution of bentonite, shear stress could be evaluated from birefringence measurements only when, for a given birefringence, the angle of extinction was the same in the unknown flow as in a known laminar flow. In turbulent shear flow this is not generally the case. For a pure liquid, ethyl cinnamate, the angle of extinction remained at 45° to the streamlines, within experimental error, under all flow conditions obtainable; thus even in turbulent shear flow the birefringence appears to arise primarily from the principal stresses. Assuming birefringence propor-

tional to mean shear stress permits construction of a velocity profile across the gap, yielding the correct value of velocity of the moving wall within limits of experimental error. Thus in this type of two-dimensional shear flow the mean shear stress can be measured by means of the streaming birefringence of a pure liquid. A.

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## REVIEW

### **The Mechanical Properties of Textile Fibres**

(Ed.) R. MEREDITH

North Holland Publishing Co. (Amsterdam) pp. XII+333+4 plates, 61s.

Seventh in the series "Deformation and Flow: Monographs on the Rheology of Natural and Synthetic Substances," this book achieves its main objectives in giving a general picture of the mechanical properties of the different textile fibres. Cellulose, protein, and synthetic fibres are dealt with; the section on protein fibres is written by L. Peters and H. J. Woods. The main data on stress-strain relations, both in elongation and in torsion, are given, together with data on creep and stress relaxation, dynamic modulus and loss angle, tensile and shear strength and breaking extension, and permanent set. The effects of changing temperature, humidity, and rate of extension are described, and information on structure and chemistry is included. The book fulfils a real need, especially with its emphasis on time-dependent properties; it is readable and well indexed.

The important but difficult question of the thermodynamic treatment of fibres which exhibit hysteresis and relaxation might well have been given a fuller discussion. It is not clear how equation (5), p. 15, which expresses tension as a sum of terms depending on changes of internal energy and of entropy, is a generalization applicable to non-ideally elastic fibres, nor whether in particular its application to the date (p. 214) for a wool fibre in water showing great tension-extension hysteresis is justified. Moreover, the effect of water absorption should have been indicated and the appropriate known extensions of the thermodynamic analysis referred to.

There are some minor errors. For example, 70% extension is too low for crystallization of rubber at ordinary temperatures (p. 9 and plate 1c). The proportionality of log stress to log time does not necessarily lead to the Nutting power law between stress, time and strain (eq. (4), p. 42). The determination of secondary creep from the extension remaining after a few minutes recovery may be convenient, but can hardly lead to a separation "in principle" of primary and secondary creep (p. 123). The sign of  $P$  in eq. (1), p. 220, is wrong. I counted over twenty misprints (e.g. Plate Processes instead of Rate Processes, p. VIII).

This book is well worth acquiring for its information on the behaviour of fibres, but where interpretation is involved it should be read critically. Some correlations between fibre rheology and molecular structure are stated for which the evidence available at present is surely too slight. For example, on p. 196 it is suggested that certain rather small differences in the stress-strain curves for two varieties of silk fibroins are due to side-chain differences. Could they not arise, for example, from different macroscopic heterogeneities along the fibre length or over the fibre cross-section? Moreover, owing to the large number of important molecular interactions involved in these condensed non-rubberlike states, there is (in my opinion) at present no theory well enough established to form the basis for such correlations.

A. S. LODGE.