

APPLICATIONS OF LIQUID CRYSTALS IN AERODYNAMIC TESTING

by

Paul Bonnett

St. Annes College



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Parks Road, Oxford

ABSTRACT
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This thesis investigates the applications of liquid crystals in three areas of aerodynamic testing, namely temperature, shear stress and pressure measurement over surfaces.

The use of the selective reflection colours from encapsulated chiral nematic materials to map surface temperatures is assessed. This method is very successful in a wide range of applications for determining heat transfer rates, but has limitations where high heat transfer rates are present, due to the thermal response time of the material structure, and the effects of temperature gradients. The thermal time constant is determined as a function of material viscosity. It is typically 10ms for a chiral nematic material at room temperature. The effect of a temperature gradient on the selective reflection is studied in terms of the pitch gradient produced in the material structure. Two improved methods are then proposed.

The first makes use of the cholesteric to isotropic transition to indicate an isotherm on a heated surface, while the second uses changes in the birefringence colours produced by an aligned nematic layer applied to a surface and viewed between crossed polarisers.

Changes in the selective reflection colours from cholesteric liquid crystals can be used to assess surface shear stress levels. This method is assessed and determined to be too problematical for accurate measurements. A new method is proposed, based on the shear induced texture change from the focal-conic to the Grandjean texture in the cholesteric phase. This method may be used to quantify surface shear stress levels as well as to provide excellent flow visualisation once flow alignment has occurred.

Tests have been conducted on the pressure sensitivity of chiral nematic liquid crystal materials. The results indicate that the change in transition temperature with pressure is of the order of $40^{\circ}/\text{kbar}$, which may not be sensitive enough for wind tunnel purposes.

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NOMENCLATURE

a	inner radius of viscometer
A	Keating equation constant
A	area
b	outer radius of viscometer
B	Keating equation constant
c	specific heat capacity
C	spring constant
C_f	skin friction coefficient
d	thickness
h	heat transfer coefficient
I	electric current
k	diffusivity
K	thermal conductivity
K'	parabola constant
l	length
M	mass
n	refractive index
p	helical pitch
P	pulse power
Q	heat
q	heat transfer rate
r	radius
R	electric resistance
Re	Reynolds number
s	Laplace constant
t	time, also t_o , t_c , t_p , t'
T	temperature, also T_o , T_g , T_{lc} , T_h , T_p
v	velocity

V voltage

x distance

α temperature coefficient of resistance

ρ density

λ wavelength

μ, η viscosity

ν kinematic viscosity = μ/ρ

ϕ angle

τ_o surface shear stress

τ thermal time constant

ω angular velocity

1. INTRODUCTION

The liquid crystalline state of matter was discovered over a hundred years ago by the Austrian botanist Reinitzer. However it has only been over the past twenty years, with the emergence and rapid growth of the liquid crystal display industry, that the technology has evolved to the current advanced state.

Interestingly one of the earliest applications of the unique properties of mesogens was the use of the thermochromic properties of cholesteric liquid crystals in simple temperature sensing devices. The development of these remained static for many years, limited by the materials, which were derivatives of cholesterol and as a consequence had a limited range of properties and were chemically unstable. The relatively recent development of non-sterol based cholesterogens has stimulated a new range of applications of cholesteric liquid crystals whose structure is sensitive to their environment. Under particular conditions, temperature, shear and pressure may induce colour changes in the material. The sensitivity to some external stimuli may be enhanced for a given application by correct mixture formulation.

The cholesteric material can be applied to a surface in neat or encapsulated form and used to map events over the whole surface area. This is the main advantage of the method over existing spot measurements from, for example, thermocouples or pressure tappings. The colour changes are immediate and reversible. Qualitative information can be seen by eye, which may be quantified where appropriate by detailed analysis of video recordings.

This thesis has the broad aim of investigating and developing a range of practical techniques which utilise chiral nematic liquid crystals to measure aerodynamic effects. Three main areas were studied, which form the three main sections of this thesis:

1. Surface temperature measurement.

2. Surface shear stress measurement.

3. Pressure measurement.

After a general introduction to liquid crystals in chapter 2, heat transfer and the methods by which liquid crystal materials can be applied to the problem of measuring and mapping surface temperature in wind tunnel conditions are discussed in chapter 3. New improved methods are also examined. This chapter concludes with a section on the measurement of various thermal parameters for liquid crystals. Chapter 4 is concerned with the measurement of surface shear stress using liquid crystals with emphasis on an attractive new technique discovered in the course of this work. The sensitivity of liquid crystals to pressure is reported in chapter 5 and chapter 6 draws together the main conclusions of the work and points out useful areas for future study.

The principle accomplishments of this thesis are:

1. The determination of the helical thermal response time for cholesteric liquid crystal materials. These results were presented at the British Liquid Crystal Society annual conference in Hull, 1987.

2. The assessment of the effects of a temperature gradient on the selective reflection from a cholesteric layer.

3. The construction of apparatus for, and measurement of, the thermal product and thermal conductivity of cholesteric liquid crystals

4. The development of a new method to map surface temperature using the colour change at the cholesteric to isotropic transition.

5. A new method was devised and developed using the changes in birefringence colours in an aligned nematic layer to map surface temperature. This method greatly extends the range of measureable heat transfer rates using cholesteric liquid crystals and overcomes the problems associated with the original method. The method is described in UK patent application number 8804177.

6. The effects of shear stress on the selective reflection colours of cholesterics were assessed as a function of lighting conditions.

7. A new method was devised and developed, using the shear induced texture change from focal conic to Grandjean in a cholesteric liquid crystal, to quantify surface shear stress. This was presented at the British Liquid Crystal Society annual conference in Strathclyde in 1988 and at a meeting at Farnborough in 1988. It is also the subject of a paper, currently in press, to be published in 'Liquid Crystals', and is disclosed in UK patent application number 8804176.

8. A pressure cell was designed, built and tested to enable transition temperature measurements to be carried out on liquid crystals at pressures up to 2000p.s.i.

The project was carried out at the Department of Engineering Science at Oxford University and at the Royal Signals and Radar Establishment (RSRE) at Malvern, making use of the liquid crystals materials and physics expertise at Malvern and the engineering expertise at Oxford.

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I am also especially grateful to the organisations who provided funding and financial assistance at various stages throughout this thesis, namely Control Sensors Ltd. BDH Ltd and the MOD.

2. LIQUID CRYSTALS

This year is the hundredth anniversary of the discovery of liquid crystals in Austria by Reinitzer. Although the term 'liquid crystal' appears contradictory it is purely a descriptive term for a large group of mainly organic materials which exhibit a phase in between the liquid and solid states. On melting the solid, a mesophase is formed which has some of the mechanical properties of liquids, but with some anisotropic physical properties, hence the term 'liquid crystal'. This unusual melting behaviour may simply be envisaged as a gradual breakdown of the forces holding the crystalline lattice together, leaving the rod like molecules with long range orientational order. The smectic phases in addition have a density wave giving rise to layer like structures. Although the materials are fluid, they have anisotropic physical properties and it is this combination which makes them of use in electro-optic and other applications. Eventually at a higher temperature (the clearing point) the long range order is lost and the materials form an isotropic liquid phase.

Materials which become liquid crystalline with temperature in this way are called 'thermotropic'. There is another type of liquid crystals, called lyotropics, which form solutions of ordered micelles depending on solvent concentration. Disc shaped molecules are also possible, but these are considered outside the scope of this thesis. The thermotropic liquid crystals used are enantiotropic, i.e. the liquid crystal phases form above the melting point.

Thermotropic liquid crystals are formed by organic materials with anisotropic molecular geometries. Typically, and for all materials used in this study, the molecules are rod shaped, of the order of 20 to 30 angstroms long and about 5 angstroms in diameter. The molecular anisotropy causes the long range ordering of the molecular axes which gives rise to anisotropy in the materials physical properties, e.g. refractive index, conductivity, viscosity etc.

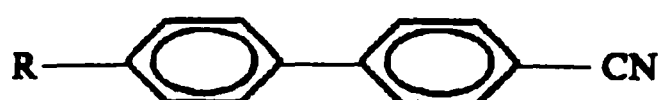
Liquid crystals form three general categories of mesophase, which are distinguished by the degree of order of their constituent molecules. These are termed the nematic, smectic and cholesteric mesophases, although the nematic phase may be considered as a special case of the cholesteric phase. In each type the long range molecular ordering is defined by a unit vector, called the 'director', which describes the average orientation of the long molecular axes. Thus although each molecule is free to undergo thermal fluctuations giving rise to short range disorder, the average direction of the long molecular axes over time and distance is defined by the director (n). The relationship between the long axes and the director is defined by an order parameter¹ (S) given by :

$$\langle \frac{1}{2} (3 \cos^2 \alpha - 1) \rangle$$

where α is the angle between the molecular axis and the director.

2.1. Nematic

The nematic mesophase is the least ordered liquid crystal phase, in which the long axes of the molecules are approximately parallel to the director. There is rotational symmetry around this direction, so the phase is uniaxial. Figures 1 and 2 show simple schematic representations of the phases and their relationship to each other. There is no short range correlation between the centres of mass of the molecules, which can translate freely. The axis of uniaxial symmetry has no polarity. A well known cyanobiphenyl nematic molecule structure is shown below:



where 'R' may be, for example C_5H_{11} .

FIGURE 1. Schematic of the arrangement of rod-like molecules in the three liquid crystal mesophases.

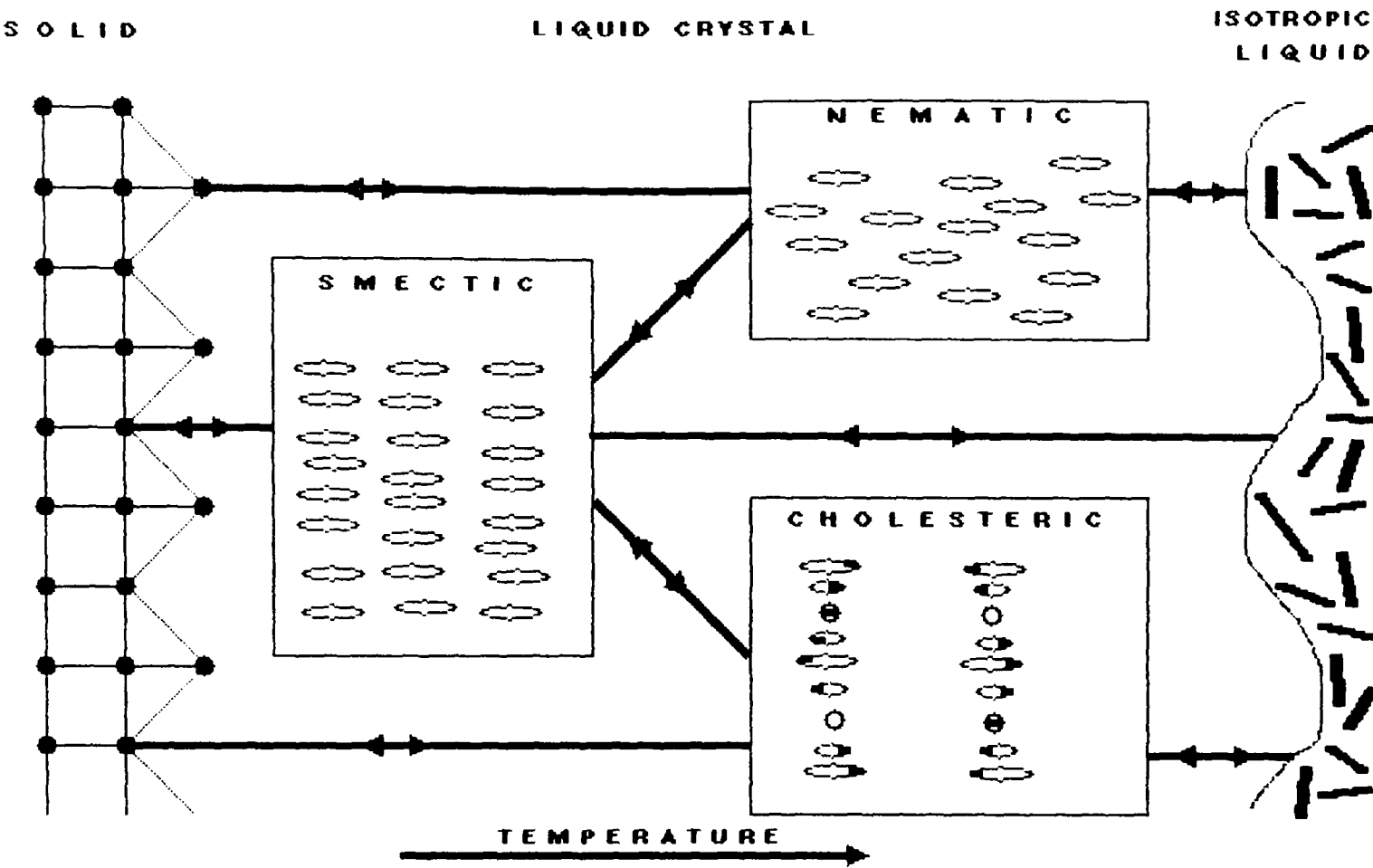
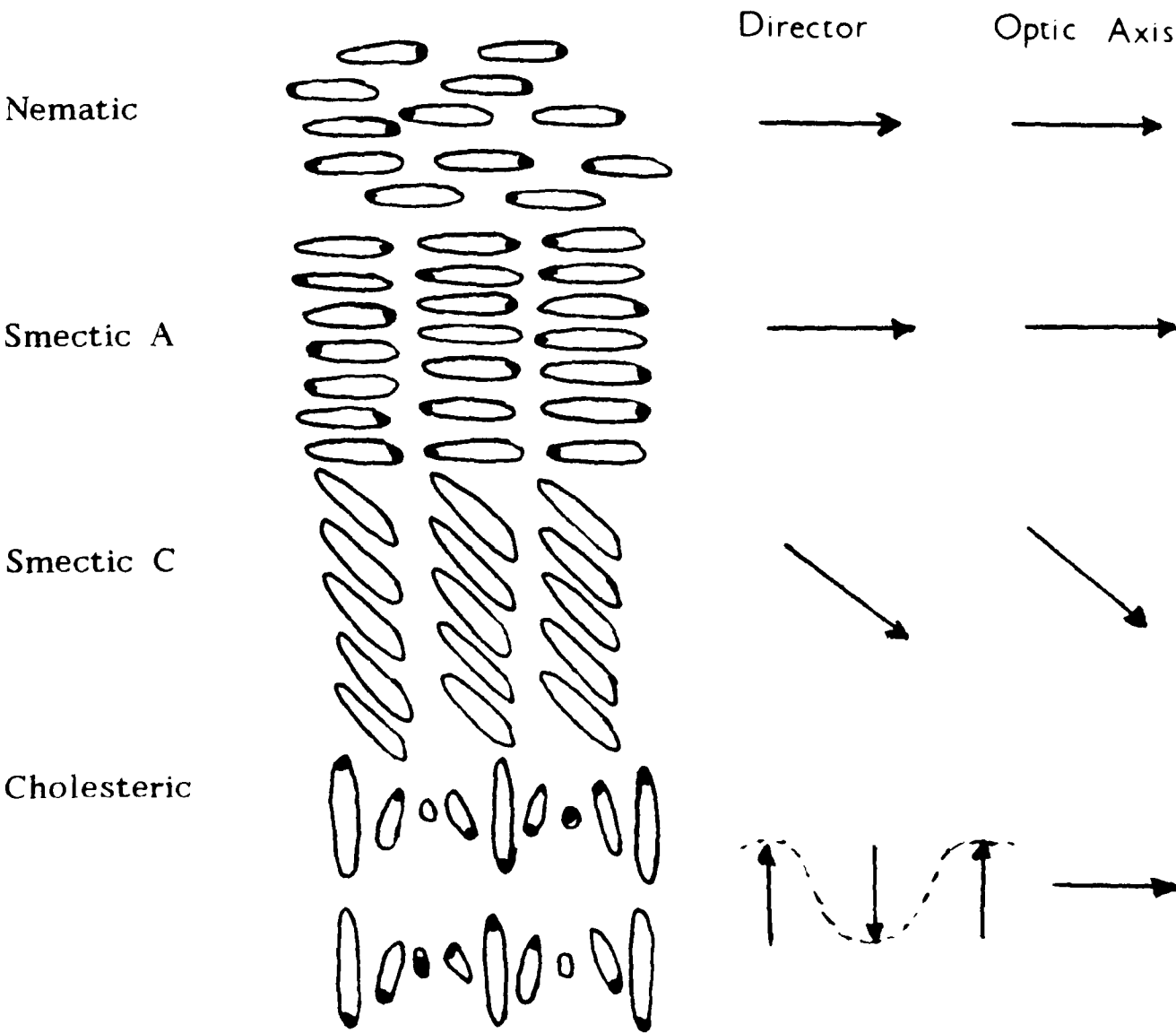


FIGURE 2. Liquid crystal molecular structures.



This is one of a class of liquid crystal which has found widespread use in electro optic displays used in watches, calculators, car dashboards and portable computers etc. These rely on the competition between electric field and surface forces to align the director. Materials with large dipole moments may be synthesised to give mesogens with a large dielectric anisotropy. The positive materials ($\epsilon_{||} > \epsilon_{\perp}$) are commonly used in display devices since the director tends to align parallel to an applied electric field. The inner surfaces of the display cell may be treated to promote perpendicular, parallel or tilted alignment of the director with respect to the surface. A wide range of optical effects may then be achieved and these provide the basis for a plethora of commercially available liquid crystal display devices.

2.2. Smectic

The smectic phase has the additional ordering of a sinusoidal density fluctuation parallel to the director. This indicates a fluid, layer like structure, shown schematically in figures 1 and 2. There are several currently known polymorphs of the smectic type, denoted with an alphabetic subscript. These are generally much more viscous than nematics. In the case of the smectic A polymorph (shown in figure 1.) the low mobility finds application in laser addressed optical storage devices. The ferroelectric properties of the non-centrosymmetric chiral smectic C phase have attracted a lot of commercial interest recently as they enable fast (μs) electro-optic switching.

2.3. Cholesteric

The cholesteric phase is formed by liquid crystal materials with chiral molecules, i.e. molecules which are different from their mirror image. Commonly this arises from the asymmetry of the tetrahedrally substituted carbon atom. (i.e. a carbon atom with four different connecting groups

arranged in a tetrahedron). This molecular asymmetry only allows packing with a small offset, which is cumulative through the material, producing a macromolecular twisted or helical structure, with a pitch defined by a 360° rotation of the director.

The offset is typically 10 to 20 minutes of arc between each layer, thus one pitch length may consist of about 1000 molecular layers. The structure is shown schematically in figure 1, but it should be emphasised that this is an idealised model and rigid layers do not exist. The structure exists only if a time average of the molecular positions is taken. Most explanations of the cholesteric phase are based on this model, although its limitations should be kept in mind. The cholesteric phase is thermodynamically similar to the nematic phase, as each layer may be considered as like a nematic in one plane. Indeed a nematic can be considered as a cholesteric with infinite pitch.

It should be noted here that the addition of an optically active isomer to a nematic will result in a cholesteric .

The cholesteric pitch may be either right or left handed depending on which of the two mirror image molecular types is present. If equal amounts of each type are present then a nematic is formed. This is termed a racemic mixture.

Cholesteric materials occur naturally as derivatives of the alcohol, cholesterol. However these materials tend to be very viscous and unstable, i.e. they tend to crystallise over time and degrade quickly when exposed to ultraviolet light. Improved, non-sterol based materials were invented in the UK in the 1970's² These chiral nematic materials are available commercially from BDH Ltd.^{C1} They are much less viscous and more chemically and photochemically stable than cholesteryl esters, and offer a wider range of physical properties.

2.3.1. Textures

Cholesterics may exist in several different textures. The texture which exhibits selective reflection is called the 'Grandjean texture' in which the helical axes are aligned uniformly normal to the surface, as shown in figure 3.(a). This texture is promoted by the use of a homogeneous surface alignment treatment such as a rubbed polymer (e.g. polyimide).

If the helical axes are uniformly parallel to the surface a microscopic pattern may be seen, resembling a fingerprint. This is referred to as the fingerprint texture - figure 3.(b).

More usually the texture is broken up into small domains of randomly orientated helical clusters giving the focal conic texture, see figure 3.(c). This is the thermodynamically preferred texture and occurs in the absence of any orienting forces. This texture scatters white light and appears almost transparent in thin layers, but cloudy in thick layers. A small aligning force provided by surface forces or shear will transform this texture into the Grandjean state.

Many cholesterics go through one or more blue phases in a narrow temperature band just before going isotropic. They appear blue in ambient light and have cubic structures.³.

2.3.2. Optical Properties

The optical properties referred to in this section pertain to the well aligned Grandjean texture and its temperature dependant selective reflection.

Selective reflection occurs when the cholesteric pitch length is approximately equal to the incident light wavelength. Incident electromagnetic waves interact with the rotating dielectric ellipsoid and the scattered light constructively interferes at the appropriate wavelength. Consider figure 4. If we consider incoming unpolarised light incident on the structure shown in figure 4, it may be resolved into any two perpendicular plane polarised waves.

FIGURE 3. Textures of the cholesteric structure.

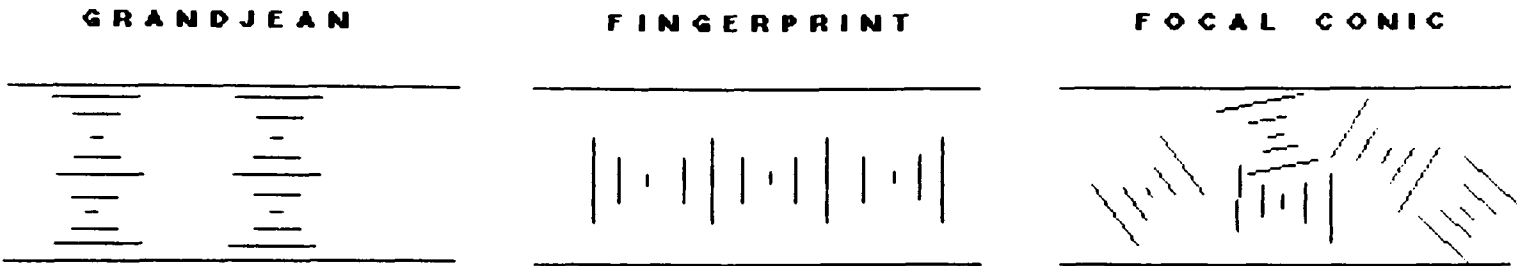


FIGURE 4. Representation of the cholesteric helical structure.

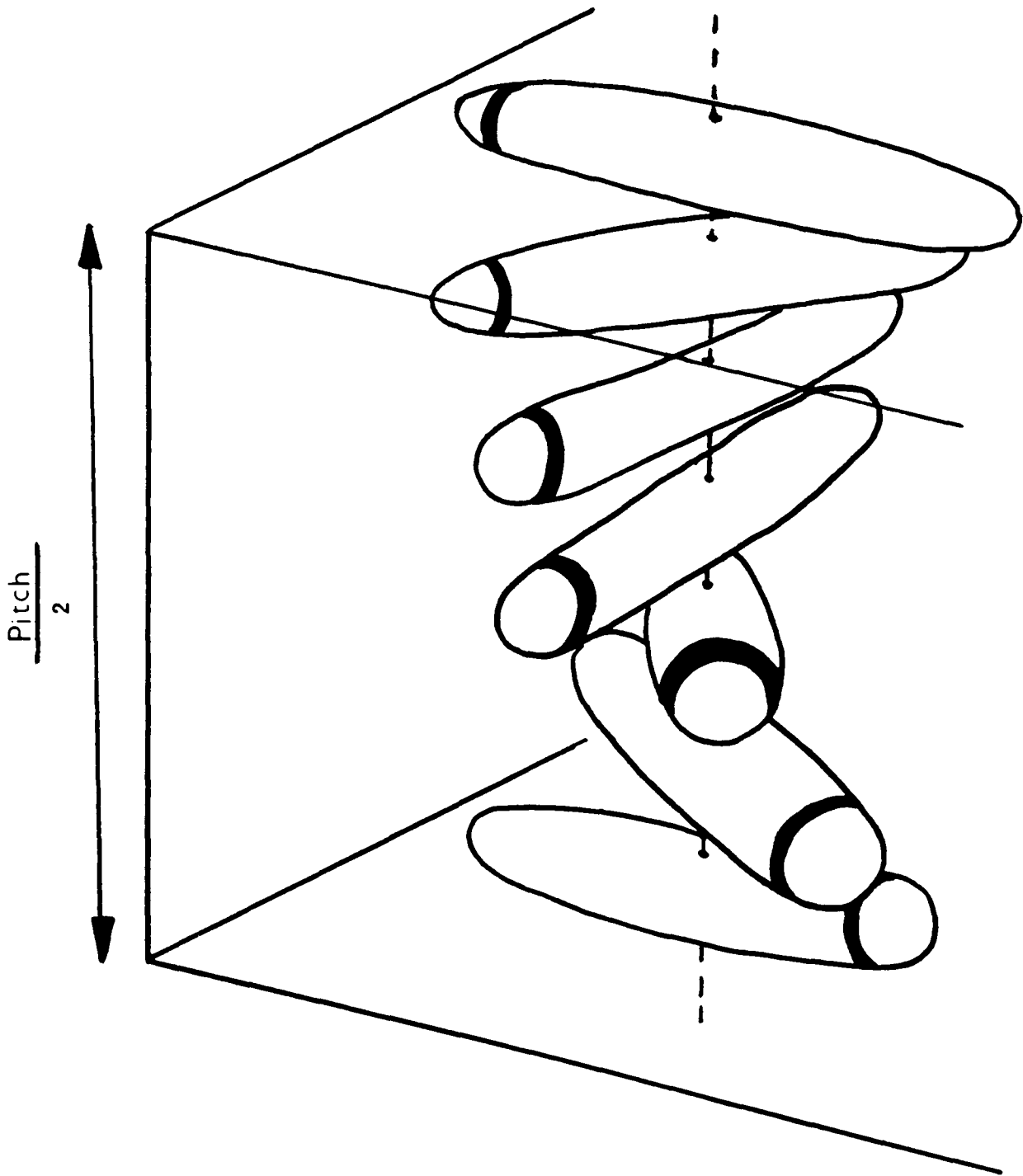


FIGURE 5. Typical reflection spectrum from a well aligned cholesteric liquid crystal sample.

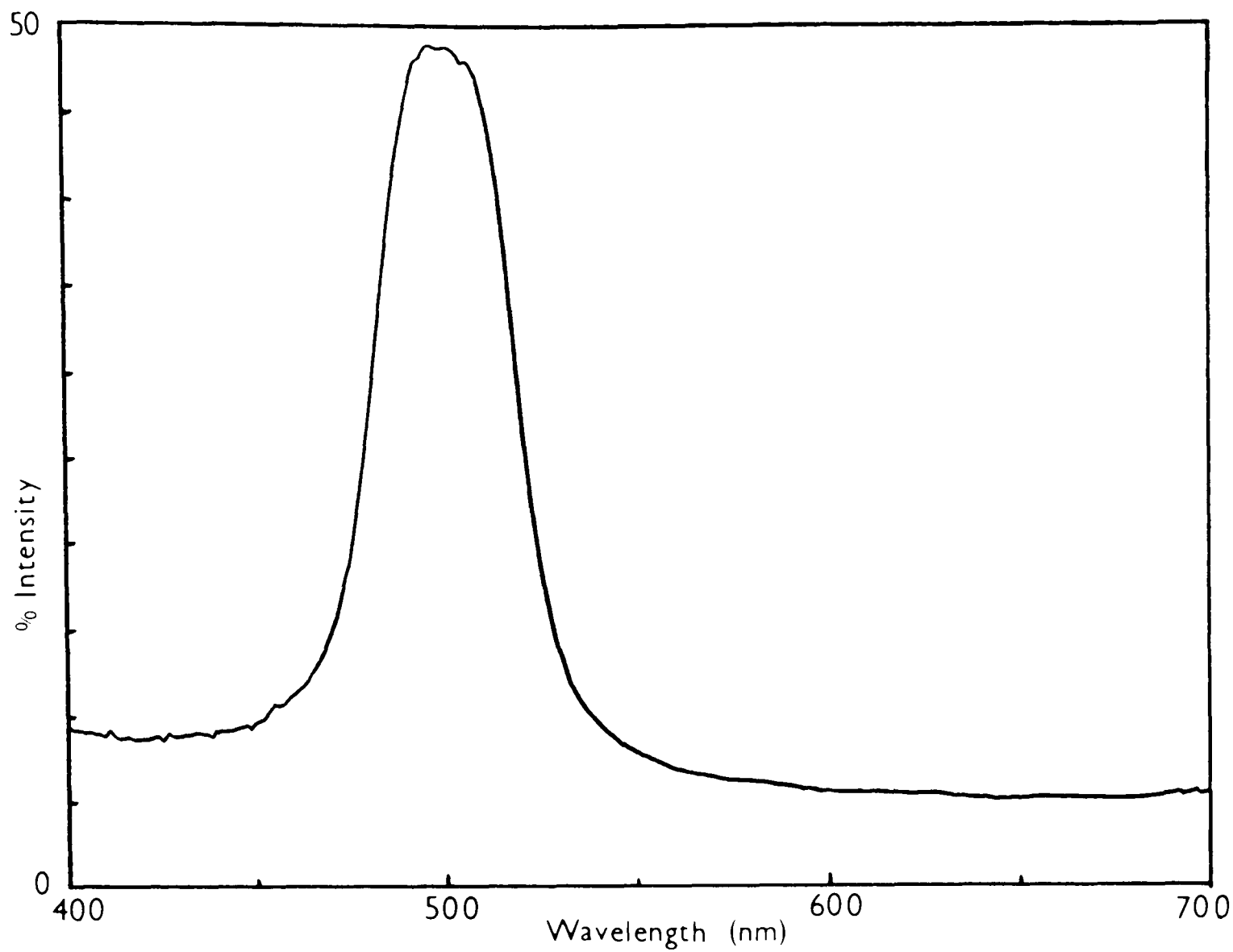
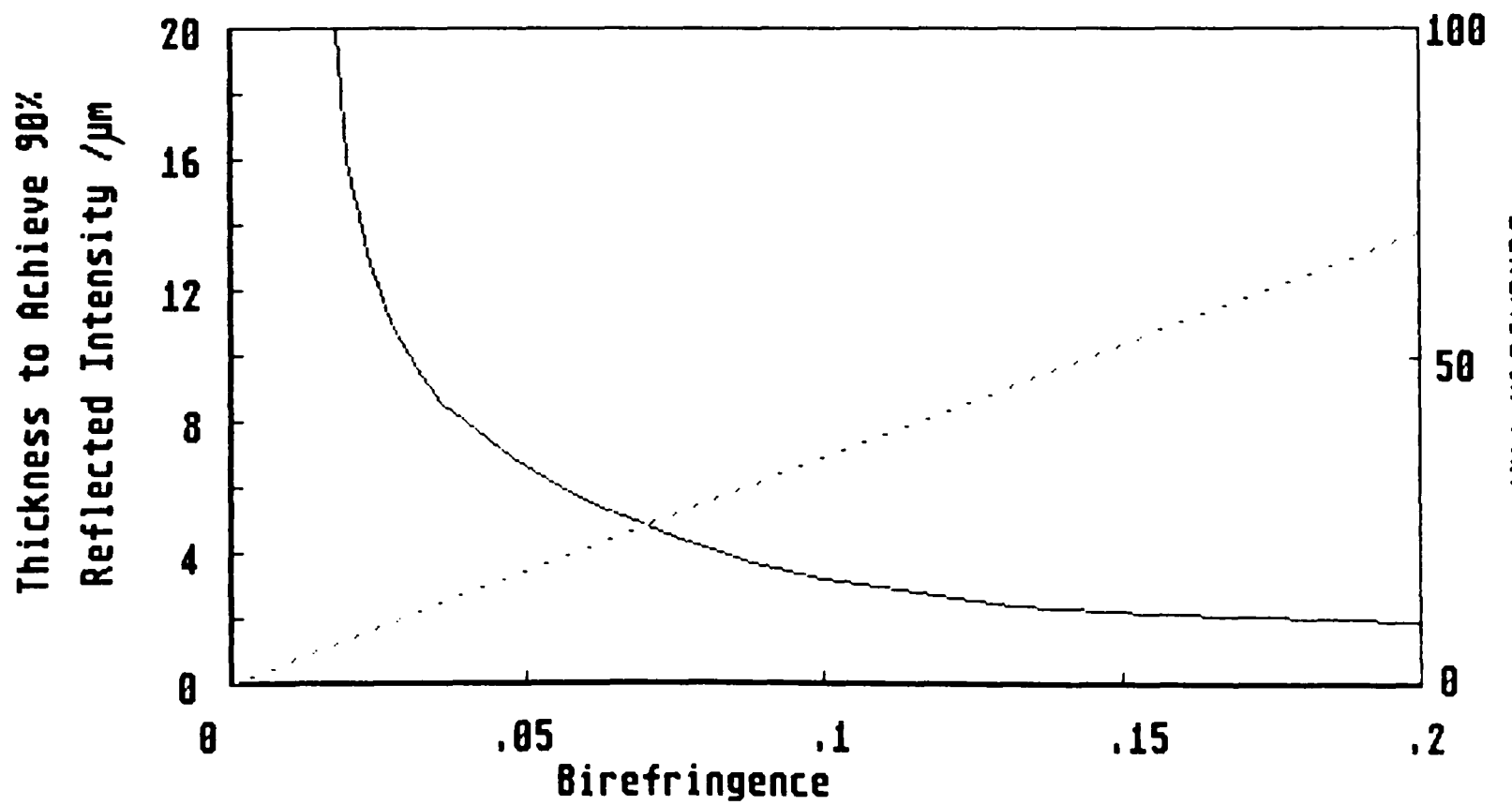


FIGURE 6. Sample thickness required to achieve 90% intensity against material birefringence. Dotted line shows bandwidth against birefringence.



For example one such plane wave may make a zero angle with the surface director. This plane wave may itself be resolved into two opposite (right and left handed) circular polarisations. If the material has a right handed helix, the right handed polarisation may rotate with the same pitch as the material helix, and experience a refractive index, n_x . This component will experience some scattering at each molecular 'layer'. This scattering will form constructive interference at the repeat distance of the material helix ($p/2$), i.e. $\lambda = n_x p$. resulting in the reflection of this component. The opposite circular component will experience a sinusoidally varying refractive index. As a result this component sees an average refractive index and is transmitted. The second plane polarised wave incident at right angles to the surface director can again be considered as two opposite circular polarised components. As before the left handed polarisation sees an average refractive index and is transmitted, while the right handed component sees n_y and constructive interference occurs when the wavelength is equal to twice the repeat distance of the material helix, i.e. $\lambda = n_y p$.

The reflection spectra using unpolarised light for a well aligned cholesteric sample is therefore rectangular in shape, with a bandwidth defined from its birefringence. So $\Delta\lambda = \Delta n.p$. where $\Delta n = n_e - n_o$. The spectrum shown in figure 5 was obtained by the author from a well aligned sample, and was measured using the technique described in Appendix 1.

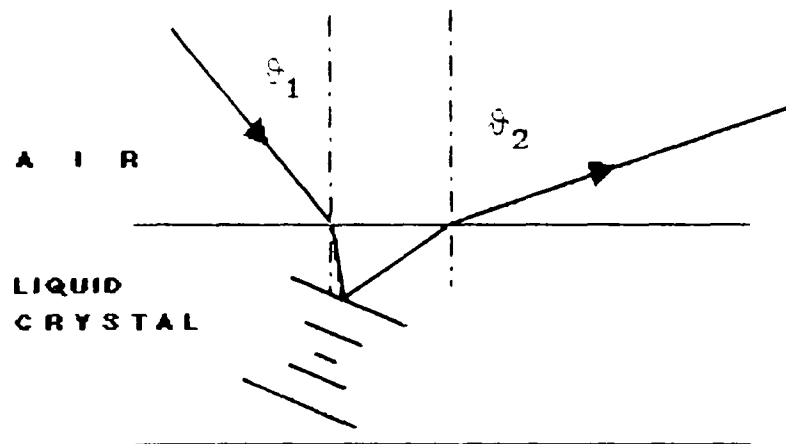
A single layer of cholesteric liquid crystal reflects at most 50% of the incident light over $\Delta\lambda = \Delta n.p$, and the reflected light is circularly polarised, having the same sense as the material helix. The intensity of the reflected light is thickness dependent up to a saturation level, and birefringence dependant. A thinner layer is required to achieve the maximum level of reflection for chiral nematics as they generally have a higher birefringence than cholesteryl esters (see figure 6) producing more light

scattering at each molecular 'layer'. As the liquid crystal does not itself absorb any light, the selectively reflected light should be viewed against a black absorbing background in order to enhance the contrast.

The deVries formula⁴ $\lambda = np$, only applies for normal incidence, but Fergason⁵ has derived an expression for all lighting and viewing angles from a polydomain sample:

$$\lambda = n.p.\cos.\frac{1}{2}\left(\sin.^{-1}\frac{\sin.\vartheta_1}{n} + \sin.^{-1}\frac{\sin.\vartheta_2}{n}\right)$$

where n is the average refractive index. This is typically about 1.6



The angle dependence is Bragg - like and analogous to X-ray diffraction from crystals. The differences arise from the fact that Bragg diffraction from crystals occurs as the result of an ordered array of discrete scattering centres, which produce many diffraction orders. For cholesteric reflection these scattering centres are not discrete, but diffuse, resulting in the removal of the higher orders and a dominant diffraction lobe producing reflection colours even at non specular angles. Figure 7 (page 14) is plotted from the authors experimental data, but matches the above equation closely.

When the incident light wavelength is less than the pitch, very high rotary powers are observed for transmitted light, of the order of 10^3 to 10^5 $^\circ/\text{mm}$ as compared to quartz, which shows rotary powers of about $10^\circ/\text{mm}$. Incident polarised light is again split into opposite circular components which

FIGURE 7. Angle dependence of the peak reflected wavelength from a well aligned cholesteric sample.

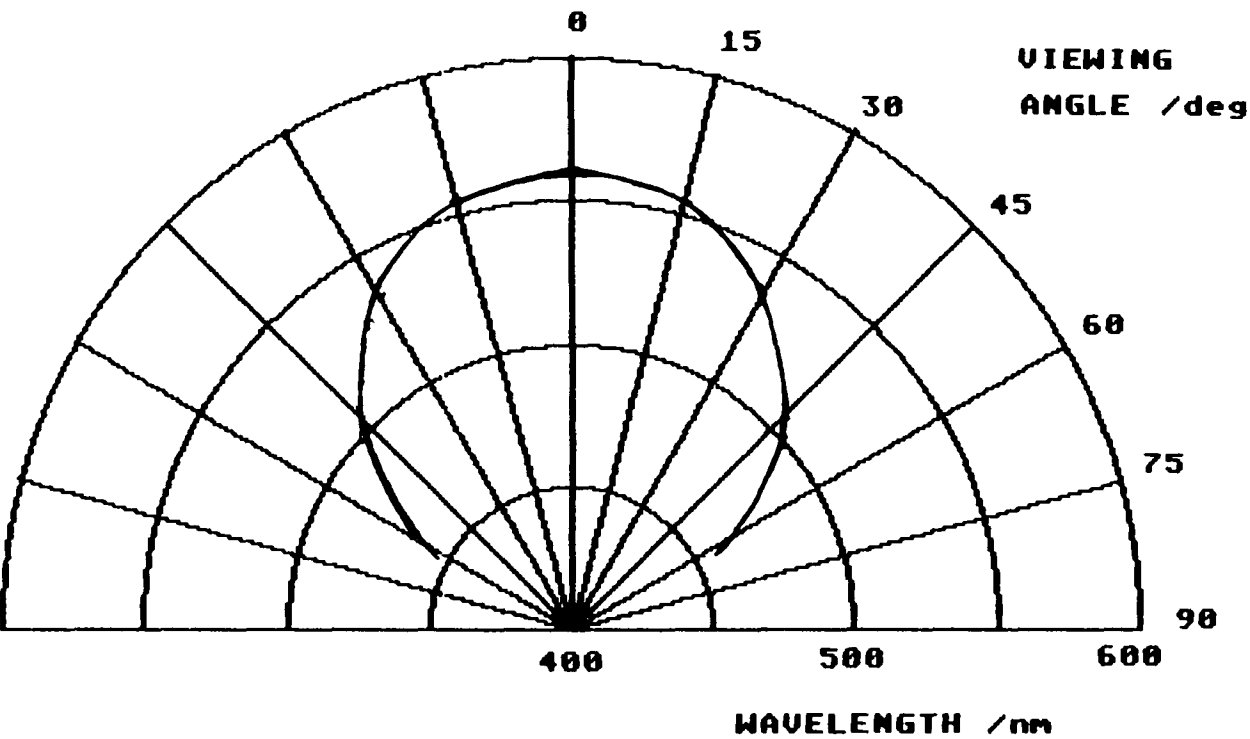
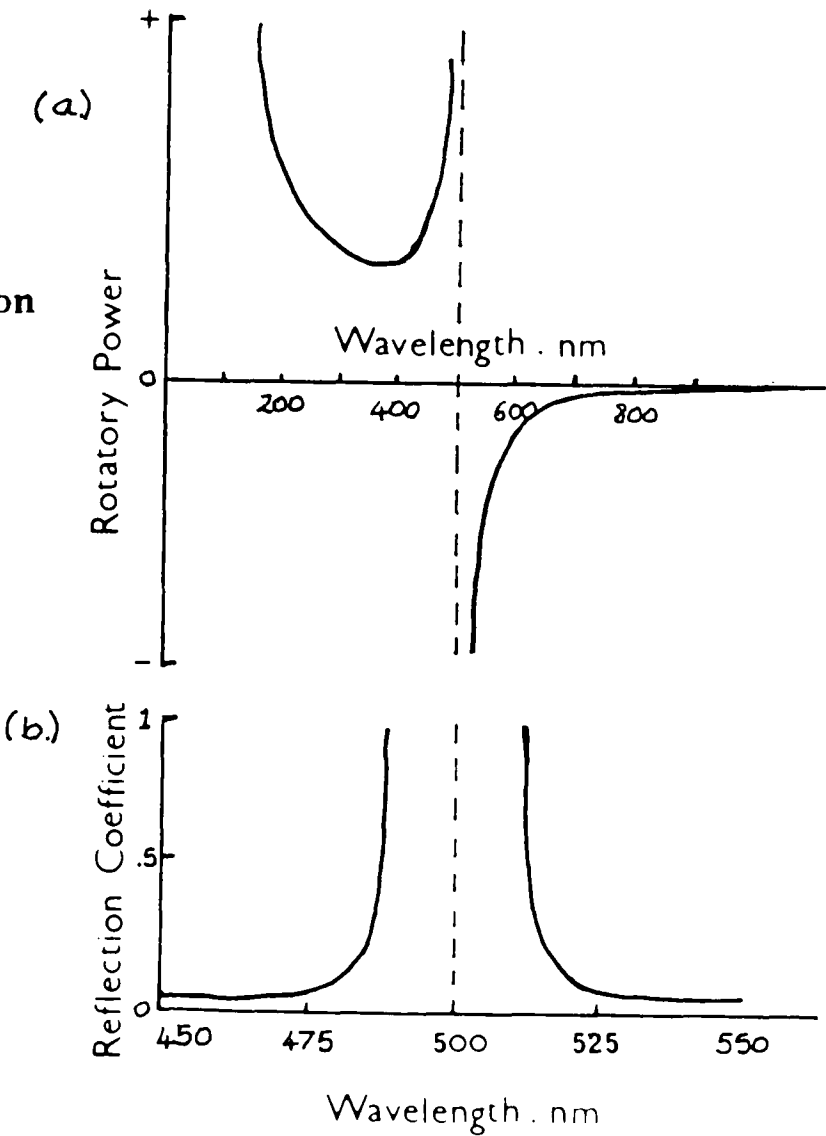


FIGURE 8. Variation of rotary power with wavelength for a typical cholesteric material. (Figure taken from BDH Ltd catalogue).

- (a). Wavelength dependence of rotation of plane of polarisation and
- (b). wavelength dependence of ideal reflection coefficient.



propagate through the material at different velocities producing a phase shift when they recombine, resulting in a large rotation of the polarisation. When the wavelength is much smaller than the pitch, the structure is able to rotate the plane of polarisation in a manner analagous to waveguiding. Hence for very small wavelengths the rotation is very high.

As the wavelength approaches the pitch the rotation decreases until one circular component begins to be reflected, in the reflection band. Beyond the reflection band the polarisation is unaffected by the structure. See figure 8.

More detailed explanations of these effects based on Maxwells equations have been presented by several authors ^{6,7,8,9}.

2.3.3. Factors Affecting the Pitch

Any of the following factors may produce a change in the wavelength of the reflected light due to their action on the cholesteric pitch, and therefore may be measured by analysing the selective reflection changes.

1. Temperature. In cholesteric materials which do not have an underlying smectic phase, or away from pretransitional regions, the helical pitch may either increase or decrease with temperature by a small amount ($\sim 1\text{nm}/^\circ\text{C}$) as a consequence of order parameter and short range molecular association changes.¹⁰ Much larger effects ($\sim 1000\text{nm}/^\circ\text{C}$) are possible in the pretransitional region of the underlying smectic phase. As the cholesteric is cooled the build up of smectic like ordering has the effect of rapidly unwinding the helix. Hence the selective reflection colour can changes very rapidly near a transition, with the red reflection being much more temperature sensitive than the blue. See figure 9. Chiral nematic materials can be synthesised to show this colour band anywhere between about -50°C to 150°C . The range over which the colour play occurs can also be tuned to cover less than a degree or over 50° . The short temperature range mixtures enable

greater accuracy to be achieved in temperature resolution, even when reading with the unaided eye. The human eye is most sensitive to the yellow band, and this band can be tuned to occupy a fraction of a degree. Since very thin (e.g. 10 - 20 μm) layers of cholesteric are required for bright reflection, very good spatial resolution and temperature resolution can be achieved¹¹. These factors have led to many applications for cholesteric liquid crystals in the non-destructive testing of, for example electronic components¹² structural weakness¹³, medical thermography¹⁴ thermal imaging¹⁵ and air and surface temperature mapping.¹⁶

2. Pressure. The primary effect of pressure appears to be to change the transition temperatures. Increasing pressure causes transition temperatures to be raised, resulting in a red shift, or lengthening of the pitch^{17,18,19}.

3. Shear Stress. The primary effect of shear is to distort the ordered helical structure and at some critical shear the ordered Grandjean texture reverts to the disordered focal conic texture. At lower shear rates a drop in reflected intensity occurs and an increase in bandwidth with an accompanying colour shift, which is normally to the blue²⁰.

4. Chemical Action. Many solvents will affect the transition temperatures and hence the pitch. Generally the transition temperatures drop with increasing solvent concentration, causing a blue shift. Some solvents, e.g. chloroform, will evaporate off and leave the liquid crystal unchanged while others, based on long chain alkanes for example, may be strongly retained. These facts have led to the use of cholesterics to detect chemical vapours.²⁴

5. Ultraviolet Light. Ultraviolet light irreversibly degrades many liquid crystals, resulting in a lowering of the transition temperatures and a colour shift to the blue. Prolonged exposure may destroy the liquid crystal phase altogether, although it should be noted that chiral nematics have a greater resistance to U.V. degradation than cholesteryl esters. Filters which cut off at 400nm may be used to enhance the light stability of the materials

FIGURE 9. Possible variations of reflected wavelength with temperature for cholesteric materials.

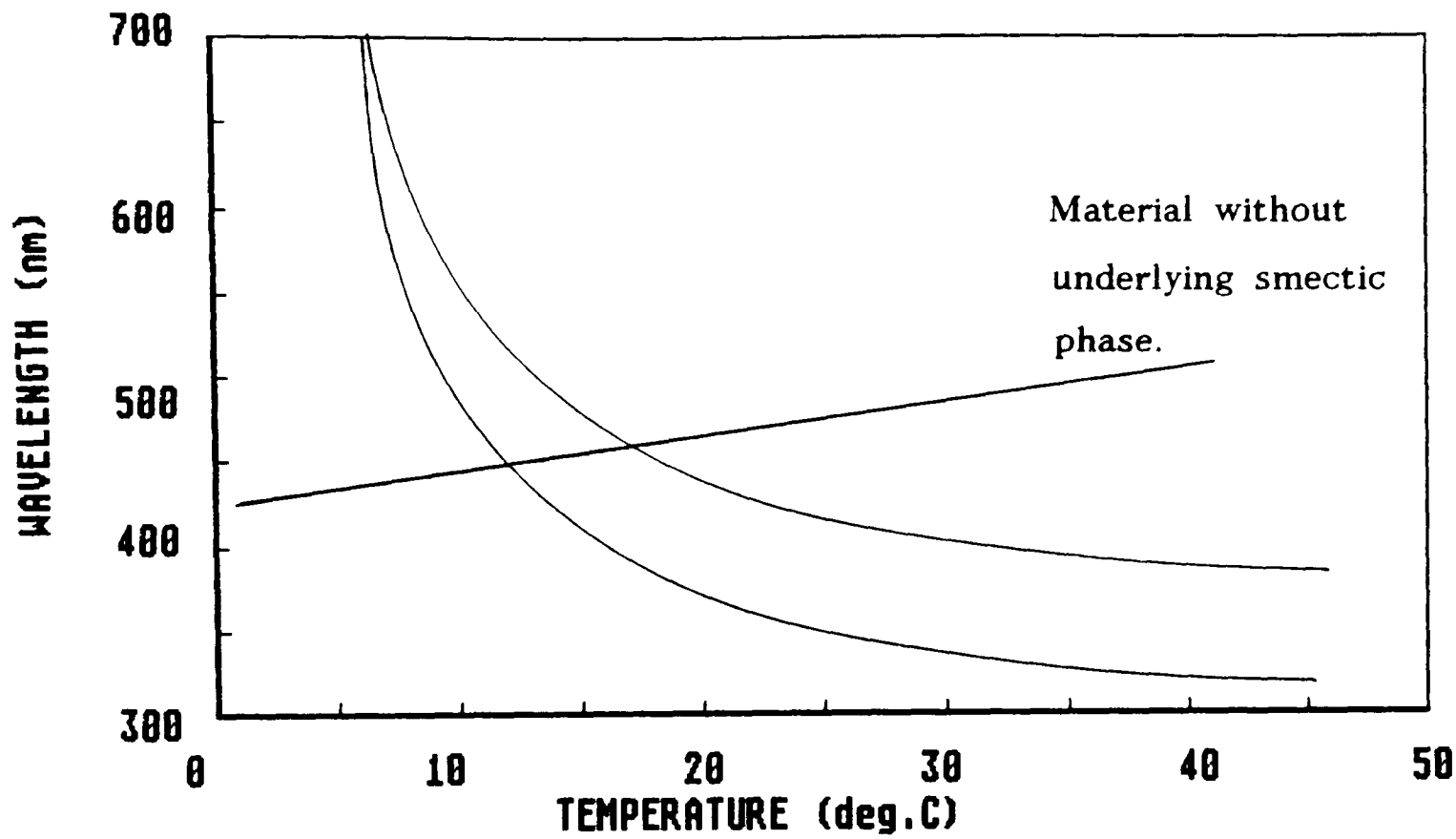
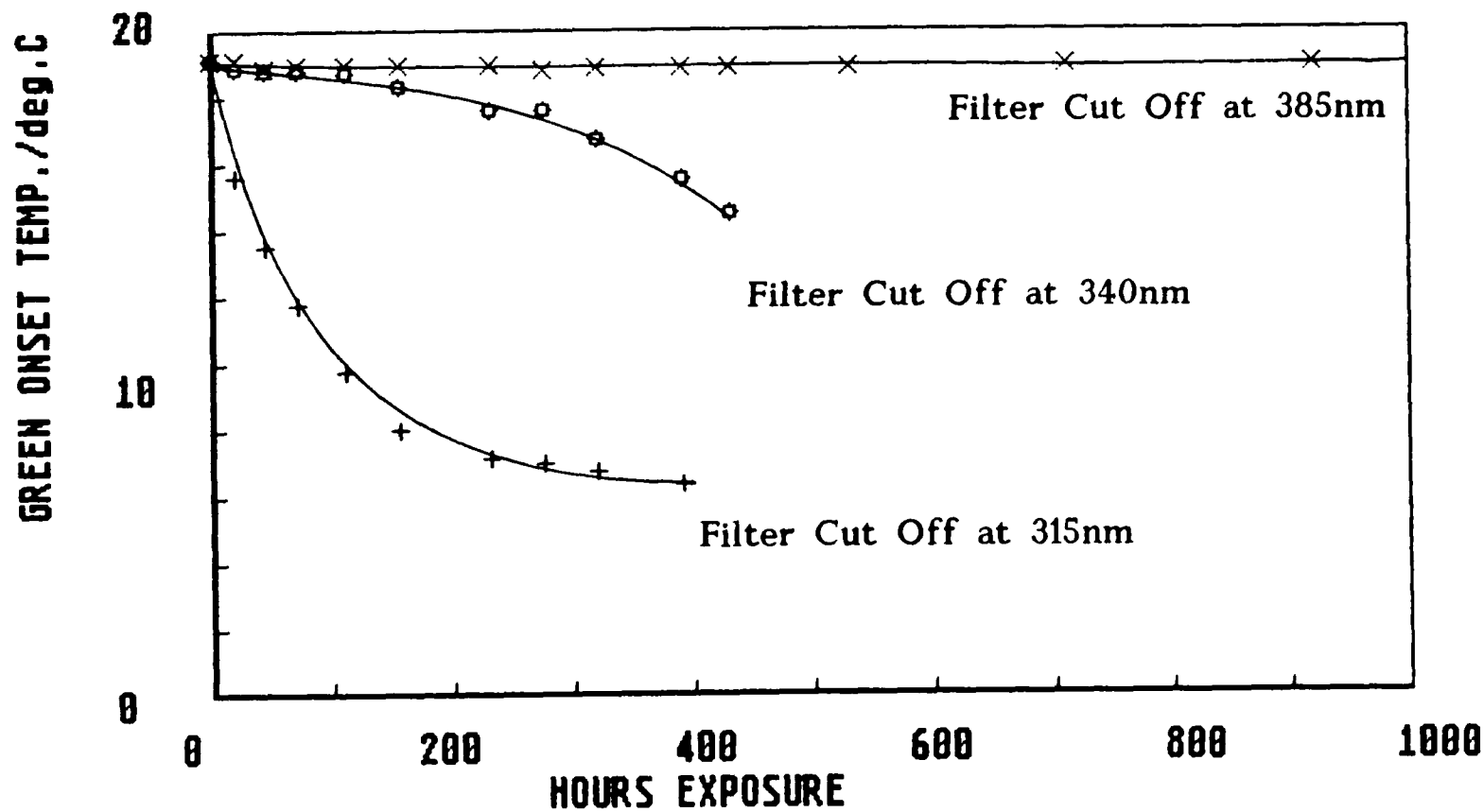


FIGURE 10. Lifetime of encapsulated chiral nematic liquid crystal materials when subjected to 10x bright sunlight conditions. Effect of filtering out the U.V. wavelengths is clearly shown.



and enhance contrast by removing the long blue 'tail' of the selective reflection. Figure 10 shows results obtained by the author demonstrating extended material lifetime when protected from U.V. radiation.

6. Electric and Magnetic Fields. Liquid crystals respond to electric fields and it is this property which is exploited in display devices. Electric fields of the order of 10^6 V/m are required to unwind the cholesteric helix²¹. These effects are considered to be outside the scope of this thesis. Further information may be found in the references mentioned at the end of this chapter.

The first three of these effects, which are of most relevance in aerodynamic testing are examined in greater detail in subsequent chapters.

2.4. Viscosity

The viscosity of liquid crystals is anisotropic and has been described^{22,23} by five coefficients, α_1 to α_5 , for nematics. These coefficients may be combined to describe any flow geometry. The coefficients are complex to measure and as an approximate guide the bulk flow aligned viscosity is generally used. For typical chiral nematics at 20°C this is in the range 10 to 100 cps. Cholesterics are non-Newtonian fluids with very shear dependant viscosities. The apparent viscosity for flow along the helical axis increases by about 10^6 as the shear drops to very low rates²⁵. For shear perpendicular to the helical axis the apparent viscosity is shear and pitch dependent. The viscosity is a very important parameter and has implications for most of the measurements carried out in subsequent chapters.

Studies of the viscosity of liquid crystals are made in chapters 3:6 and 4:3.

General introductions to liquid crystals and cholesteric liquid crystals may be found in references 24 to 30, and 1.

3. HEAT TRANSFER

3:1. HEAT TRANSFER

Heat may be transferred when a temperature gradient is present by three mechanisms, conduction, convection and radiation.

Conduction occurs when heat energy is transferred through a body by electrons and lattice vibrations for a conducting solid, and by phonons and molecular collisions for other solids and liquids. Conductive heat transfer is defined by the Fourier Equation, which for the one dimensional case is:

$$q = - K.A. \frac{\delta T}{\delta x}$$

q is the heat transfer rate, K the thermal conductivity, A the area and $\delta T/\delta x$ the driving temperature gradient. This may be used to derive the one dimensional heat conduction equation:

$$\frac{\delta^2 T}{\delta x^2} = \frac{\rho.c. \delta T}{K \delta t}$$

which may be used to solve unsteady heat conduction problems. c is the specific heat capacity and ρ is the material density.

Convection. Natural convection occurs in liquids and gases as the result of density differences created by a temperature gradient. The cooler, denser fluid has a greater gravitational force on it and so falls, while the hot fluid rises. Forced convection is the result of some external driving force, which results in higher gas velocities and hence higher heat transfer rates. Convection past a solid surface may cause heat conduction at the surface between the solid and the fluid. This heat transfer is dependent on the density

and velocity of the fluid. Convective heat transfer is defined by Newtons law of cooling:

$$q = h.A.(T_1 - T_2) \quad - - - 3.1$$

h is the heat transfer coefficient, and is dependant on the fluid properties and flow characteristics.

Radiation is the transfer of energy by electromagnetic waves. This is a complex effect, affected by surface emissivity and geometry. At 'normal' temperatures radiation is usually insignificant compared to conduction or convection. Radiative heat transfer is defined by:

$$q = F_e.F_a.\sigma.A.(T_1^4 - T_2^4)$$

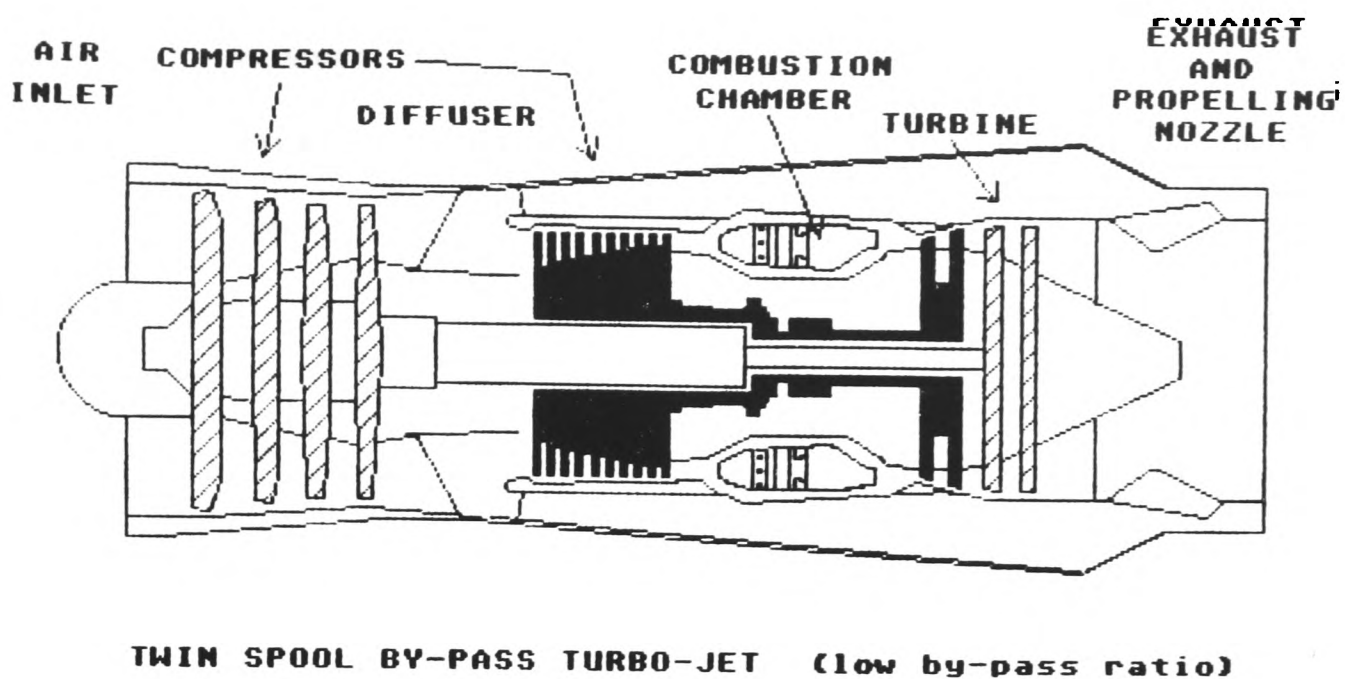
where σ is Stefan's constant, F_e is an emissivity function and F_a is a geometric view factor.

3:1.1. Application to Aerodynamics

A knowledge of the heat transfer characteristics of bodies is very important in aerodynamics, particularly in high speed and reentry situations and also in the jet engine environment. where many parts must operate at very high temperatures (1500°C). Efficient methods of cooling, which are essential to the design of energy efficient propulsion systems, can be assessed from measurements of heat transfer.

A schematic diagram of a typical gas turbine engine is shown in figure 11. The inlet air passes through a series of compressors. The pressurised air is then mixed with fuel and burnt in the combustion chambers. The expanding hot gases drive the turbine blades, which turn the compressors. The thrust is produced by the exhaust gases which exit at high velocity from

FIGURE 11. Schematic diagram of a modern gas turbine engine.



the rear of the engine. Fuel efficiency is achieved by using engines with a high bypass ratio, which increases the propulsive efficiency of the engine. In order to drive the bypass fan a core engine with a high specific output (and hence high turbine entry temperature) is required. In 1945 typical turbine entry temperatures were about 1000 K, but currently 1500 to 1800 K can be achieved. This has led to more fuel efficient and smaller engines, e.g. Boeing 757 engines consume 40% less fuel per passenger than its predecessor, the Boeing 727.

The improvement was brought about mainly by cooling the turbine blades with cool air from the compressors. The blades not only have to be kept well below their melting temperatures, but also the temperature must be even over the blades to prevent the stresses caused by temperature gradients.

There are three main methods of cooling the blades which are currently in practical aero engine use.

(i) Convection cooling, where cool air from the compressors is passed through channels in the blades. These channels may have complex arrays of ribs and pedestals to disturb the air flow and increase heat transfer

from the blades to the gas.

(ii) Film cooling, used in conjunction with convection cooling, where air is ejected from small holes in the blade to form a thin film of cool air over its outside surface.

(iii) Impingement cooling, where cool air is blown into the blade and then blown through small holes so as to impinge on the internal surface of the blade.

Plate 1 shows a cutaway photograph of a turbine blade showing the cooling channels, pedestals and film cooling holes.

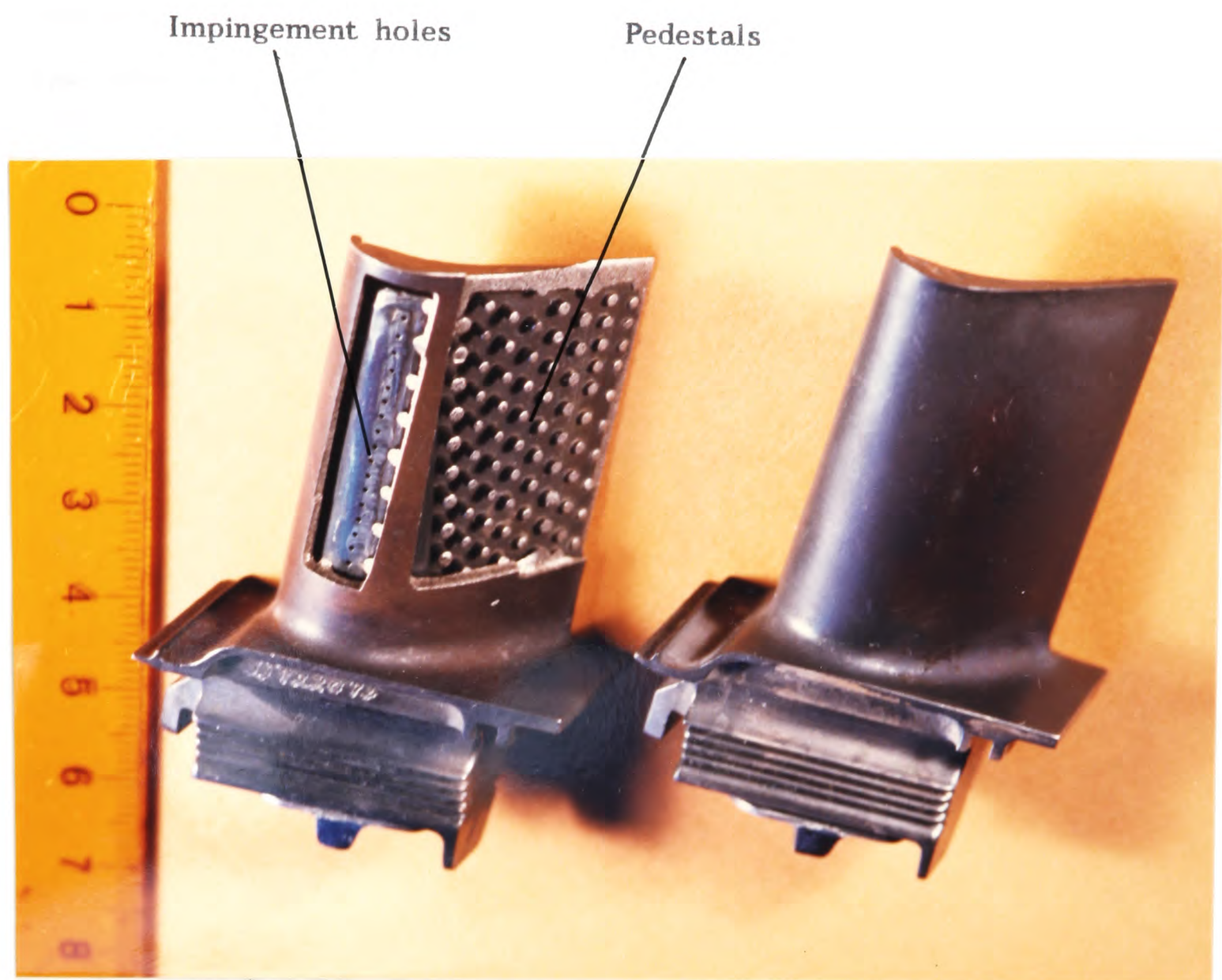
The cooling produced by these methods is too complex to calculate accurately, so scaled models are constructed and tested in wind tunnels. Tests have to be as thorough as possible before going to the expense of producing final blades for an engine. Turbine blades may rotate at around 1000mph while hot gas at 1700°C flows past them at 1700mph. Measurements clearly have to be precise to eliminate temperature gradients and cool the blades to their operating temperature of around 1200°C . Efficient cooling also prolongs the blade lifetime.

Accurate point temperature measurements on a model surface can be achieved with resistance thermometers, but much more information may be obtained from thermal mapping methods, which enable hot or cold spots to be seen and heat transfer to be visualised. Liquid crystals are not the only method of doing this, but unlike phase change paints, they are reversible and therefore enable real time and readily assessable measurement. Their limitations and advantages in this application will be discussed in section 3:2.

3:1.2. Determination of Heat Transfer

To determine the heat transfer coefficient at any point on the model it is necessary to know the elapsed time, the gas and model temperatures at that point and time, the initial temperature and the thermal properties of the

PLATE 1. Gas turbine blades. The cutaway blade clearly shows the internal pedestals used to enhance heat transfer, and the impingement cooling holes used to blow cool air from the compressors onto the blade inside surface at calculated hot spots.



model.

Most of the tests done at Oxford using liquid crystals are on perspex models due to the similarity of their thermal properties and ease of model manufacture. Run times are kept short to eliminate lateral conduction and limit penetration of the heating pulse into the material, producing transient surface temperature heat transfer. In practice hot flow is established quickly and the surface temperature monitored with the liquid crystal, for example. From the local temperature, the local heat transfer may be found. The heat conduction equation therefore reduces to the one dimensional case:

$$\frac{dT}{dt} = k \cdot \frac{d^2 T}{dx^2}$$

The heat transfer coefficient, h , can be defined from:

$$q = h \cdot (T_g - T_w)$$

where q is the heat transfer rate, T_g and T_w are the gas and wall temperatures, respectively.

The Laplace transform of this is

$$\bar{q} = \frac{h \cdot T_g}{s} - h \cdot \bar{T}_w \quad - - - 3.2$$

$$\text{At } x = 0, \quad q = -K \cdot \frac{dT_w}{dx}$$

Using the Laplace transform and solving, this becomes

$$q = \sqrt{\rho c K} \cdot \sqrt{s} \cdot \bar{T}_w \quad - - - 3.3$$

Combining 3.2 and 3.3 gives

$$\bar{T}_w = \frac{h.T_g}{s(\sqrt{\rho c K}.\sqrt{s}+h)} \quad - - - 3.4$$

$\sqrt{\rho c K}$ refers to the wall material. 3.4 may be inverted to give

$$\frac{T_w - T_i}{T_g - T_i} = 1 - e^{-\gamma^2} \cdot \text{erfc}(\gamma) \quad \text{where } \gamma = \frac{h.\sqrt{t}}{\sqrt{\rho c K}}$$

and T_i is the initial model temperature.

Hence the heat transfer coefficient can be calculated. If the model properties are constant with temperature a qualitative picture of the heat transfer pattern can be seen immediately from the model.

Details of this calculation may be found in Schultz and Jones³¹.

3.2. THE USE OF LIQUID CRYSTALS FOR HEAT TRANSFER MEASUREMENT

There are a number of ways to measure temperature using liquid crystals, but the most obvious is the use of the temperature dependent selective reflection colours shown by cholesterics. Other methods will be dealt with in a later chapter as possible improvements to this method.

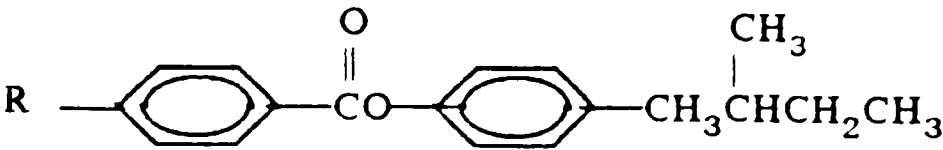
To achieve good temperature resolution materials are required which change colour quickly with temperature, hence cholesterics with underlying smectic phases are used. Figure 12 shows the general structure and colour/temperature calibration for the TM74/TM75 chiral nematic materials.³² These mixtures were widely used throughout this work since they were readily available from BDH Ltd, and offered a versatile system to achieve colour plays between -30 to +40°C. Each colour occurs at a specific temperature, but the yellow edge appears brightest to the human eye and usually occurs over a sharp, well defined temperature band, so this edge was selected to indicate an isotherm.

The colours may be seen with the liquid crystal either in its neat form or encapsulated.

In the neat form very bright colours may be produced and previous papers have reported very good spatial resolution^{33,10} . of the order of micrometers. However the layer thickness may be uncertain, and if used on exposed surfaces the material may be subjected to shear stresses³⁴. This can be overcome by covering the liquid crystal layer with a thin plastic film. This method was employed in the USA^{35,36} where resistive heating, black backing, liquid crystal and covering layers were all put on the model surface. This is quite an elaborate technique which introduces problems of constructing the multilayer and calculating its effects on the heat transfer.

This technique is being superseded by the use of encapsulated liquid crystals in a hot flow situation (i.e. hot gas passing over a room temperature

FIGURE 12. Structure and colour play characteristics of the cholesteric liquid crystals TM74 and TM75. These are benzoate ester derivatives of S(+)-4-(2' Methylbutyl) phenol. (Figure taken from BDH catalogue)



where R = C_nH_{2n+1}- ; C_nH_{2n+1}O- ; n = 0 to 3

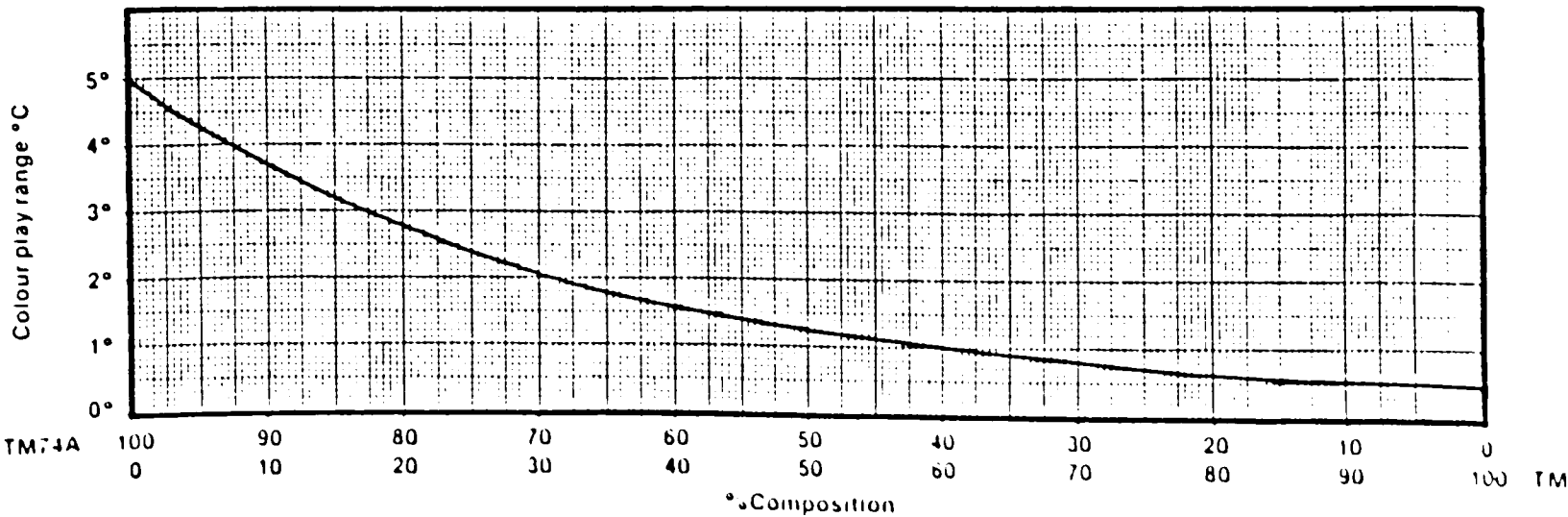
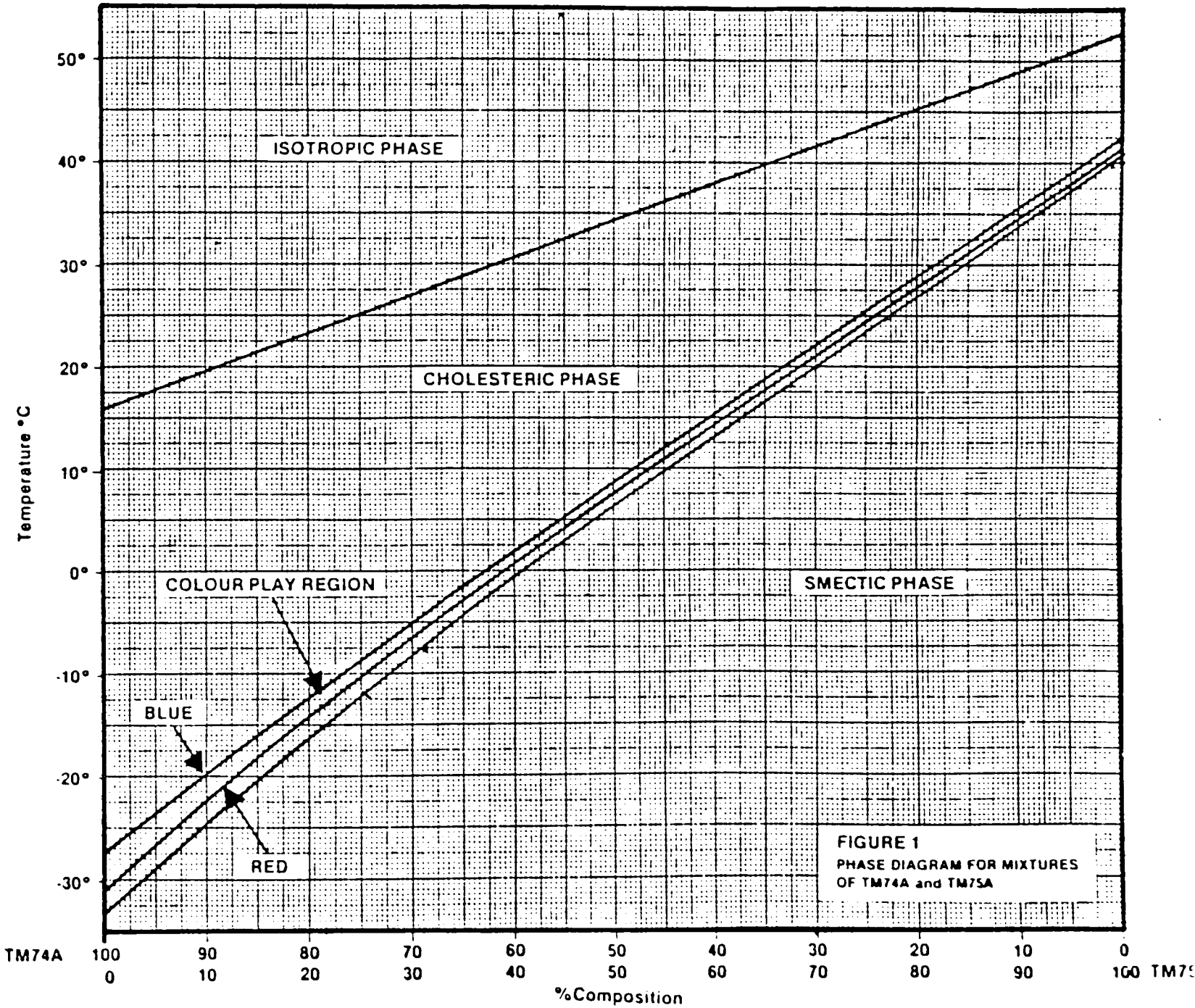
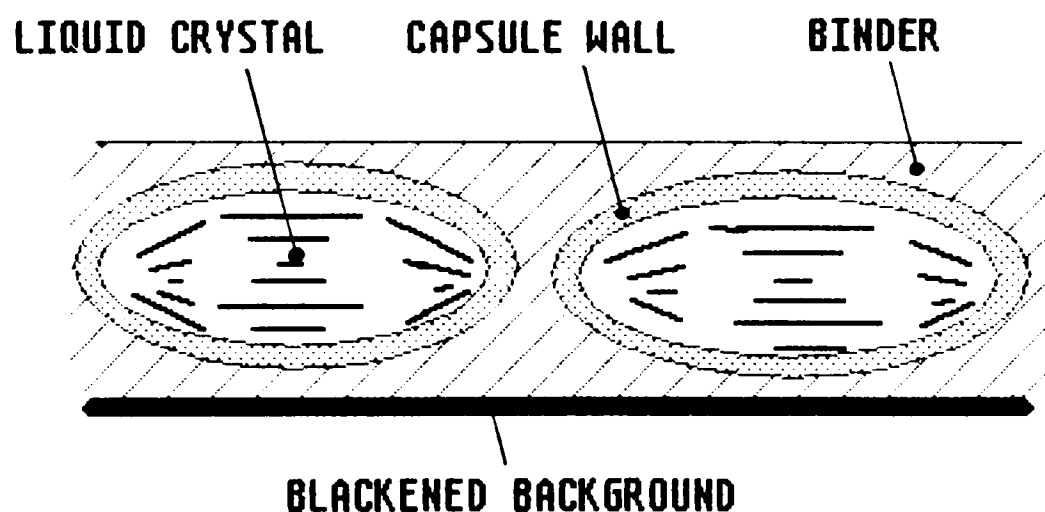


FIGURE 2
COLOUR PLAY RANGE FOR MIXTURES
OF TM74A AND TM75A

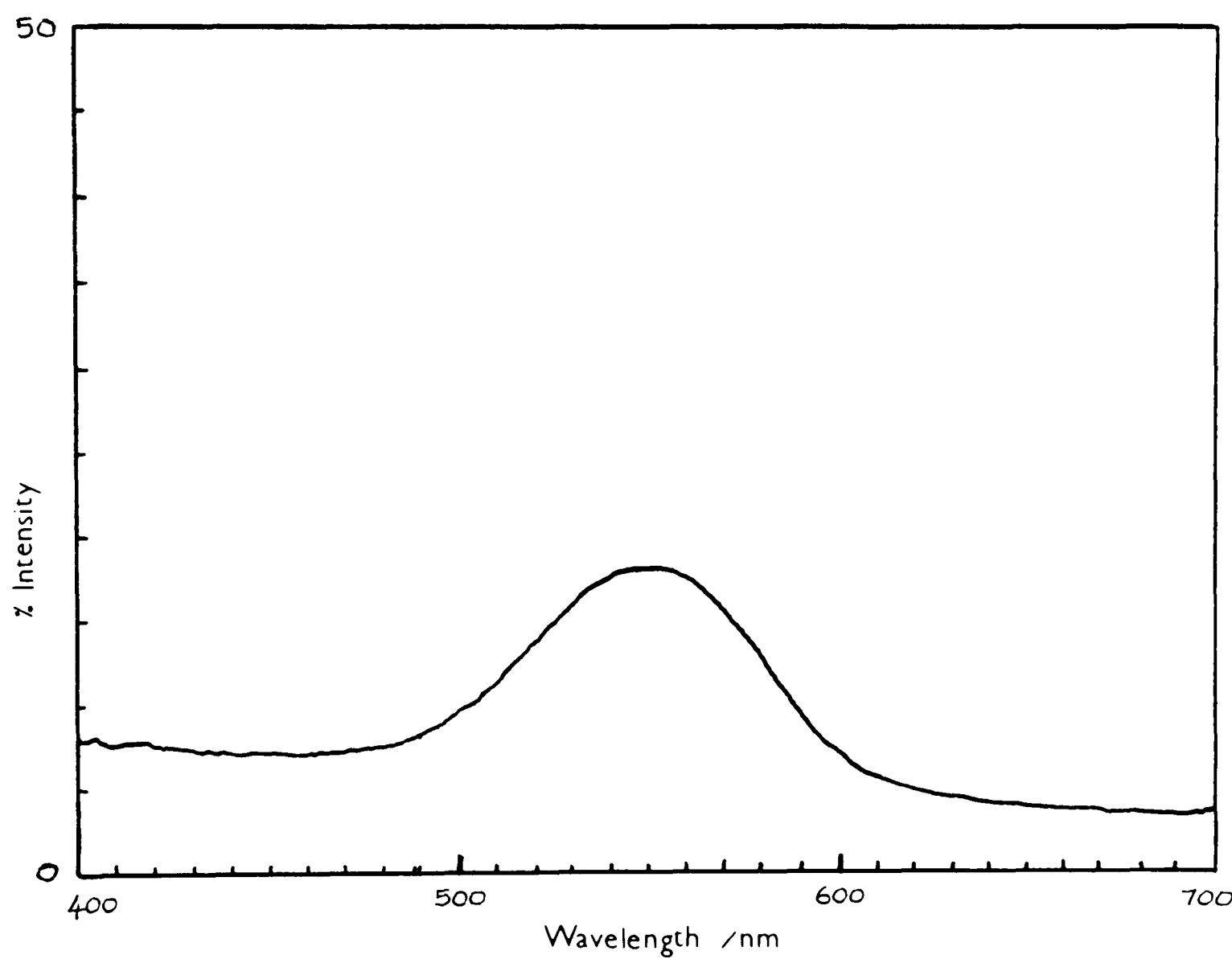
model). The liquid crystal is contained inside small ($\sim 10\mu\text{m}$ diameter) gelatin capsules dispersed in a polymer matrix³⁷. Encapsulation provides protection from shear forces and contaminants. Depending on the binder used the resulting layer can be fairly soft and water soluble (e.g. with PVA binder) or hard, flat and water resistant (e.g. Jon Cryll^{C2} or Mowlilith binders). Some work has been done on optimising the binder and at present the Mowlilith binder is giving the best results in terms of layer stability and brightness. Even layers may be achieved by silk screen printing or spraying on to blackened surfaces. To avoid solvent contamination from the black paint clear perspex models can be used with the black layer on the back. When this cannot be done it is best to avoid solvent based sprays as the solvent may affect the liquid crystal calibration. It is preferable to use a water based matt black paint.

FIGURE 13. Schematic diagram of dried capsules of liquid crystal applied to a flat surface.



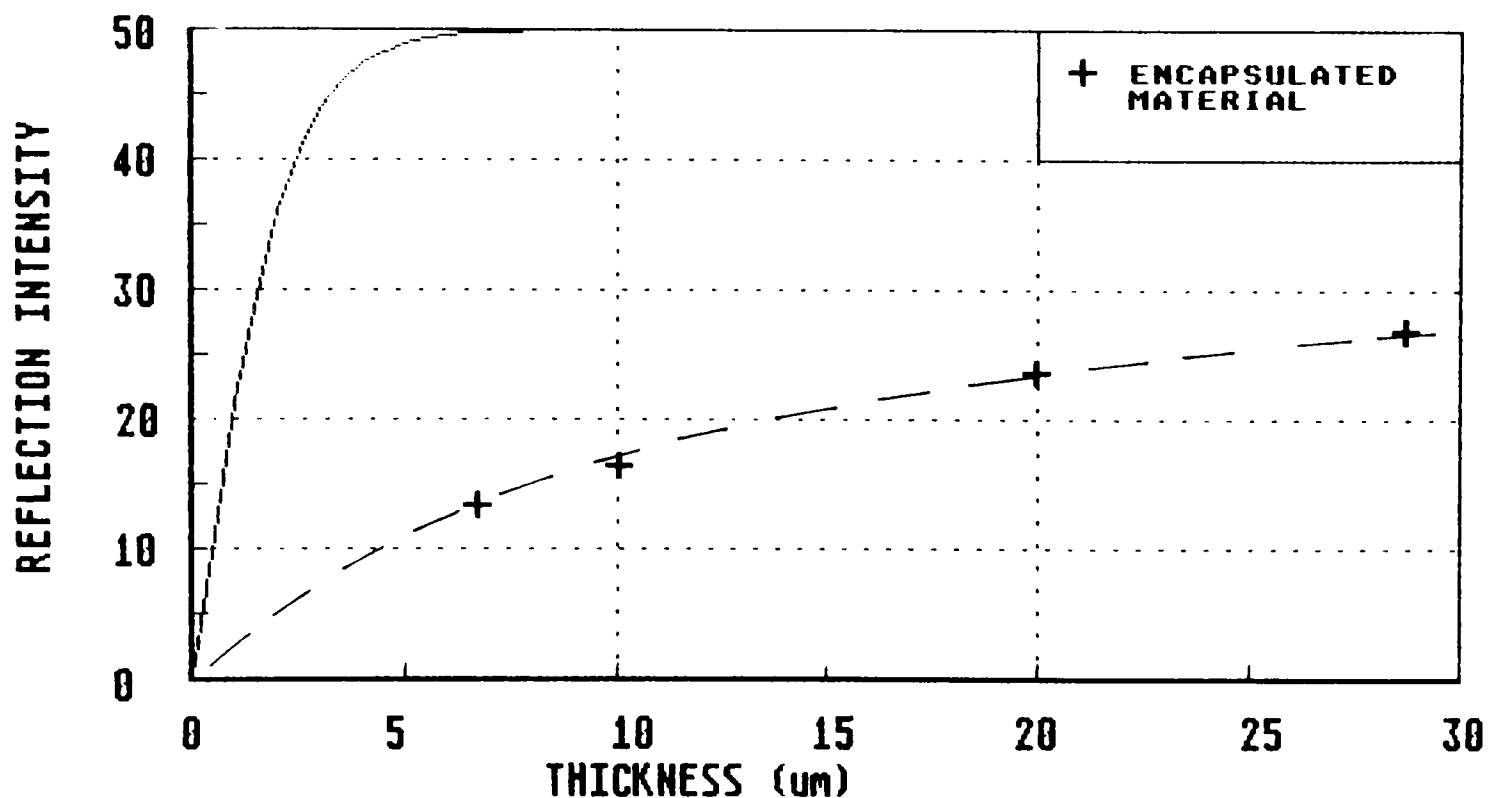
On drying the capsules deform into oblate spheroids giving some alignment to the liquid crystal, see figure 13. The degree of alignment is relatively low as can be seen by comparing a typical reflection spectra from an encapsulated layer (see figure 14, measured by the author) with a spectrum from a well aligned layer (figure 5, p.11). The reflected intensity is

FIGURE 14. Typical reflection spectrum for an encapsulated cholesteric liquid crystal material.



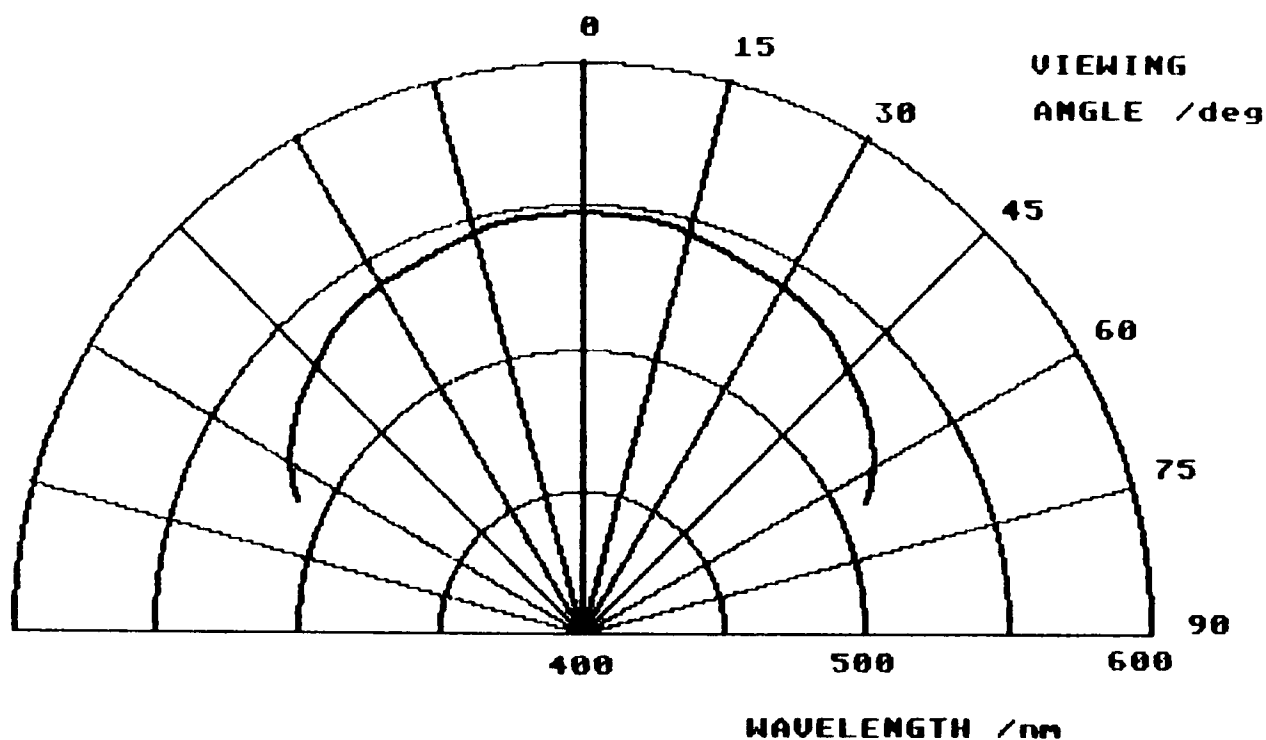
thickness dependent, as shown in figure 15. Saturation is not reached until around 30 μm , although a single 10 μm encapsulated layer will give reasonable brightness with the high Δn chiral nematic materials. Figure 15 shows the results for normal incidence illumination and viewing. The solid line is the thickness dependence for a well aligned neat liquid crystal layer and the points represent measurements made on an encapsulated layer of the same material.

FIGURE 15. Variation of reflected intensity with thickness for both encapsulated and neat liquid crystal layers.



Encapsulation also affects the angle dependence of the reflection peak, which is reduced due to the distribution in angle of the helical axes over the surface. This produces a wider (higher $\Delta\lambda$) reflection spectrum of lower intensity, as shown in figure 16.

FIGURE 16. Angle dependance of the peak reflected wavelength from an encapsulated cholesteric liquid crystal.



Encapsulation has the great advantage that two or more (up to five have been tried) encapsulated mixtures may be mixed together and sprayed onto a surface to achieve multiple colour bands at different temperatures. A consequence of using more than one mixture is the reduction in brightness resulting from the dilution in aspect ratio for a particular mixture. The reflection bands can be recorded using a video camera.

Care should be taken to avoid the other factors which may affect the pitch described in section 2, in particular strong or prolonged U.V. exposure. Two additional factors may affect encapsulated materials:.

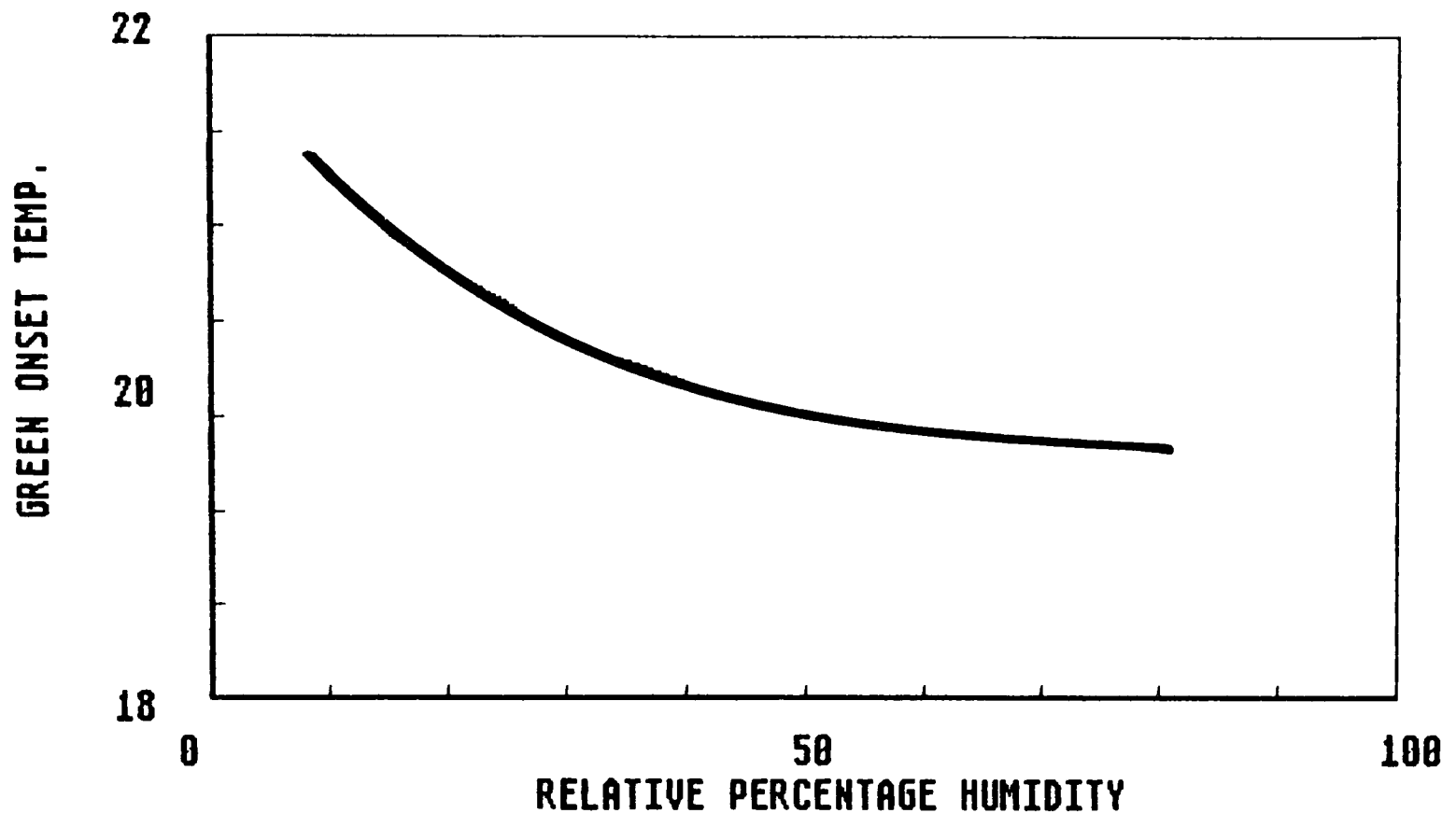
(a). Humidity. The gelatin/binder mixture may absorb moisture and expand, causing a reduction in pressure on the liquid crystals and decreasing the transition temperatures. A change of 2°C can result from a change of between 25 to 70 % relative humidity, as shown in figure 17, (measured by the author). These results were obtained using a PVA binder.

(b). Bacterial Growth. Gelatin is a very good culture for growing bacteria and even with antifungicides included in some encapsulated mixtures, the mixture lifetime will be prolonged if it is stored in a fridge.

Encapsulated cholesteric liquid crystals have been used very

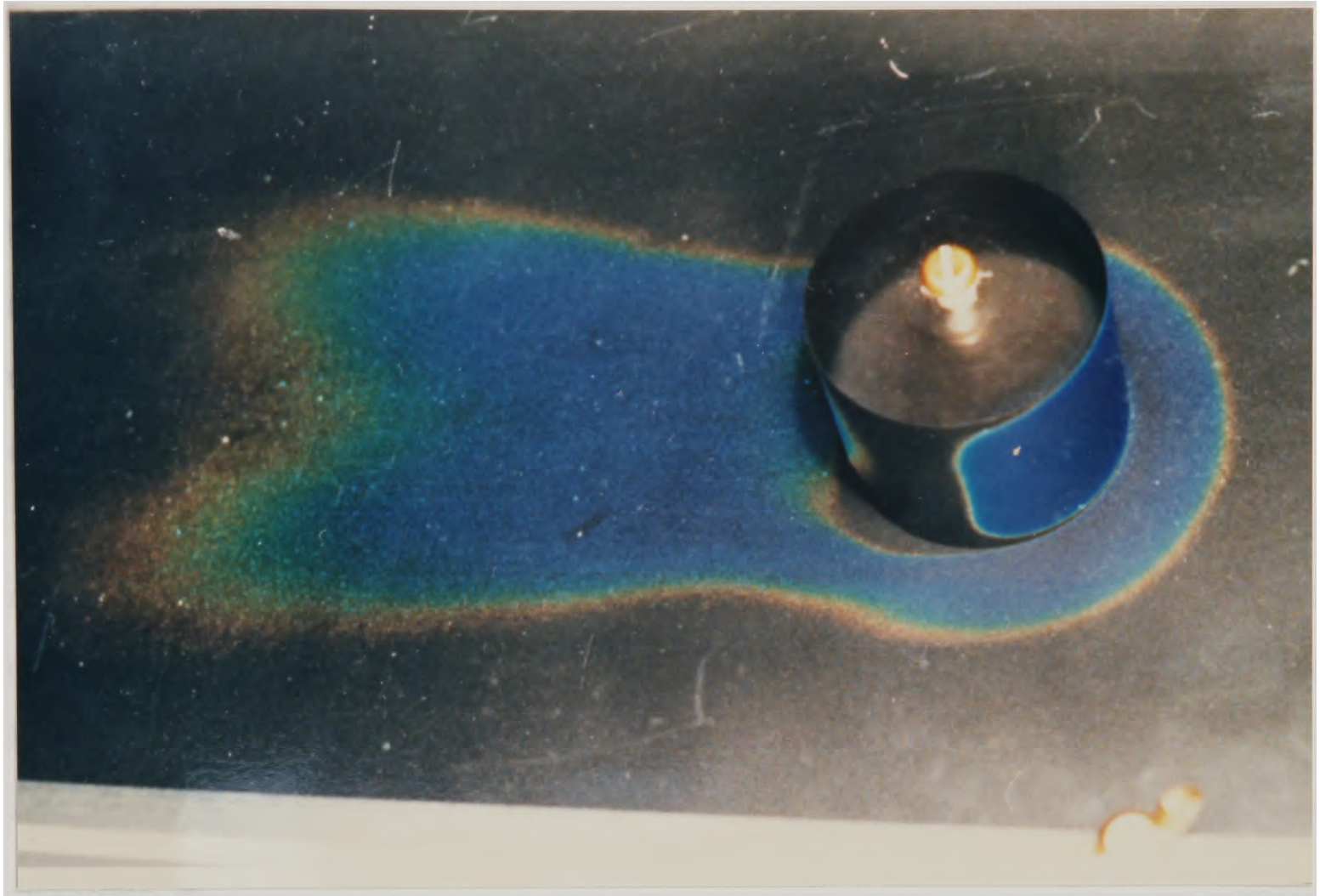
successfully at Oxford to measure surface temperatures in order to calculate

FIGURE 17. Variation of encapsulated liquid crystal colour play temperature with humidity.



heat transfer. Dr. P. Ireland has examined flow and heat transfer around pedestals³⁸ (see plate 2). Col. A. Byerley has looked at heat transfer around film cooling holes³⁹ and Mr. Z. Wang has examined the effects of surface roughness on heat transfer.⁴⁰ These tests were all done at low speeds with fairly low heat transfer rates present (<1000 W/mK). High speed tests in high heat transfer environments have been attempted but no reflection colours were observed. In this section the reasons for this problem are identified, examined and solutions proposed and developed. The most obvious areas for investigation were the *thermal response time* of the liquid crystal colour change and the effects of a *temperature gradient* on the selective reflection.

PLATE 2. Use of liquid crystals to visualise the surface temperature pattern associated with the horse shoe vortex flow around a pedestal. Hot gas is flowing from right to left. The pattern develops with time and allows the heat transfer coefficient to be mapped around the pedestal.



3:3. THERMAL RESPONSE TIME

The thermal response time of any temperature indicating device normally consists of two factors.

The first is due purely to thermal conduction and is the time taken for the device to heat up to the temperature it is meant to be measuring.

The second is intrinsic to the device and is the time taken for the device to indicate a certain temperature after it has reached that temperature. In the case of cholesteric liquid crystals this would be the time taken for the helical pitch to change to a different length corresponding to the change in temperature. This would involve molecular motion and may therefore be expected to be influenced by viscosity.

The thermal conduction time can be calculated fairly accurately from a knowledge of the thermal properties of the materials. Measurement of liquid crystal thermal properties is described in this thesis under 'Parameter Measurements'. Thermal conduction was then modelled according to the analysis of Carslaw and Jaeger⁴¹. The relevant equations are shown in Appendix B.

Much of the work at Oxford involves transient wind tunnel tests to measure surface heat transfer. In these tests it is necessary to measure the surface temperature history of the model. As the rate of change of surface temperature may be high, it is necessary to know the response time of the liquid crystals for use as a surface temperature thermometer.

Previous workers have reported thermal time constants for cholesteryl ester materials of the order of hundreds of milliseconds^{42,4}. More recent work at Oxford University⁴³ has measured the response time of 10 μ m encapsulated layers of chiral nematic liquid crystals as being of the order of 3ms. This indicated that the helical time lag plays a significant role

in the total response time of the layer, relative to the thermal conduction time.

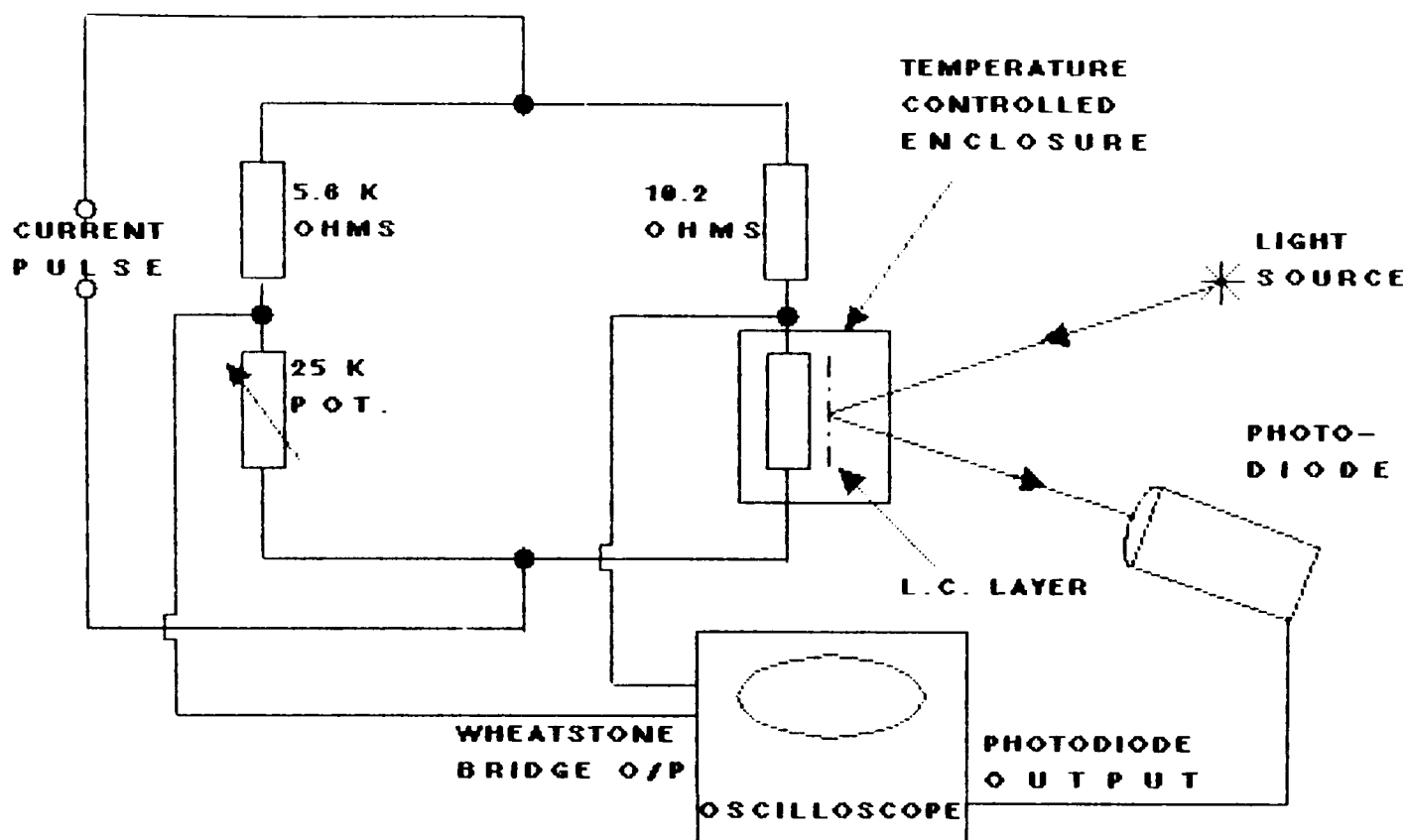
The aim of the present work was to extend the experiment conducted at Oxford and to calculate a time constant for the materials and investigate ways of improving it.

3:3.1. Time Constant Experiment

The apparatus used is shown schematically in figure 18. This comprised of a thin film resistance gauge, coated with liquid crystal, incorporated into a Wheatstone bridge circuit. By applying a DC current step the resistance gauge acted as a heater and raised the temperature of the liquid crystal through its colour play, which was monitored with a photodiode. The actual temperature of the gauge was determined from the out of balance voltage of the bridge, which resulted from the gauge change in resistance with temperature. The temperature coefficient of resistance for each gauge was known, and this enabled the gauge temperature to be found. The temperature varied essentially as the square root of time. The temperature of the liquid crystal layer was then calculated from a thermal modelling program and compared with the temperature as indicated by the photodiode output.

The other resistors in the Wheatstone bridge were chosen for their temperature stability, and hence didn't change with temperature. The gauge material was a thin layer of indium tin oxide ($10 \text{ } \Omega/\square$ ITO) on a glass substrate. These gauges were found to have a temperature coefficient of resistance of about $4 \times 10^{-4} \Omega/^{\circ}\text{C}$. and were used in preference to platinum gauges (which have a higher temperature coefficient of resistance) because they form a transparent film enabling a black absorbing layer to be applied to the back of the substrate rather than between the gauge and the liquid crystal layer, which would introduce an undesirable thermal resistance between the gauge and liquid crystal.

FIGURE 18. Schematic diagram of pulsing circuit for time constant measurement experiment.



The gauge was contained in an enclosure in which the temperature was controlled using a flow of dry nitrogen gas passed through a vortex tube and heater. The ambient temperature around the gauge could thus be maintained between -50 to 60°C to within $\pm 0.2^{\circ}\text{C}$.

The gauge was illuminated through the glass front face of the enclosure by a 55W halogen lamp, driven by a D.C. power supply. The light was first passed through a filter which cut out infra-red and ultra-violet light to limit heating and degradation of the liquid crystal. The light reflected from the liquid crystal layer was focused onto a silicon photodiode (RS 308-067) which had a much faster response time ($\sim 30\mu\text{s}$) than the times being measured in the experiment.

When the current pulse was applied to the bridge the gauge temperature rose and produced an output from the bridge as described in figure 19. The gauge temperature was thus known continuously. It was not always possible to perfectly balance the bridge each time so some offset was

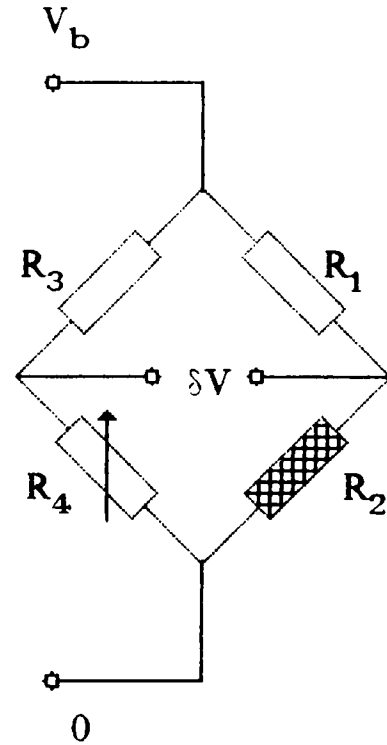
FIGURE 19. Method of calculating the temperature rise of the gauge from the output of the Wheatstone bridge.

The bridge is initially balanced, hence:

$$\frac{R_3}{R_4} = \frac{R_1}{R_2}$$

If a voltage V_b is applied across the bridge, the resistance of R_2 changes to $R_2 + \Delta R_2$ and an output δV is produced from the bridge.

$$\frac{\delta V}{V_b} = \frac{R_4}{R_3 + R_4} - \frac{R_2 + \Delta R_2}{R_1 + R_2 + \Delta R_2}$$



but as $\Delta R_2 \ll R_1$ and R_2 :

$$\frac{\delta V}{V_b} = \frac{R_1 \Delta R_2}{(R_1 + R_2)^2}$$

by definition : $\alpha \Delta T = \frac{\Delta R_2}{R_2}$ hence : $\Delta T = \frac{1 \cdot \delta V \cdot (R_1 + R_2)^2}{\alpha \cdot V_b \cdot R_1 \cdot R_2}$

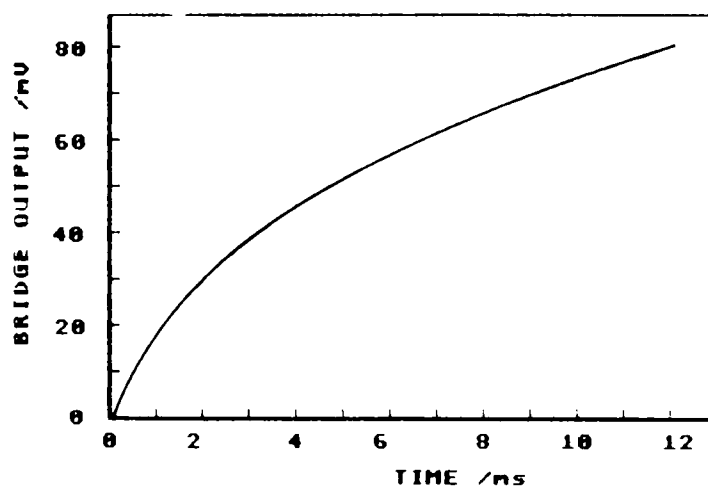
V_b is the voltage across the bridge δV is the bridge output voltage

ΔR_2 is the resistance change in R_2 due to resistive heating

α is the temperature coefficient of resistance of gauge R_2

ΔT is the temperature rise of gauge R_2

The output of the bridge, and hence the gauge temperature, rises in a parabolic manner over short times.



present on the bridge output. Calibration plots of bridge output against $\sqrt{t}$ had to be made in order to determine the starting point of the heating curve. Over the range of times investigated the heating curve was, to a first approximation, parabolic, hence the gauge temperature could be obtained from:

$$\Delta T = K.P.\sqrt{t}$$

where P is the pulse power in Watts and K is a constant, determined from the bridge output.

The current pulse was generated by electronics specially designed and produced at RSRE. This pulse box provided a set voltage for a set time. The maximum output was 10 Joules for a 10 Ω resistance.

The liquid crystals used were mainly mixtures of TM74A and TM75A available from BDH Ltd.^{C1} Hallcrest^{C3} also provided some cholesteryl ester mixtures for purposes of comparison.

A steady state calibration was done initially to determine the temperature of the liquid crystal which produced a maximum output from the photodiode, looking at light reflected from the liquid crystal layer. The TM74/75 materials were mixed to give a narrow colour play band at a number of temperatures between -30 to 40°C. The ambient temperature was set 5°C below the Sm-Ch transition temperature in all the pulse experiments. Thus a particular mixture, e.g. mixture A, having a colour onset at 42.6°C would be cooled to 37.6°C before pulsing. The heating pulse would be applied and after a certain time the colour display would be observed by the photodiode. For this mixture the photodiode produced a peak output corresponding to the equilibrium colour of the mixture at 43.4°C. The measured time delay before the photodiode peak occurred increased with mixture viscosity. The gauge was then pulsed at a range of pulse powers and the bridge output ($\propto$ gauge temperature) and photodiode output were displayed on a storage oscilloscope.

This produced heating curves as indicated in figure 20, from which a time constant must be calculated.

The parabolic temperature rise of the gauge was calculated from the bridge output, giving the gauge temperature, T_g , as shown in figure 20.

$$T_g = \frac{\delta V}{V_b} \frac{(R_1 + R_2)^2}{R_1 R_2 \alpha} = K.P.\sqrt{t}$$

This temperature history was used to calculate the thermal conduction into the liquid crystal layer, and hence to compute the liquid crystal temperature, T_{lc} , at the outer edge of the layer. This temperature can be approximated by a delayed parabola starting from a time t_c , as shown schematically in figure 20.

$$\text{Thus } \Delta T_{lc} = K'.P.\sqrt{t-t_c}$$

K' is the constant for this heating curve.

In the following derivation, times are measured from t_c , and are therefore given by:

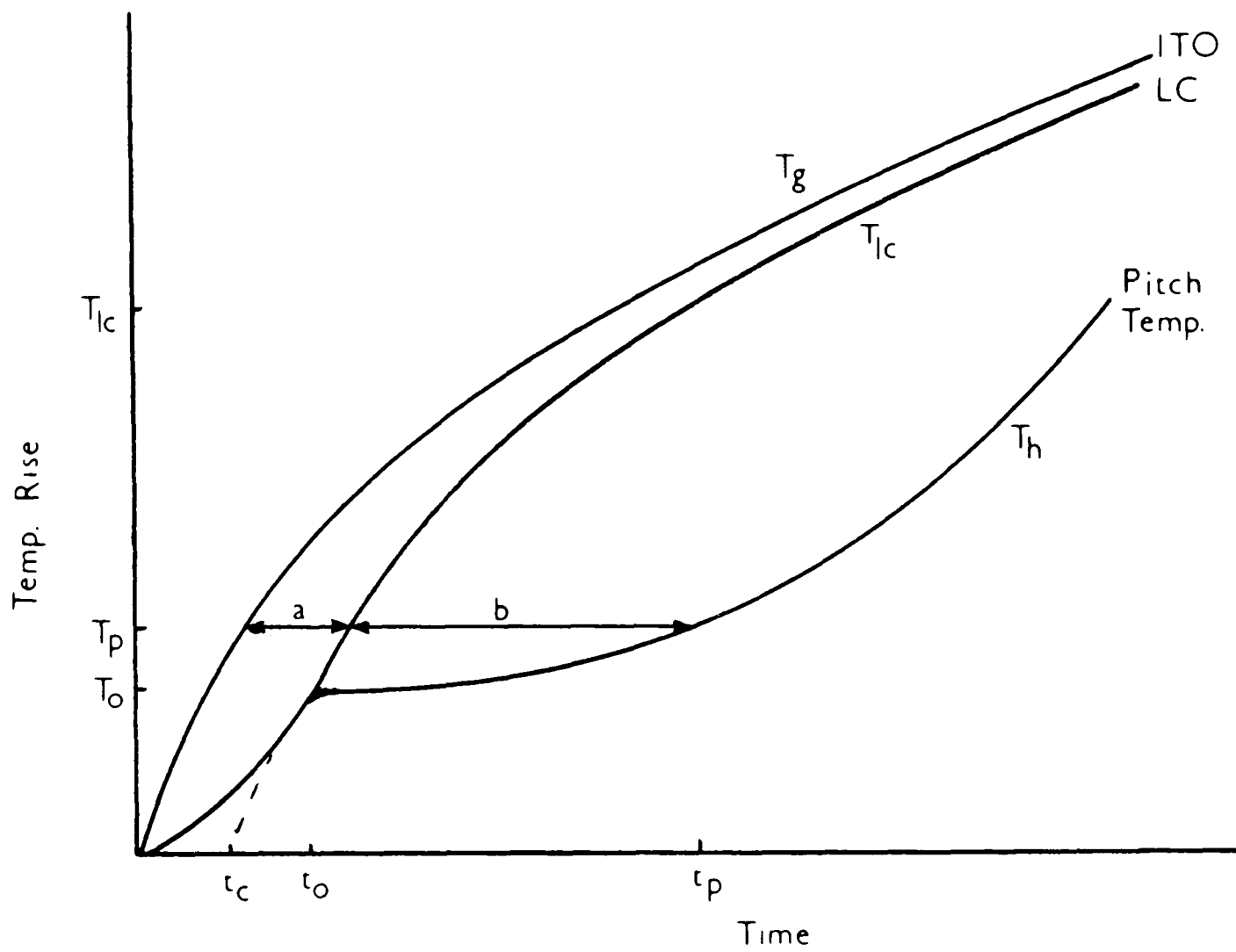
$$t' = t - t_c$$

The Sm-Ch phase change occurs after a time t_o , when the temperature has risen to T_o (i.e. by 5°C). The peak photodiode output occurs at time t_p , when the liquid crystal colour indicates a temperature T_p (which is known).

It is a simple matter to measure a time constant when a step change in the required condition occurs, but in this case a parabolic temperature rise is produced. The aim is to determine the thermal time constant under these conditions.

A standard relaxation time constant equation was used to describe the response of the liquid crystal. This equation assigns a temperature, T_h , to represent the helical pitch of the liquid crystal and has a value equal to the

FIGURE 20. Schematic of the temperature changes with time occurring when an ITO layer, covered with encapsulated liquid crystal, on a glass substrate, receives a current pulse. Also shows temperature lags at colour peak, T_p , 'a' due to thermal conduction and 'b' due to thermal response time of cholesteric helical structure.



temperature that the liquid crystal would have for this pitch if it were in equilibrium. Thus $T_{lc}-T_h$ is the temperature difference "driving" the helical structure towards equilibrium. T_p is the helix temperature observed when the liquid crystal gives peak reflection as detected by the photodiode.

$$\text{Hence } \frac{dT_h}{dt} = \frac{T_{lc} - T_h}{\tau}$$

integrating:

$$\left[T_h \right]_{T_o}^{T_p} = \int_{t_o}^{t_p} \frac{K' \cdot P \cdot \sqrt{t'}}{\tau} dt - \int_{t_o}^{t_p} \frac{T_h}{\tau} dt$$

T_h as a function of time is unknown. A first approximation would be to make it equal to T_o , but it is slightly more accurate to make it a linear function between T_o and T_p . This is a reasonable assumption if a narrow colour band mixture is used and the photodiode peak occurs soon after the material becomes cholesteric.

$$\text{Let } T_h = T_o + C.(t'-t'_o) \text{ where } C = \frac{(T_p - T_o)}{(t_p - t_o)} \text{ is a constant.}$$

$$\text{Hence } T_p - T_o = \int_{t_o}^{t_p} \frac{K' \cdot P \cdot \sqrt{t'}}{\tau} dt - \int_{t_o}^{t_p} \frac{T_o}{\tau} dt - \int_{t_o}^{t_p} \frac{C.(t'-t'_o)}{\tau} dt$$

$$\tau(T_p - T_o) = K' \cdot P \cdot \frac{2}{3} (t_p'^{3/2} - t_o'^{3/2}) - T_o(t_p' - t_o') - C \left[\frac{1}{2} (t_p'^2 - t_o'^2) - t_o'(t_p' - t_o') \right]$$

$$\frac{\tau \cdot (T_p - T_o)}{P \cdot t_o^{3/2}} = K' \cdot \left[\frac{2}{3} \left(\frac{t_p'}{t_o'} \right)^{3/2} + \frac{1}{3} - \frac{t_p'}{t_o'} \right] - t_o'^{1/2} \cdot C \cdot \left[\frac{1}{2} \left(\frac{t_p'}{t_o'} \right)^2 + \frac{1}{2} - \frac{t_p'}{t_o'} \right]$$

- - - 3.5 -

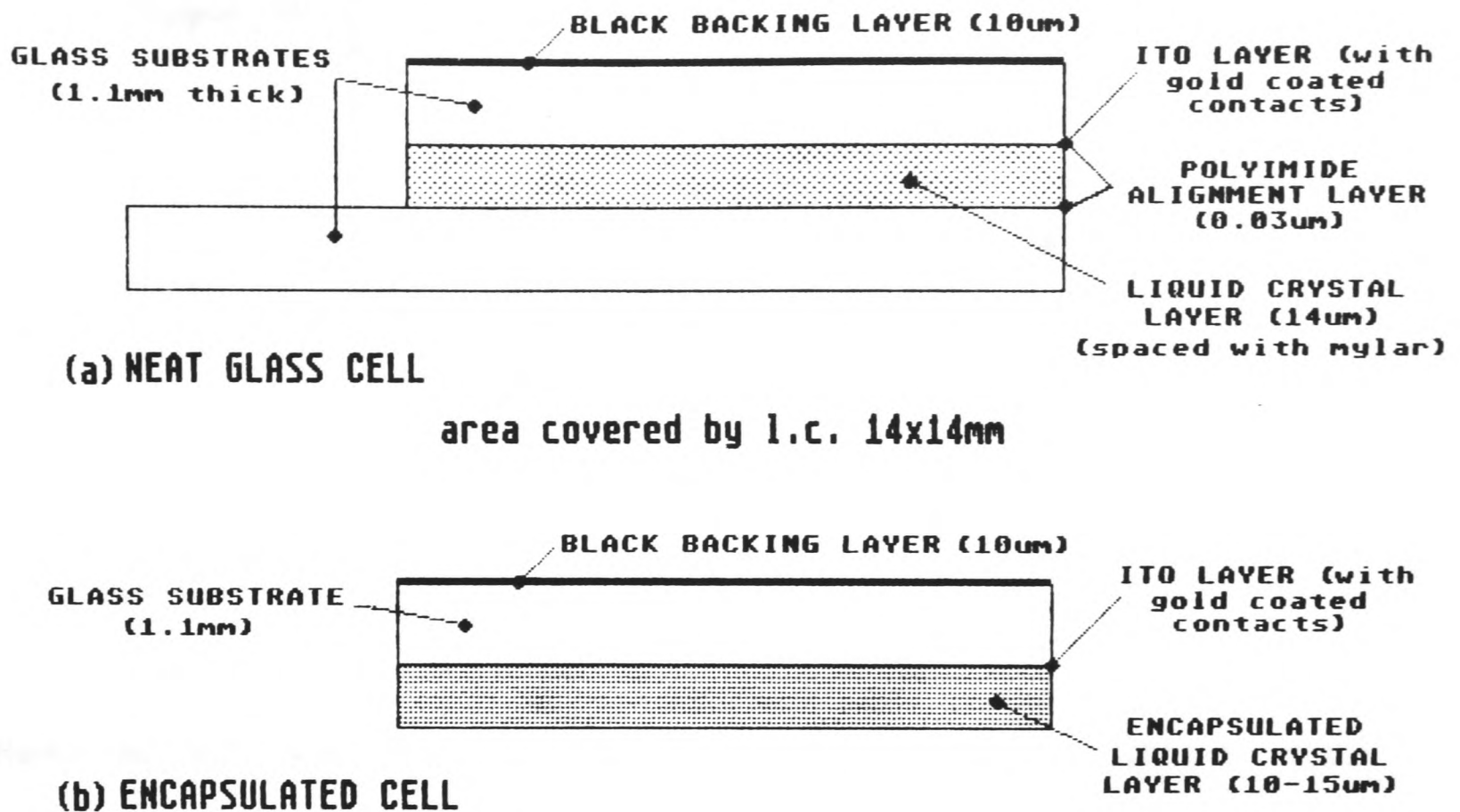
t_p' is measured from the photodiode output, along with the modelled heating curve for the liquid crystal layer, which also gives t_o' and K' . The temperatures T_p and T_o are measured for each material on the Mettler and by equilibrium calibration of the photodiode. The pulse power, P , is set on the pulse box for each experiment. Hence the only unknown in the above equation is the time constant, τ . This was found using a suitable plot of equation 3.5 to produce a straight line of slope τ for various pulse powers.

The final term in this equation is the difference between assuming that $T_p = T_o$ and that T_h is linear between T_o and T_p , and represents a difference of nearly 7% in the result for the time constant, τ . The average time constant was found for a range of pulse powers.

Initially it was intended to use two types of sample. The first of these involved neat liquid crystal mixtures in glass cells which were spaced with mylar spacers and resulted in cells 14 μ m thick. The spacers separated two 1.1mm thick pieces of glass, which had ITO and planar alignment coatings on the inner surfaces. Connections to the ITO were made via vacuum deposited gold contacts. The alignment layer, rubbed polyimide, was only about 300 angstroms thick so had a negligible effect on heat conduction. The back of the cell was coated with matt black paint. See figure 21.(a).

The second sample type was encapsulated liquid crystal mixtures which were silk screen printed on to 1.1mm thick ITO coated glass. Contacts to the ITO were again made via gold electrodes and the reverse of the cell coated with matt black paint. The thickness of the dried encapsulated layer was measured using a z-axis microscope. See figure 21.(b).

FIGURE 21. Schematic diagram of the glass cells used to pulse (a) the neat liquid crystal and (b) the encapsulated liquid crystal material.



The temperature coefficient of resistance of each ITO gauge was measured in a temperature controlled bath.

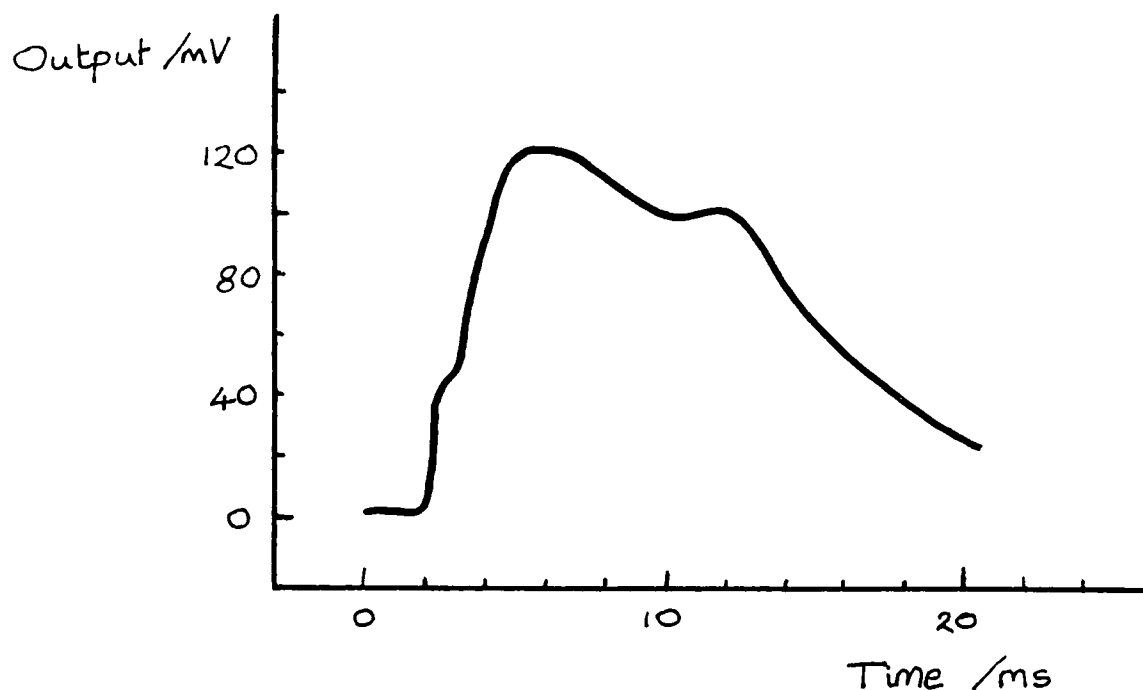
Results

Results are presented for both neat and encapsulated samples. It was found that the glass cells did not produce reliable results due to flow in the cells. Good results were obtained, however, for the encapsulated samples where flow of the liquid crystal was much more restricted.

3:3.2. Neat Material in Glass Cells

The photodiode output traces for the neat material showed some structure, usually three peaks, as accurately reproduced in figure 22. As the pulse power increased the peaks became lower and occurred at shorter times for a given mixture. At lower temperatures, when the mixtures had higher viscosities, the peaks occurred after longer times, and were low and broad.

FIGURE 22. Typical photodiode output for pulsed neat cholesteric liquid crystal in glass cell. (15 volt pulse applied for 20 ms)



Hence at high heat pulse energies with viscous materials (at low initial temps.) the peaks became difficult to discern. The steady state calibration, shown in figure 23, did not show as many peaks, so attempts were made to identify the extra peaks with specific colours or phase changes, using three methods:

.(i). Colour filtering of the reflection only produced a drop in the intensity of the photodiode output, without changing its shape, indicating that pure single colours were not present.

.(ii). High speed photography of the liquid crystal layer revealed that different colours were present at the same time, see plate 3. The colours appeared to be due to flow of the material towards the two open ends of the cell, creating a discontinuity along the centre. The flow properties of liquid crystals are quite complex, and have an effect on the reflected colour.

.(iii). High speed photography through a microscope while the cell was being pulsed also indicated a very disordered texture, see plate 4, with no dominant colour and no extensive region of Grandjean texture.

Thus in the case of neat liquid crystal samples complex flow

PLATE 3. Frame from a high speed photography sequence showing how flow effects can alter the colour of neat liquid crystal in a glass cell when it is subjected to a sudden heat pulse.

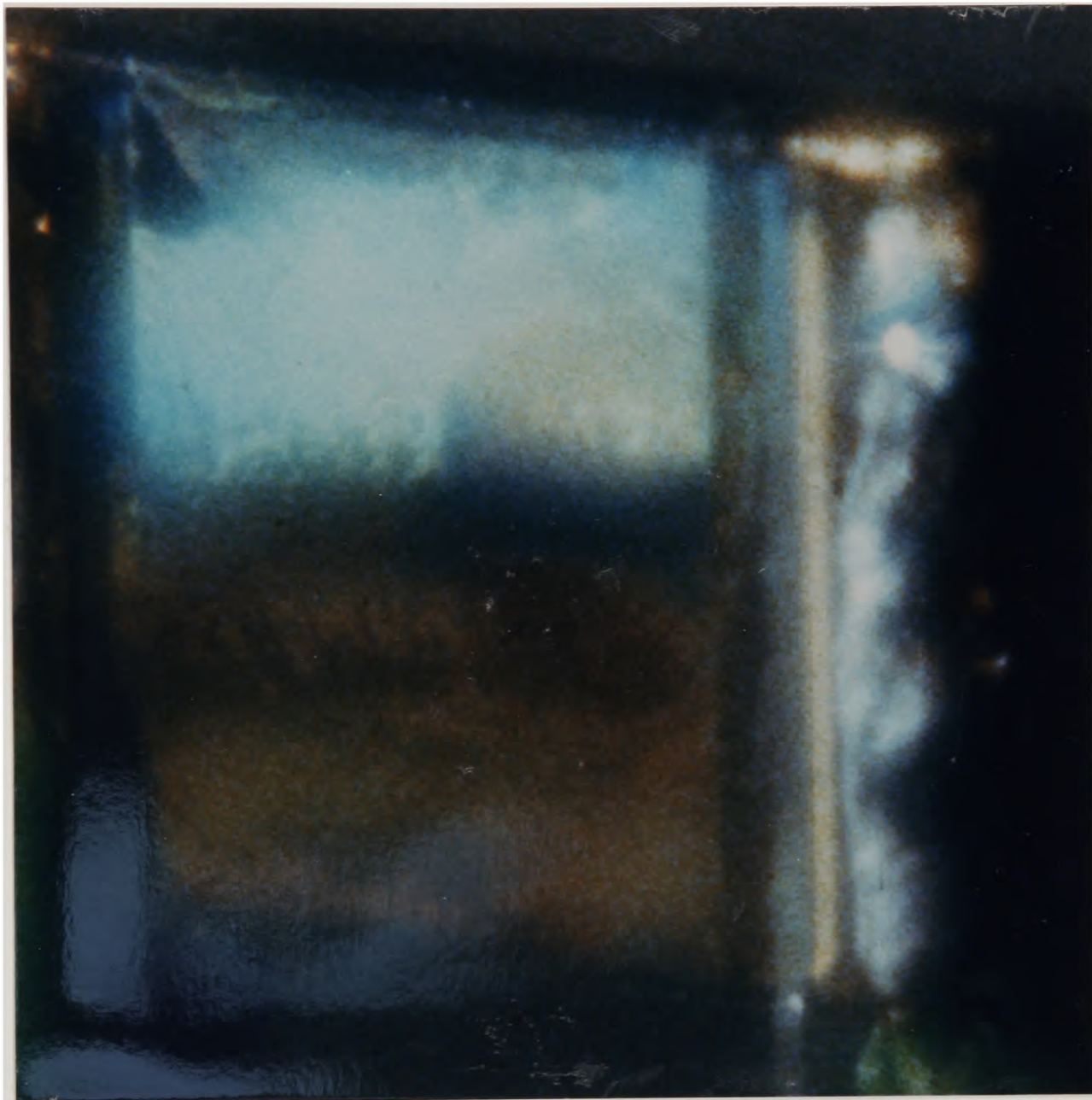


PLATE 4. Frame from a high speed photography sequence shot through a microscope. The neat cholesteric liquid crystal is viewed in a glass cell in transmission as it is being subjected to a heat pulse. Clearly good single crystalline alignment does not exist.

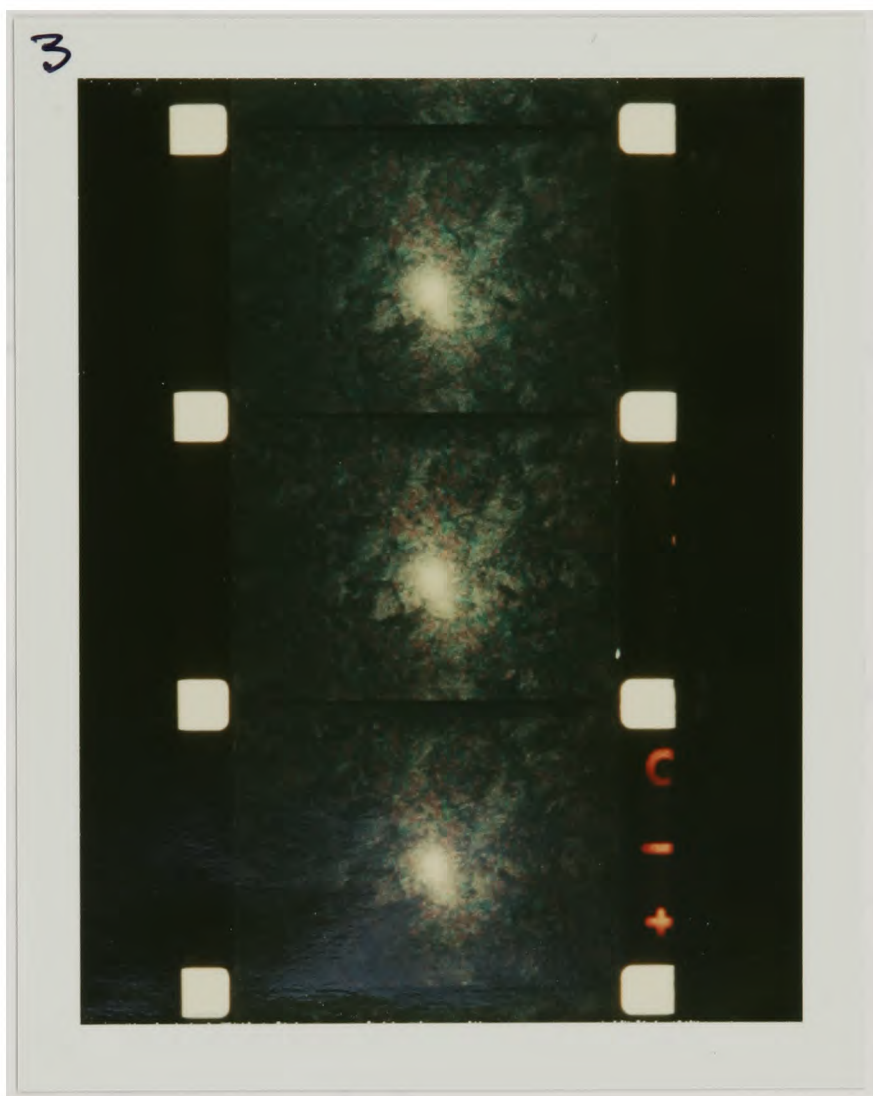


FIGURE 23. Calibration of photodiode output for neat material in glass cell. Peak occurs in yellow band.

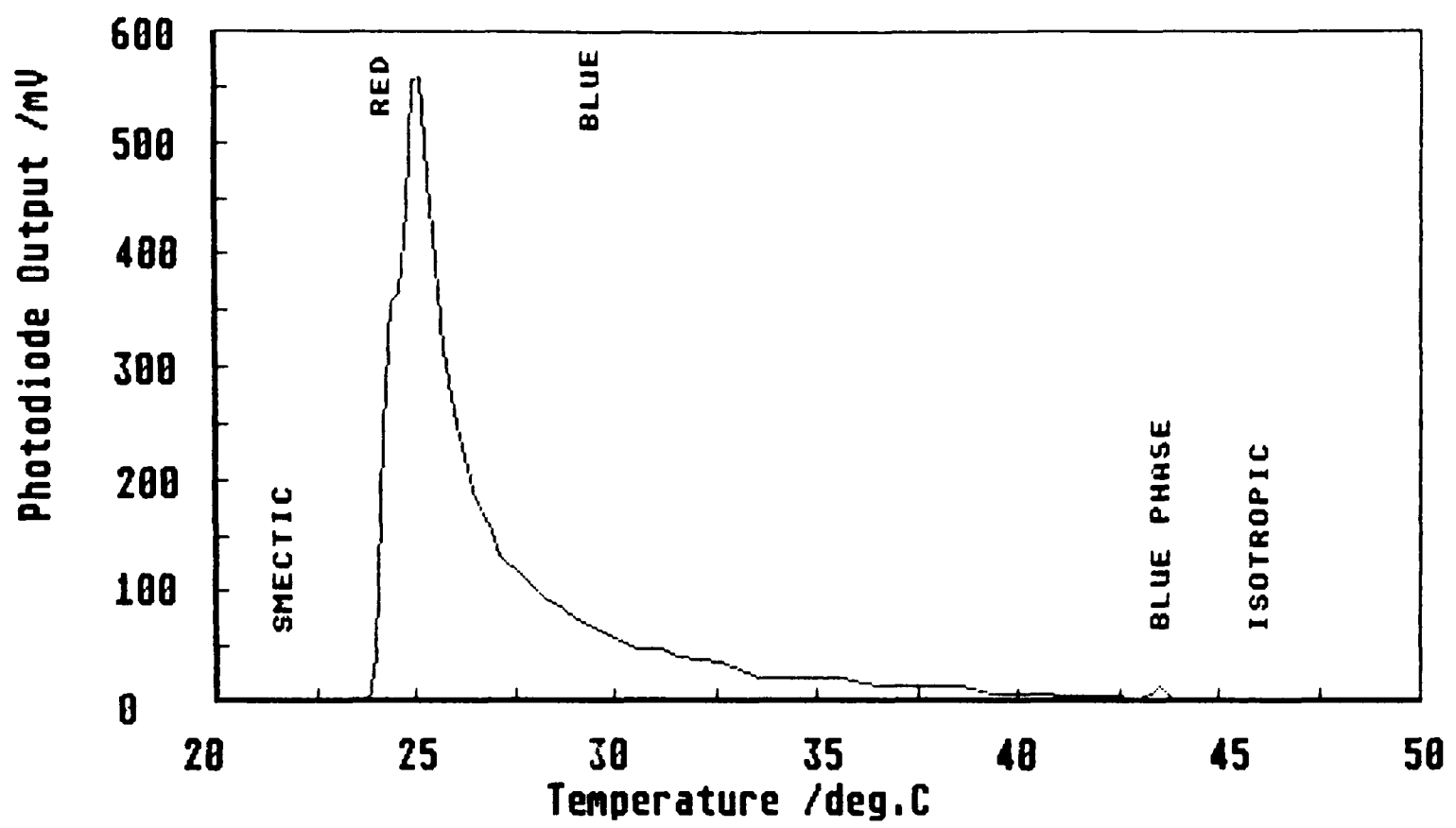
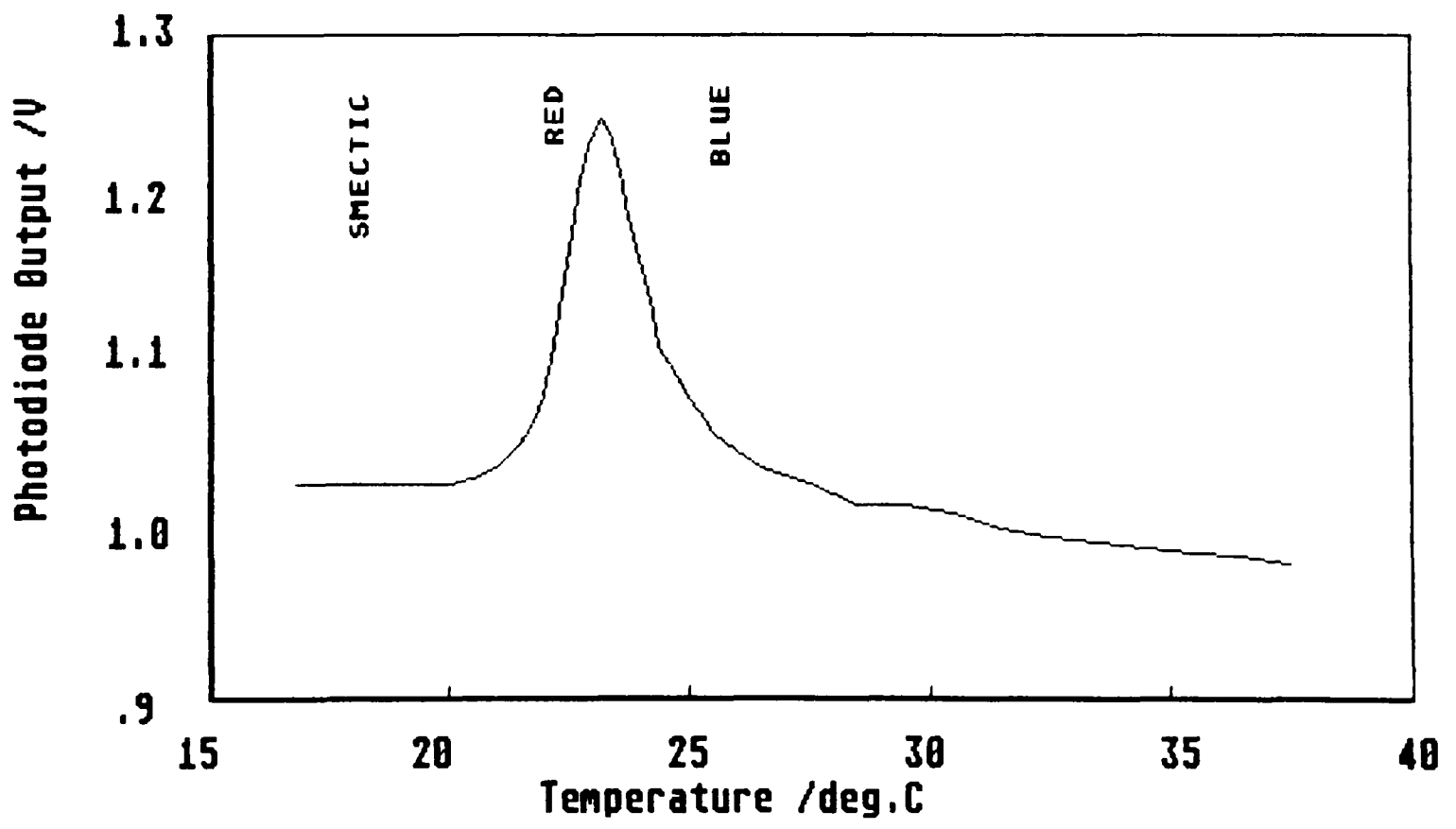


FIGURE 24. Calibration of photodiode output for encapsulated liquid crystal layer.



originating from the thermal shock wave precluded the possibility of obtaining colour/temperature readings and further work was abandoned in favour of encapsulated material.

3:3.3. Encapsulated Materials

Photodiode output traces for these layers showed single peaks, similar to the equilibrium traces (see e.g. figure 24) and these behaved in the same way as the neat cells with increasing pulse power and viscosity, i.e. peak height reduced with increasing pulse power and spread with increasing viscosity.

Although there is a temperature gradient through the layer, the photodiode takes an integrated response through the layer. Hence at high pulse powers, when high temperature gradients were produced, the photodiode output traces were broader. The peak output was still assumed to represent the equilibrium peak temperature. Temperature gradient effects will be discussed in more detail in a later chapter.

Colour filtering of the reflection *did* result in a change in shape of the photodiode output, indicating that the colour band was being scanned over time, rather than having a colour mixture at all times, as was the case for the glass cells. Thus the peak of the photodiode output gave an indication of the pitch state of the liquid crystal. High speed photography was also able to discern the colour in the layer, in the correct sequence, even though the colours were fairly weak.

None of the strong flow effects apparent with the neat glass cells were observed with the encapsulated samples

The encapsulated materials used are shown in table 1, in the order in which they were measured. Ambient temperature was set 5°C below the first appearance of selective reflection. The mixtures all have essentially the same colour play range. A range of viscosities was achieved with chiral

TABLE 1. Encapsulated mixtures used in the time constant experiment. All are mixtures of TM74/75 unless otherwise stated.

Mixture Name	Colour Onset /° C	Temp. of Peak P/D Output /° C	Bulk Viscosity /cp	Comments
A	42.6	43.4	66	
B	31.1	32.0	100	
C	18.3	19.4	172	
D	8.0	9.4	287	
E	-8.6	-7.3	530	
F	-11.5	-9.3	812	
G	22	23	146	
H	-11.9	-10.4	828	
I	31.8	32.5	95	+PCH304
J	2.5	3.7	380	
K	38.1	38.8	1275	Chol. Ester
M	-7.8	-6.1	665	
N	17	20	167	Wide band

nematic mixtures, and one cholesteryl ester was used to achieve a very high viscosity - mixture K.

The temperatures of the thin film gauges rose according to the equation :

$$\Delta T_f = K.P.\sqrt{t} \quad \text{with } K = 0.1 \text{ when } t \text{ is in ms.}$$

K' and t_c were then calculated for the encapsulated layer thickness in each case using thermal modelling programs written on a BBC 32K microcomputer. See Appendix B.

Plots of $\Delta T/P$ verses $\sqrt{t}$ justified the use of a straight line for T_{lc} and show curves for T_h , as shown in figure 25, which show an increase in the time lag with viscosity, indicating an increase in time constant with material viscosity. Suitable plots were then drawn in order to determine the time constant from equation 3.5. Each pulse at a different power produced a point for a certain mixture on plots like figure 26. Typically about 8 to 10 pulse powers between 10 and 150W were used for each mixture, resulting in good straight lines, as shown in figure 26. The results are tabulated in table 2 and plotted against material viscosity at the Sm-Ch transition in figure 27.

The results show that a typical room temperature chiral nematic mixture has a time constant around 10ms, whereas a room temperature cholesteryl ester has a time constant in excess of 100ms.

The time constant figures are longer than the actual response times measured because the time constant only comes into play as the material passes into the cholesteric phase. The time delay only refers to the time taken to reach the colour (in the yellow) giving peak photodiode output from the smectic to cholesteric transition and not from the starting temperature at which the pulse was applied. The actual response times measured were of the order of 3ms at room temperature, and are in good agreement with earlier results reported by Ireland and Jones⁴³.

TABLE 2. Results of time constant experiment for encapsulated mixtures, tabulated in order of mixture bulk viscosity.

Mixture Name	Time Constant /ms	Est. Error	Viscosity /cp
A	8	1.5	66
I	11.5	2	95
B	9	1.5	100
G	10.5	1.2	146
N	14	1.5	167
C	12.3	1	172
D	21	2	287
J	35	2	380
E	38	4	530
M	40	10	665
H	80	9	828
K	110	12	1275

FIGURE 25. Temperature rise over power against square root of elapsed time for encapsulated liquid crystal layer.

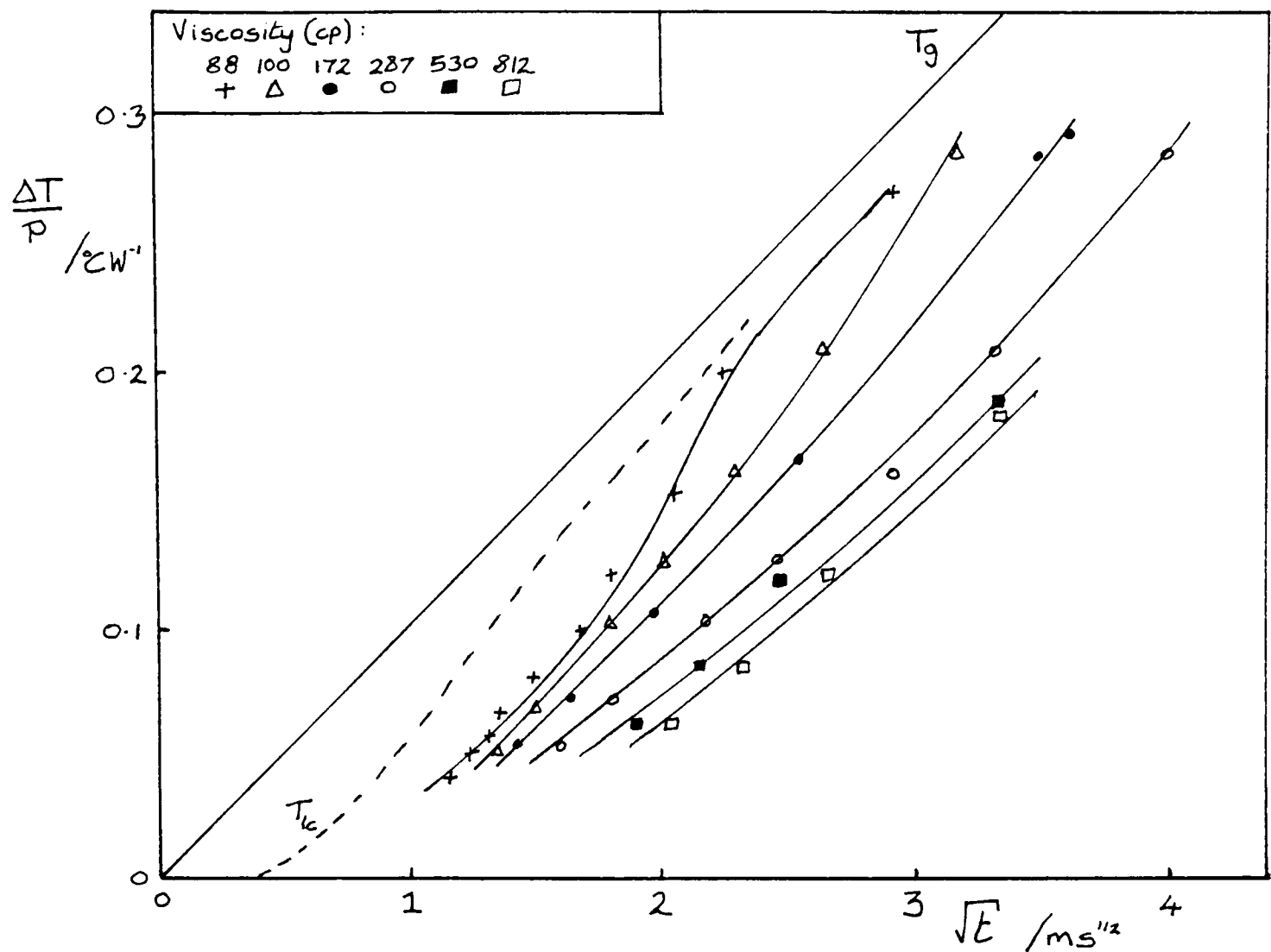


FIGURE 26. Thermal time constant calculation for encapsulated liquid crystal.

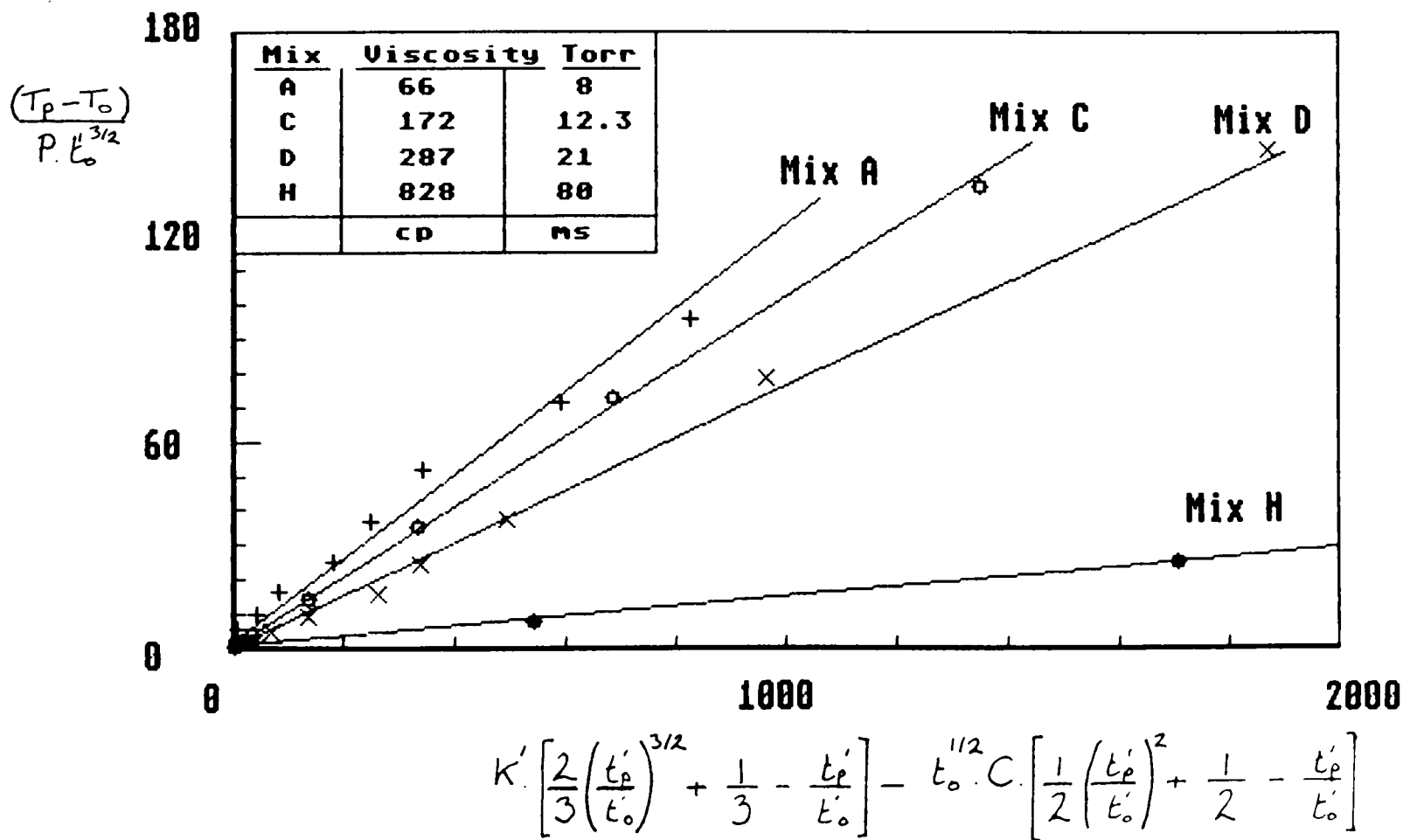
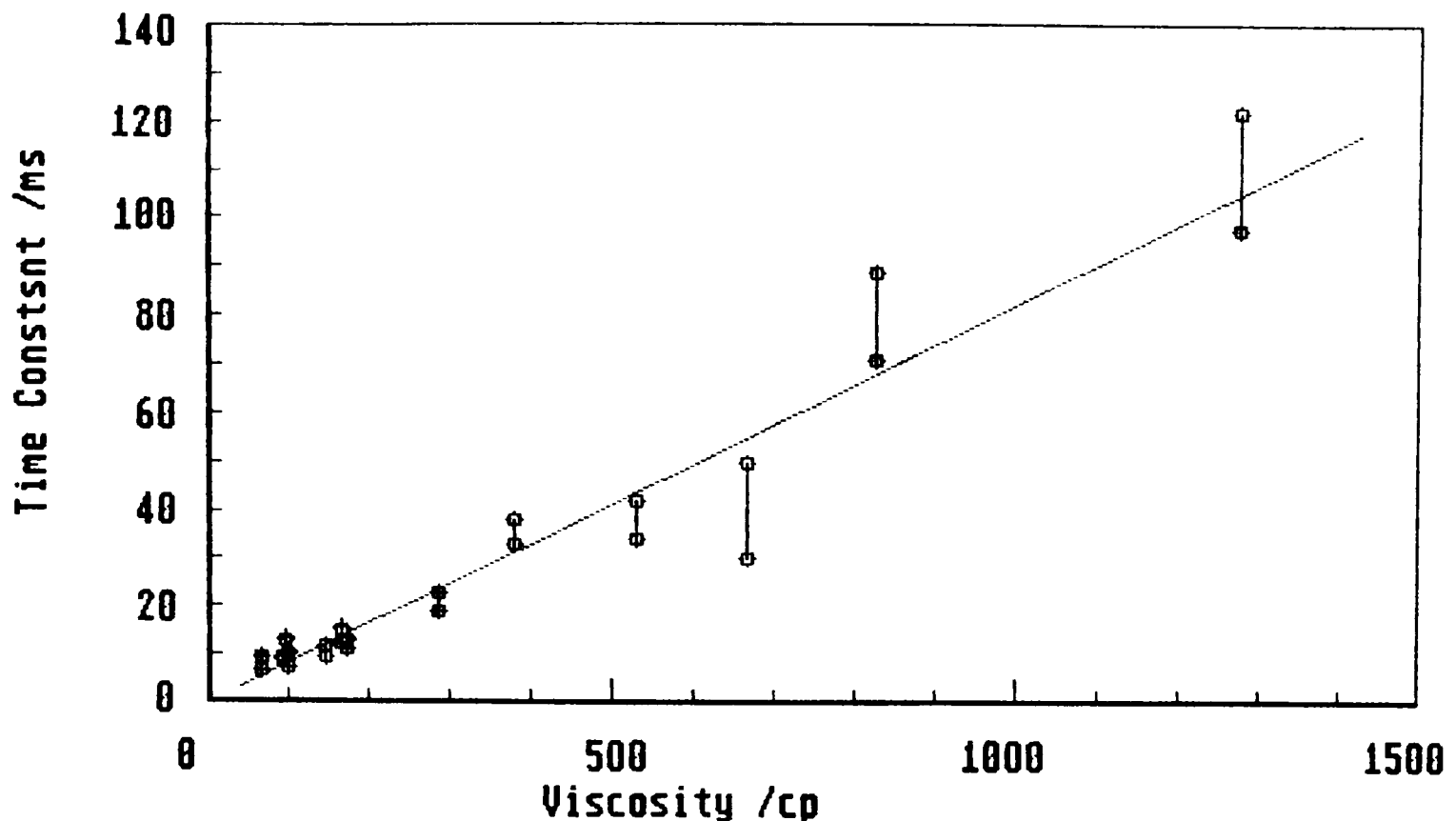


FIGURE 27. Time constant against material viscosity for encapsulated materials.



The time constant of the colour change depends on the location of the measured point (peak output) from the smectic to cholesteric phase transition since the calculations above assume that the two are fairly close. A larger separation in terms of temperature would lead to a greater thermal lag.

The time constants limit the use of the cholesteric colour change method to the measurement of heat transfer rates below about 1000 W/m²K.

3:3.4. Errors

The errors given for the time constants arose from the spread in the results for each pulse power. These errors can be attributed to the following factors:

(i.) Temperature control - The cell enclosure was controlled to within $\pm 0.2^{\circ}\text{C}$ of the required temperature even at the worst case below room temperature. Transition temperatures and colour temperatures were also measured to $\pm 0.1^{\circ}\text{C}$ on either a microscope hot stage or a Peltier cell.

(.ii.) ITO Temperature Coefficient of Resistance - The largest source of error was the behaviour of the ITO film resistance. Each individual gauge was measured, but some large variations were noticed when the same gauge was remeasured after use. This may have been due to inefficient contact to the ITO. It was found that all gauges responded to the same heat pulse to within 10% of each other, but there still remains some uncertainty as to the stability of ITO films. Confidence in the measurement was affirmed by averaging the results over several cells.

(.iii.) Thermal Properties - Errors could be produced in the thermal modelling calculations from four factors:

The pulse power, P , was known accurately to within half a percent, so can be discounted as a source of error.

The difference $T_p - T_o$ is a small quantity, typically $\sim 1^\circ\text{C}$, hence a 10% error could easily be achieved, resulting in a similar error in the time constant.

t_p was measured from the photodiode output and could be measured very accurately, depending on the trace shape. At worst the error in t_p would be around 3%, causing an approximate 7% change in the time constant.

The parabola heating constant of the liquid crystal material, K' , was calculated from the thermal modeling programs, and was typically 0.11, for times measured in milliseconds. A 10% change in K' could cause a 22% variation in the time constant. Fortunately K' is fairly insensitive to changes in the thermal diffusivity. e.g. a rise in the thermal diffusivity of 10% results in a drop in K' by only 1.5% for a 10 μm layer. See Appendix 3.

Time constants have been shown to be dependent on the material bulk viscosity which can be reduced by the addition of additives such as PCH304. The new viscosity of the mixture is estimated from

$$\ln.\eta_{\text{mix}} = x.\ln.\eta_A + (1 - x).\ln.\eta_B$$

where x is the mole fraction of material A. At 20 °C PCH304 has a viscosity of about 10cp, nearly ten times lower than a TM74/75 mixture at the same temperature. Low viscosity nematics can be used to improve the performance of this technique, particularly at low temperatures, although a limit is soon reached beyond which further improvements are very difficult. A suitable room temperature mixture may contain about 20% PCH304 in a TM74/75 mix, resulting in a viscosity of 60cp instead of around 100cp.

Much greater improvements in response time are desirable, but these are unlikely within the range of known materials. Therefore new methods were investigated using liquid crystals in order to achieve a better response time and also to overcome the temperature gradient problems, which are discussed below.

3:4. TEMPERATURE GRADIENT EFFECTS

The effects of temperature gradients are a major limitation when using encapsulated cholesteric liquid crystals to measure temperature at high heat transfer rates. Under these circumstances temperature gradients of the order of 10^6 °C/m may be produced across the 10µm layer. This produces a pitch gradient in the material structure which can reduce the reflected intensity and may destroy the Grandjean selectively reflecting texture.

Clearly the actual pitch gradient produced is dependent on the rate of change of pitch with temperature in the material. Cholesterics which are relatively temperature insensitive (low $d\lambda/dT$) will be much less effected by a temperature gradient than sensitive materials. So materials where the colour can be most accurately calibrated to a particular temperature are much more easily destroyed by temperature gradients. The pitch gradient produced will not be linear in a linear temperature gradient, but will depend on the material temperature and the value of $d\lambda/dT$ at that temperature. For most materials a given temperature gradient will produce a higher pitch gradient in the red than in the blue reflection region.

A study of the effects of temperature gradients was initiated with the aim of developing an improved method which could survive large temperature gradients and remain bright. It was a further intention of the study to establish a better understanding of the physics of the pitch change - whether there was a smooth continuous change, or if the pitch could sustain a small temperature gradient with little change and then distort at some threshold gradient. Previous workers have shown that complex thermo-mechanical coupling processes can take place in the presence of a temperature gradient⁴⁴. In the present case the temperature gradient would be parallel to the helical axis. Rotatory motion has been observed in this case by

Lehmann⁴⁵ and this is explained by Leslie's theory²¹ for small temperature gradients. Pleiner and Brand^{46,47} have shown that convection instabilities may occur at critical temperature gradients, dependent on pitch. For materials with pitch lengths in the visible region they predicted that a thermo-undulation instability would occur at a critical temperature gradient, where static undulations in the layered structure would replace flat layers as the minimum energy configuration.

For example, for heating a neat cholesteric layer from above their theory would predict a critical temperature gradient of about 20,000 K/m for a 10 μ m layer with a pitch of 0.34 μ m, using the values found in reference 46. This value can readily be achieved in wind tunnel testing and may explain why the colours disappear at high heat transfer rates.

3.4.1. Heat Pulse Experiment

In the first instance the dynamic temperature gradients which occurred during the heat pulse experiment used to measure the thermal time constants in the previous chapter are examined. These gradients were calculated using the thermal modelling programs explained in Appendix B.

Results are shown in figure 28 for both the neat and encapsulated cells. The dotted line indicates a typical pitch gradient for the encapsulated materials used. The temperature gradients in the encapsulated case did not become very high, hence visibility of the selective reflection was maintained during the pulse. This was because the encapsulated layer could only cool by natural convection to the air, whereas the neat material could lose heat by conduction to the outer glass slide. The effect of this temperature gradient on the selective reflection can be estimated by plotting the peak intensity against the pulse power, if it is assumed that the power is proportional to the temperature gradient through the layer at the time the peak occurred. This plot has been done in figure 29. The plot is initially low, probably where the

FIGURE 28. Calculated temperature gradients in pulsed encapsulated and neat liquid crystal films as a function of time. The dotted line indicates a typical pitch gradient for the encapsulated materials used.

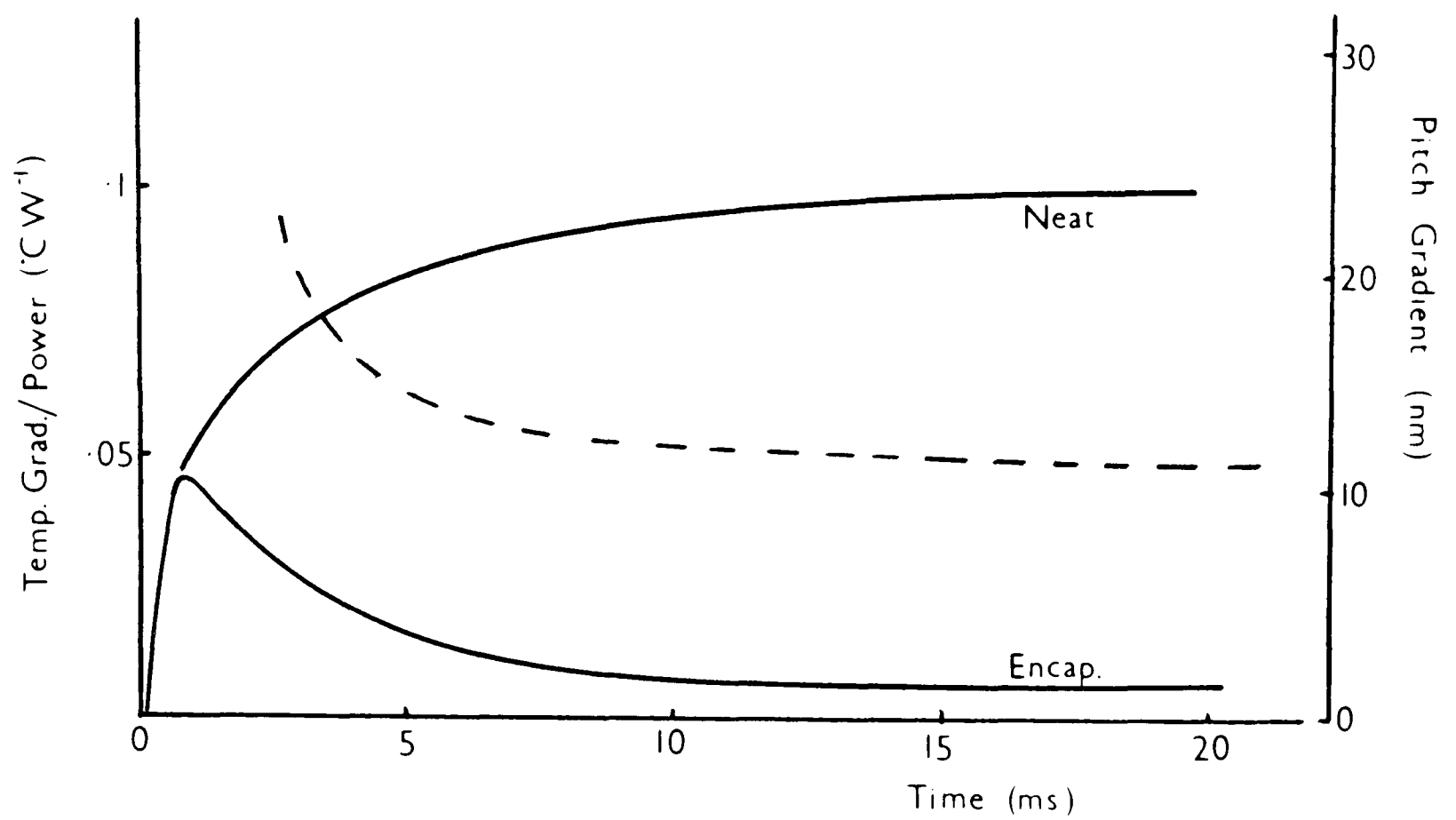
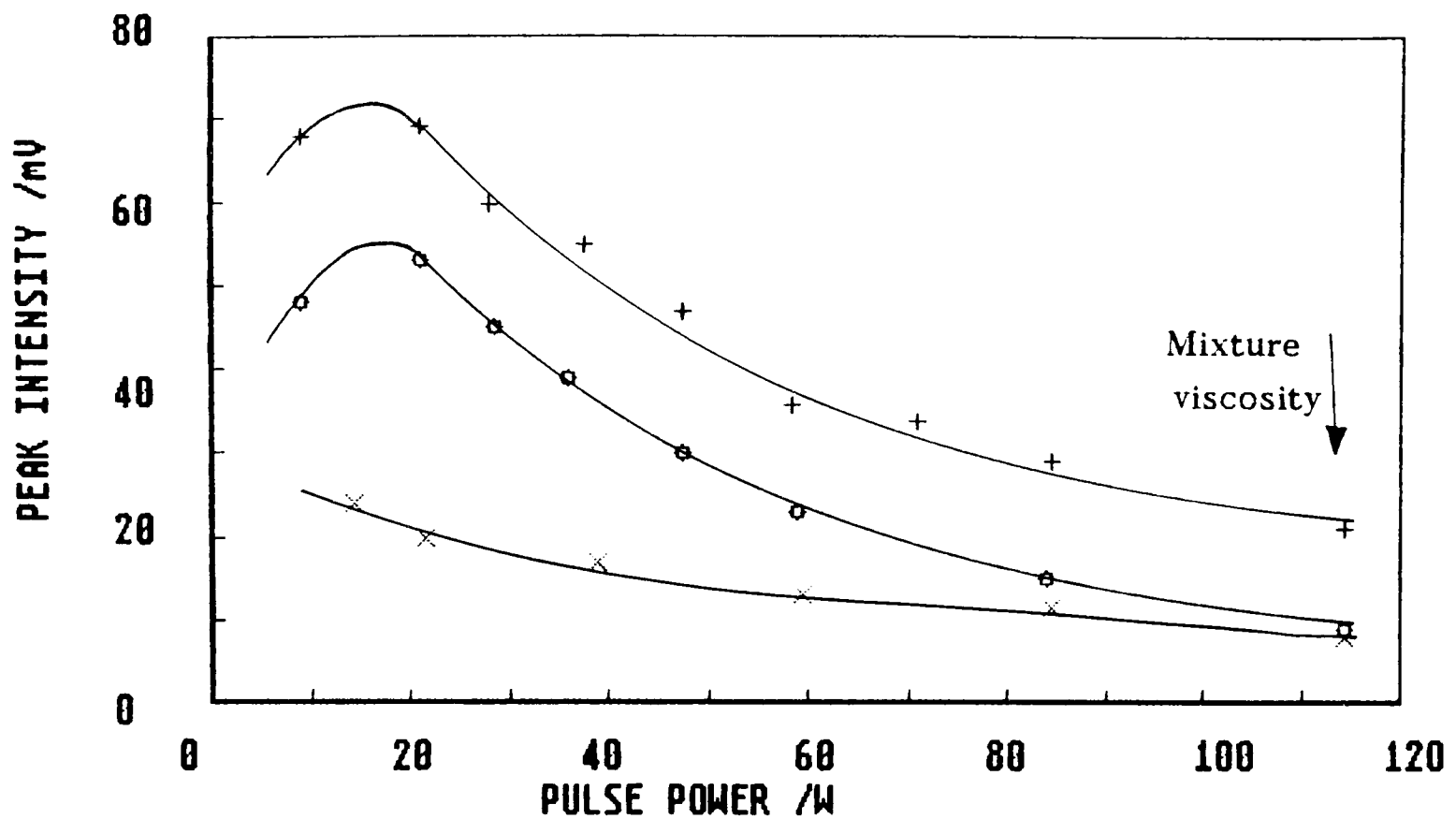


FIGURE 29. Reflected intensity as a function of pulse power for pulsed encapsulated mixtures.



pulse power is too low to heat all the cell into the cholesteric. It then reaches a maximum and drops smoothly as the pulse power is increased. In other words the reflected intensity appears in this case to drop off continuously as the temperature gradient is increased.

A second test was done with two encapsulated mixtures, one with a narrow colour band and one with a wide colour band. These were both pulsed with a range of pulse powers and the reflected intensity measured with a photodiode. As expected the wide band mixture remained brighter than the narrow mixture at high pulse powers, indicating that the intensity drop is only a result of structure distortion due to a pitch gradient, rather than a convective flow effect, which would effect both mixtures.

3:4.2 Static Experiment

It was decided to separate the effects of a static temperature gradient from those present with a variable gradient. A cell was required

which could provide a large static temperature gradient across a thin layer of liquid crystal. The gradient had to be measured and the liquid crystal reflection measured at the same time. Figure 30 shows the special cell constructed for this purpose. The lower glass slide was attached with thermally conductive paste to a temperature controlled heat sink and the bottom of the slide was sprayed matt black. A nickel gauge was evaporated onto the top surface of this slide to allow the temperature of this surface to be measured. The upper glass slide was spaced from the lower one with 12 μ m mylar spacers. A nickel gauge was also evaporated onto the inner surface of the upper slide, while the outer surface had an ITO layer on, through which current could be passed to heat the upper surface. Hence large static temperature gradients could be applied across the liquid crystal layer by heating the ITO and cooling the heat sink. The nickel gauges were then used to measure the gradient by measuring their resistances on sensitive meters.

FIGURE 30. Design of cell to measure the effect of a temperature gradient on the reflection spectrum of a cholesteric liquid crystal.

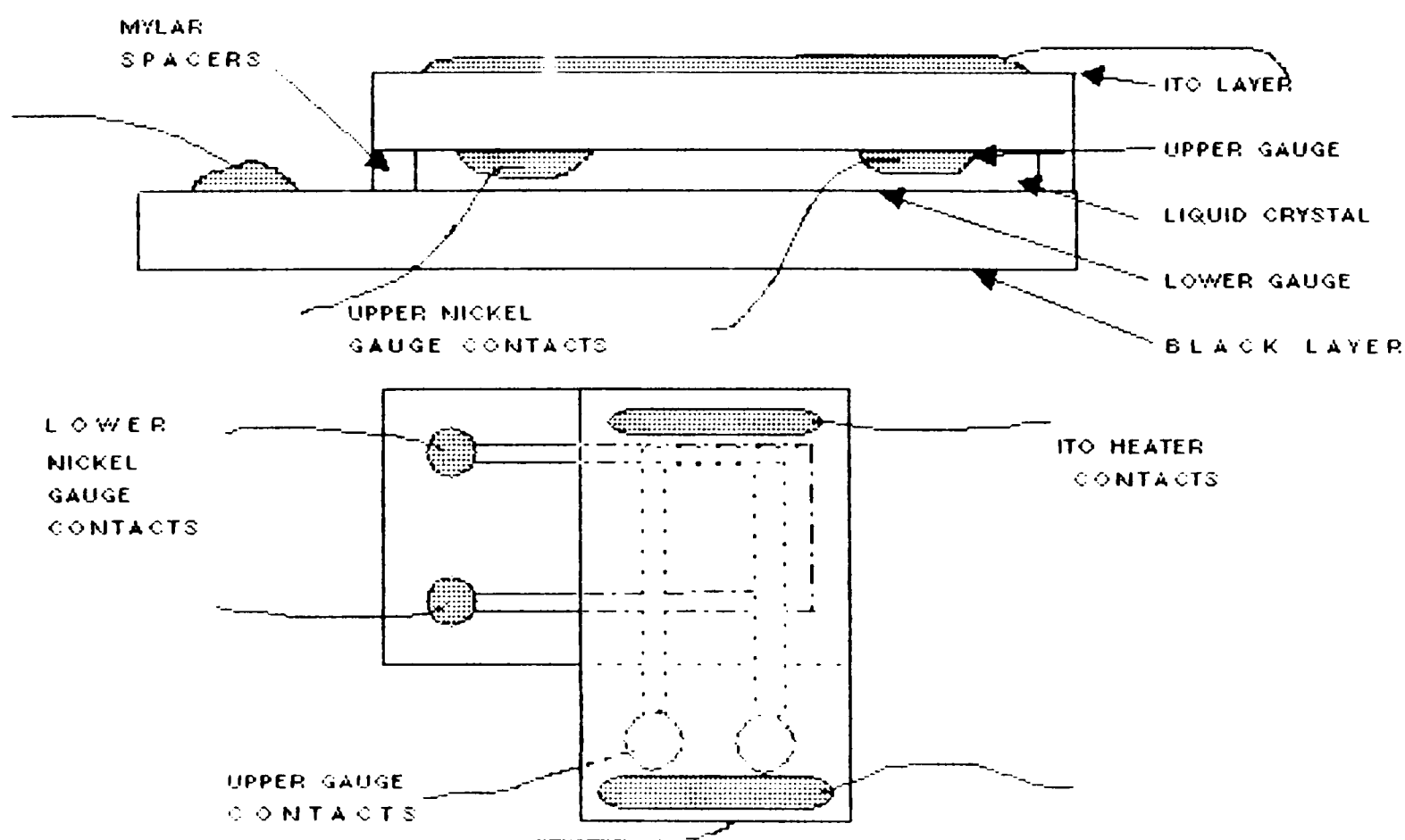
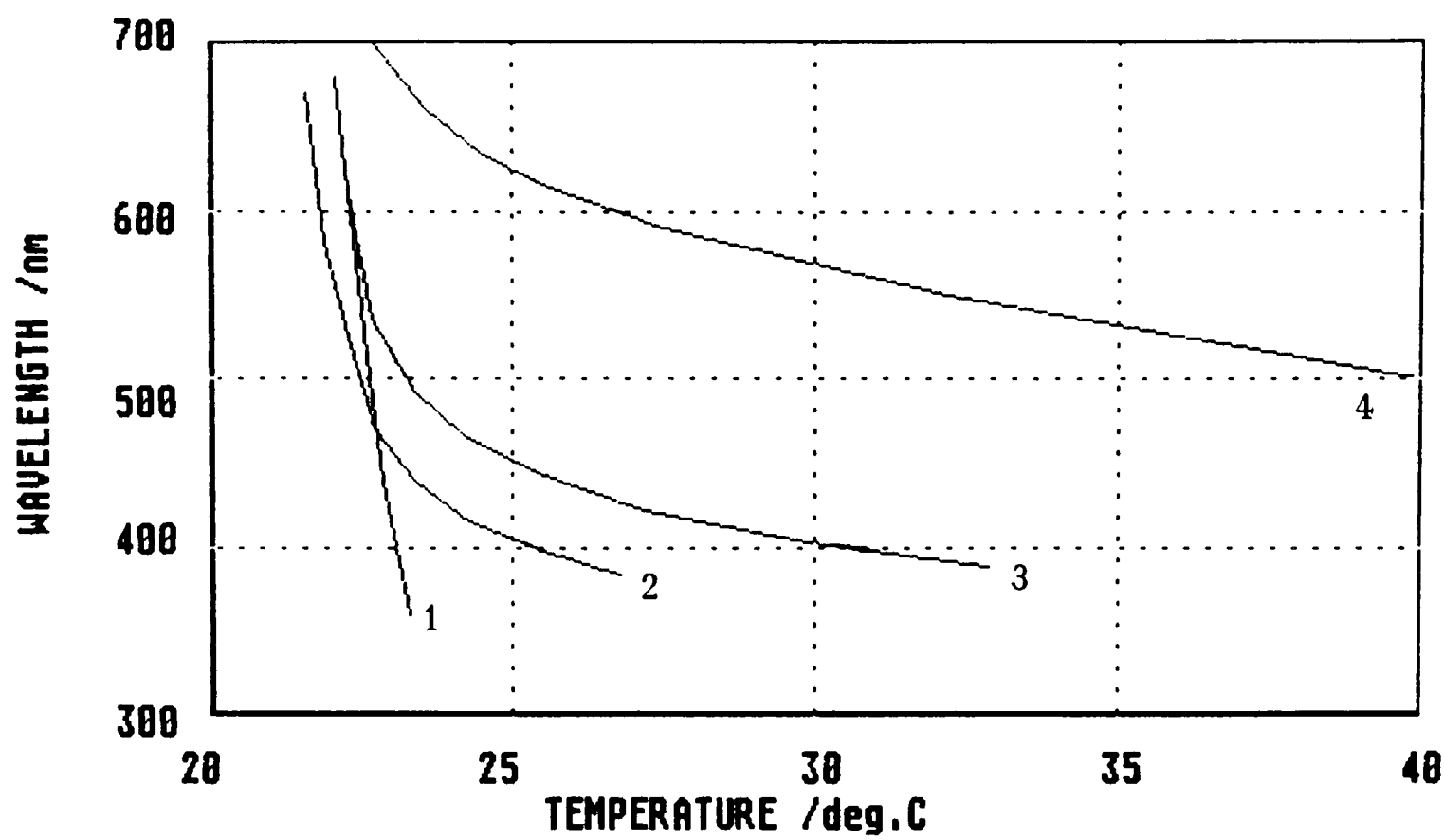


FIGURE 31. Materials used to study the effects of a temperature gradient.



Mixture No.	Composition	Sm-Ch	Ch-I
1	70% TM75A 30% TM74A	21.8° C	41.8° C
2	50% Mixture 1 50% Mixture 4	21.5° C	41.4° C
3	63% Mixture 4 37% Mixture 1	21.3° C	41.0° C
4	70% TM75B 30% TM74B	21.0° C	40.9° C

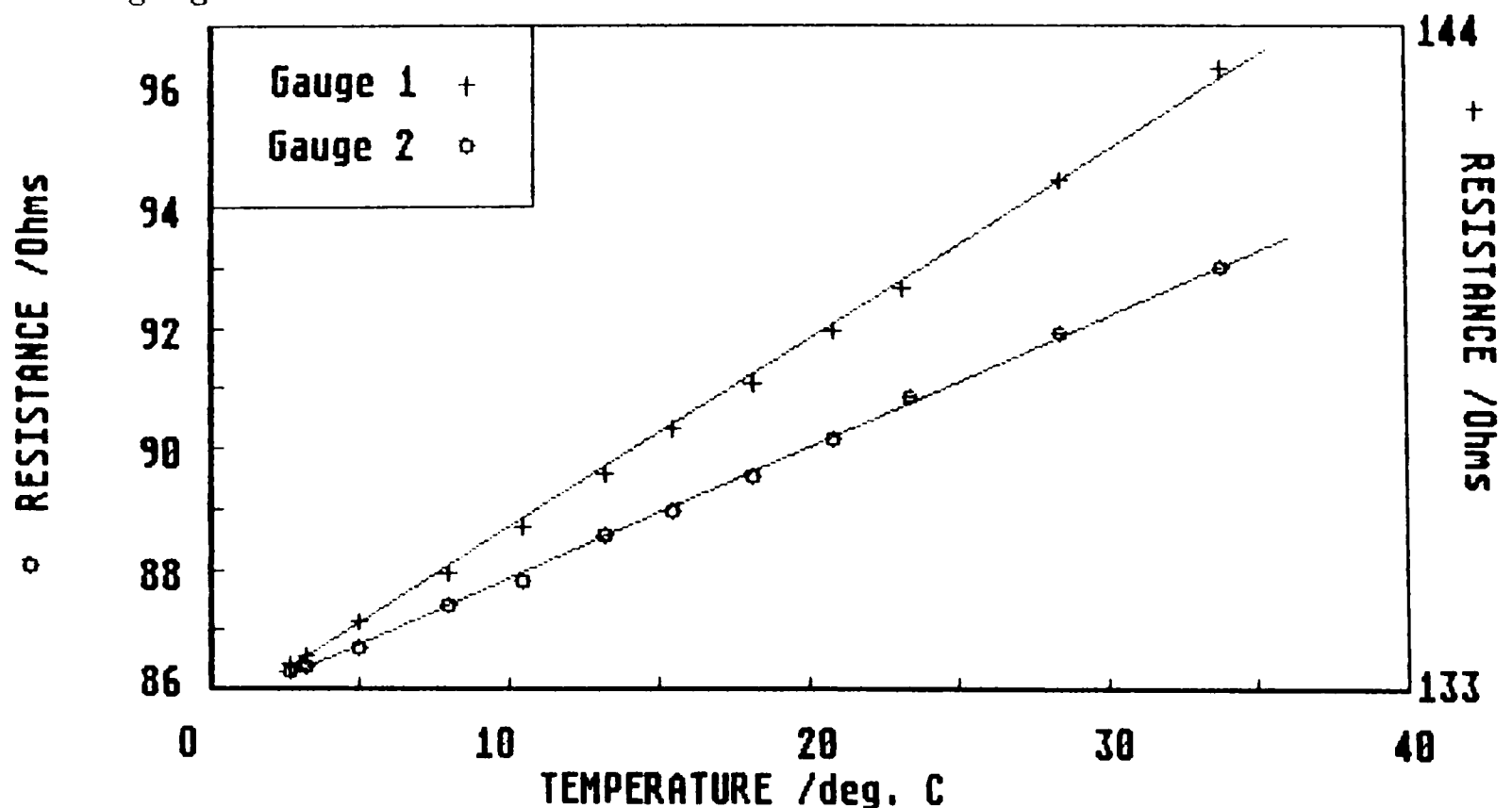
The gauges were designed to leave a clear area in the centre of the test cell where the cholesteric reflection could be measured with a Rofin-Sinar spectrum analyser. The spectra were referenced to the Kodak white standard, which was sprayed onto an ITO covered glass slide.

The materials used were neat mixtures of TM74/75 A and B. Numerous tests were done in an attempt to apply this method to an encapsulated layer, which would correspond more closely with the practical application of the materials, but these have been unsuccessful so far due to the difficulty in laminating a dried encapsulated layer in glass. Four neat materials were used, called mixtures 1 to 4, which are described in figure 31 with their colour/temperature calibrations. Mixture 1 is the most temperature sensitive, whilst mixture 4 is relatively insensitive.

3:4.2.1. Results

The nickel gauges were recalibrated before each experiment and the slope of their resistance/temperature plots were found to be fairly constant, as shown in figure 32.

FIGURE 32. Resistance verses temperature curves for evaporated nickel gauges.



For each material the temperature gradient was increased until either no resolvable signal was obtained by the spectrum analyser, or until the temperature limit of the system was reached. The maximum temperature differences applied to each material were:

1: 1.7°C ; 2: 9.6°C ; 3: 11.5°C ; 4: 26.2°C

across a cell spacing of 15 to 20 μm . The spectrum of material 4 was still readable at the maximum gradient applied. The pitch gradients corresponding to these temperature gradients were of course different for each material. Each material was initially at a pitch giving a reflection peak at around 500nm. Increasing the temperature gradient had little effect on the peak intensity, except for mixture 1, (which had the narrowest reflection band) whose spectra decayed very rapidly when a gradient was applied. The peak generally shifted to the blue, and at large gradients the spectra showed a strong blue peak with low reflection in the red, as a result of the $d\lambda/dT$ curve of the mixture.

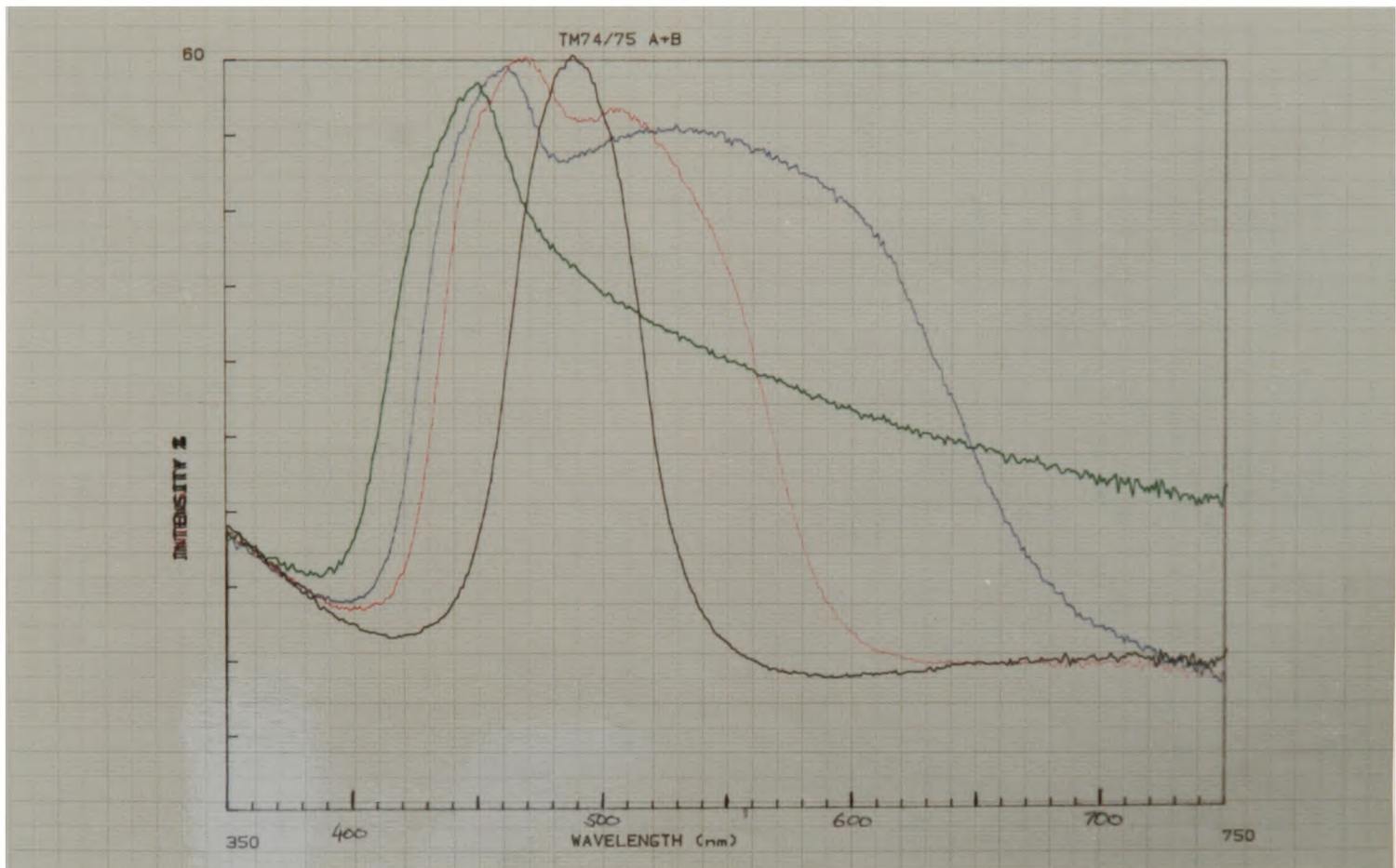
A typical sequence of spectra are shown in plate 5. a and b, for mixtures 2 and 3. They both clearly show the expected widening of the bandwidth, and a shift of the peak towards the blue with the red reflection intensity dropping. An unexpected feature is the appearance of a double peak in the spectra. This occurred at very approximately 80,000°C/m, or at a pitch gradient of about 5×10^6 nm/m. At high temperature gradients the samples developed tiny bright specks of different colours, which was comparable to the appearance of the pulsed neat cells shown in plate 4. In both cases this was thought to be due to the structure breaking and forming two regimes of pitch length from each inner surface separated by a discontinuity at the centre of the cell in a plane parallel to the substrates. As the temperature gradient increased the red peak diminished in intensity, while the blue remained high, due to the greater temperature sensitivity ($d\lambda/dT$) at the red end of the spectrum.

PLATE 5. Effect of temperature gradients on the reflection spectra of mixtures 2 and 3 in 15 μ m glass cells.

(a). Mixture 2. Key: Black - 22.6 to 22.7 $^{\circ}$ C. Red - 21.8 to 23.5 $^{\circ}$ C. Blue - 21.6 to 23.8 $^{\circ}$ C. Green - 20.4 to 24.7 $^{\circ}$ C. These curves may be compared to the simple theoretical curves of figure 33.

(b). Mixture 3. Key: Black - 23.1 to 23.2 $^{\circ}$ C. Red - 22.6 to 23.7 $^{\circ}$ C. Green - 22.3 to 25 $^{\circ}$ C.

a.



b.

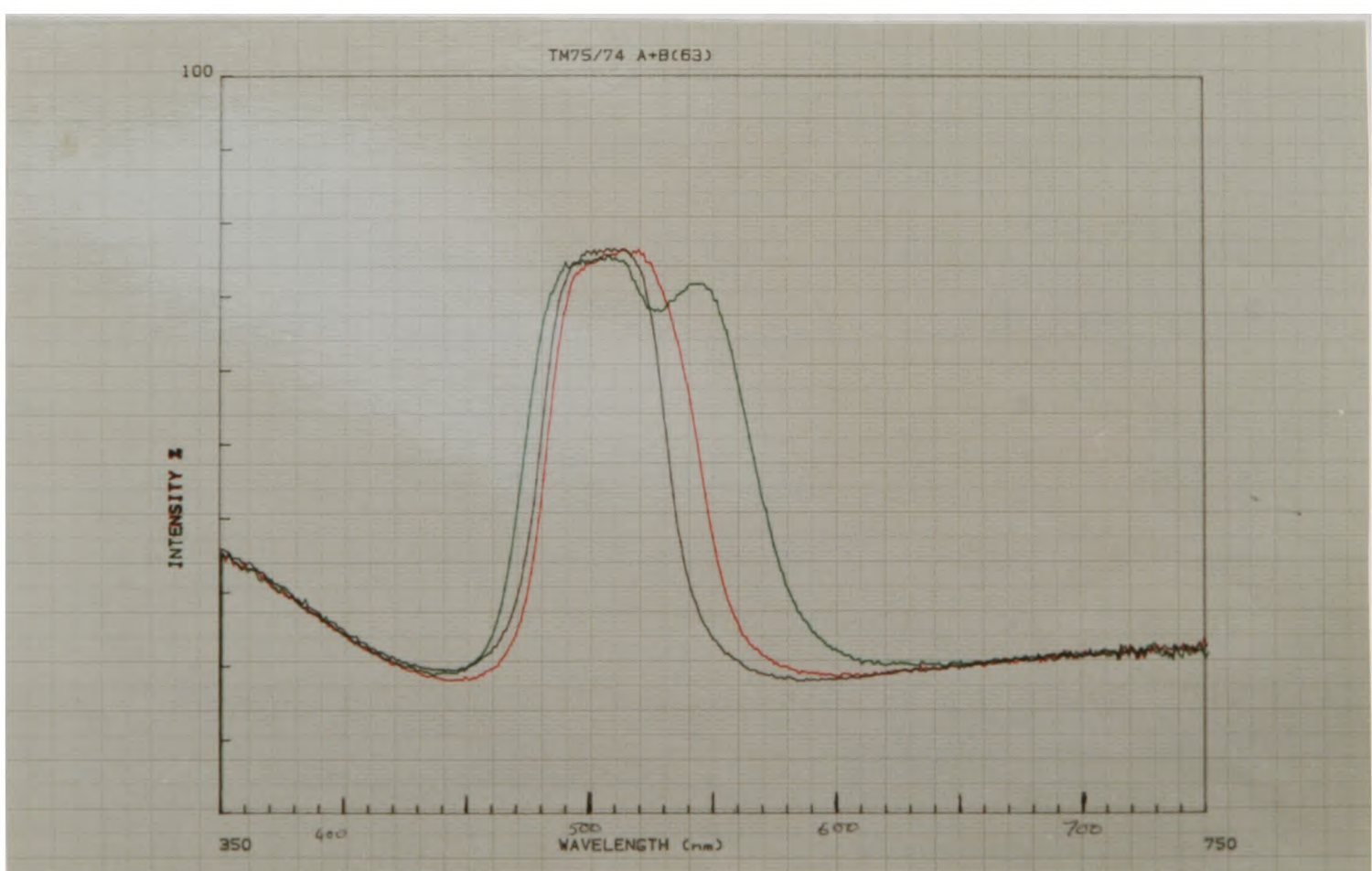


FIGURE 33. Reflection spectra bandwidth against pitch gradient.

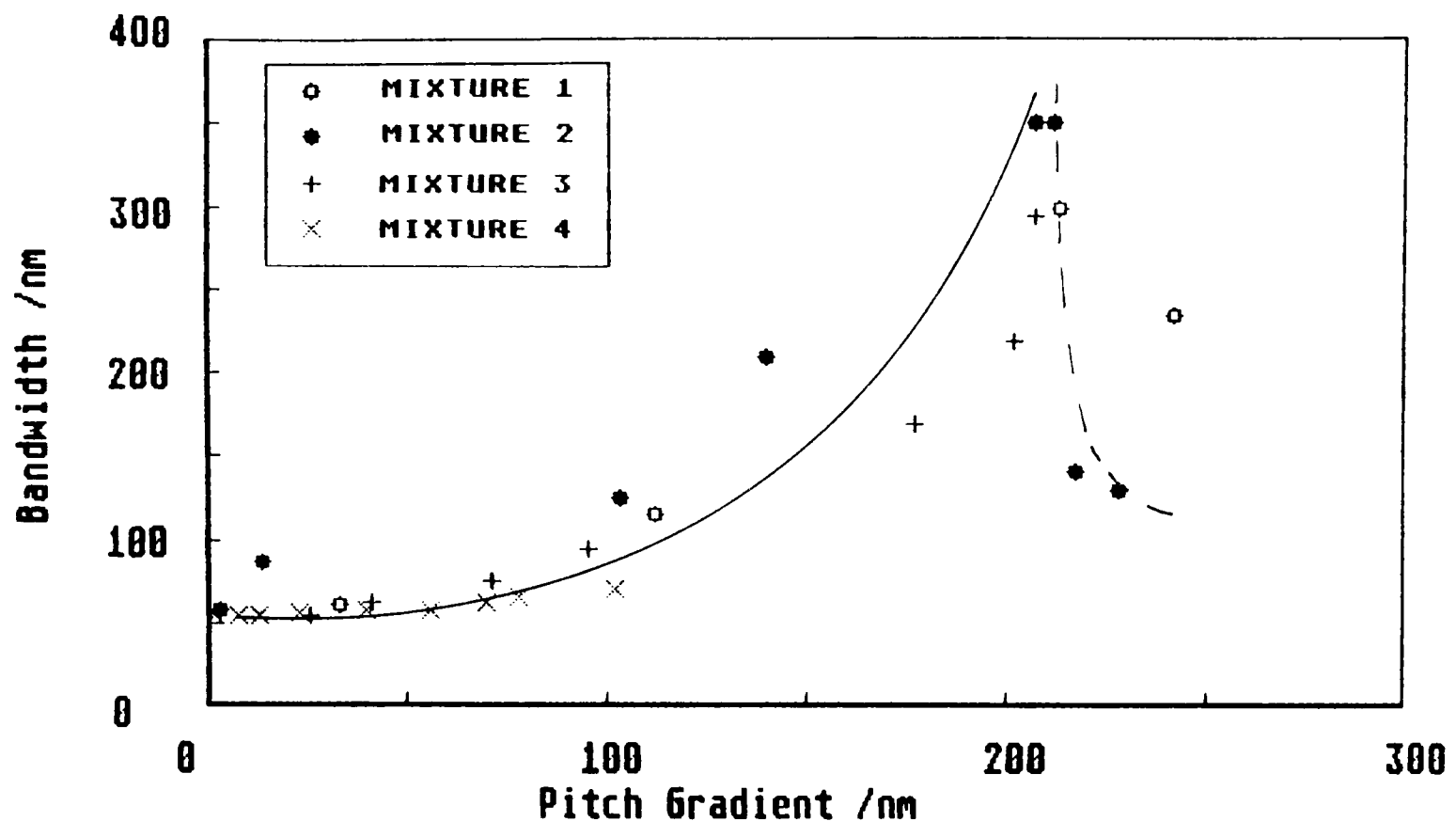
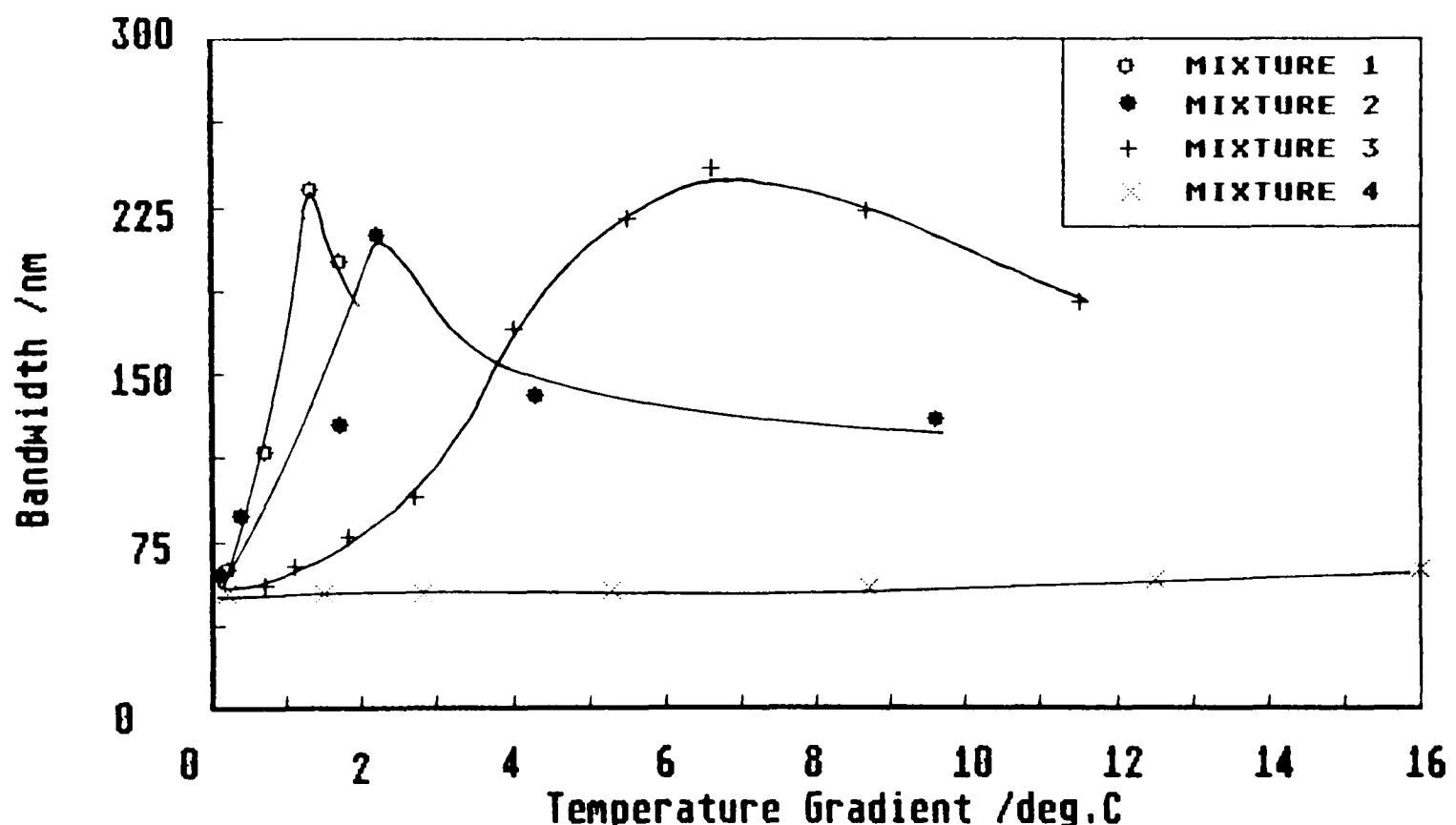


Figure 33 shows plots for each mixture of bandwidth against calculated pitch gradient. These all fall on the same line indicating that the nickel gauge temperature results were consistent between the samples. The width of the reflection spectra continue to increase with pitch gradient, but the bandwidth, defined as the width of the reflection peak at half maximum, drops, as indicated on the figure with the dotted line, as the reflected intensity in the red drops quite quickly as there is a higher pitch gradient at the red end than at the blue end of the spectrum, for a particular temperature gradient, resulting in a very low reflected intensity in the red. This is a direct result of the $d\lambda/dT$ characteristics of each material, and the fact that the reflected intensity at each wavelength is proportional to the thickness of material with a pitch corresponding to that reflected wavelength. This results in an apparent reduction in bandwidth at high gradients to give a blue peak with a low reflection across the red. Figure 34 presents the bandwidth against temperature gradient for each mixture. This clearly shows the expected changes, with mixture 4 being relatively insensitive to the presence of a

temperature gradient, whereas mixture 1 degrades very quickly. The position of the band shifts to the blue in each case. The pitch gradient was calculated from the nickel gauge temperature measurements and a knowledge of the wavelength/temperature calibration of each mixture. The average refractive index was assumed to be 1.6 in each case. Mixture 4 didn't reach large pitch gradients even at very high temperature gradients, but each of the other mixtures showed a divergence of the apparent bandwidth at around 200nm.

FIGURE 34. Selective reflection bandwidth against temperature gradient.



3:4.2.2. Analysis

In order to interpret the above results a simple computer model was prepared to calculate the reflection spectra, making the assumption that the pitch would vary continuously through the layer when in the presence of a linear temperature gradient. The model developed was designed to give a simple approximation of an ideal system under normal viewing and lighting

conditions. (Other theoretical models are presented in references 48 and 49).

To do this two equations were used to calculate (a). the $d\lambda/dT$ characteristics of each material, and (b). the reflected intensity for a certain thickness of material of known birefringence and pitch.

Pindaks⁵⁰ equation was chosen to calculate (a) since this was found to give better fits than Keatings⁹ equation for low $d\lambda/dT$ mixtures. Pindaks equation is:

$$p = p_{\odot} + A.(T - T_{\odot})^{-\nu}$$

where p is the material pitch at temperature T . $T_{\odot}$ is the smectic to cholesteric transition temperature, $p_{\odot}$ is the pitch at the cholesteric to isotropic, and A and ν are constants used to give a good fit.

Good fits for the mixtures were obtained using the following parameters,:

<u>Mixture</u>	<u>$p_{\odot}$(nm)</u>	<u>A</u>	<u>ν</u>
1	200	150	0.80
2	340	95	0.70
3	350	150	0.70
4	480	200	0.65

The equation used to describe reflected intensity at normal viewing and lighting conditions was first given by Fergason⁴ :

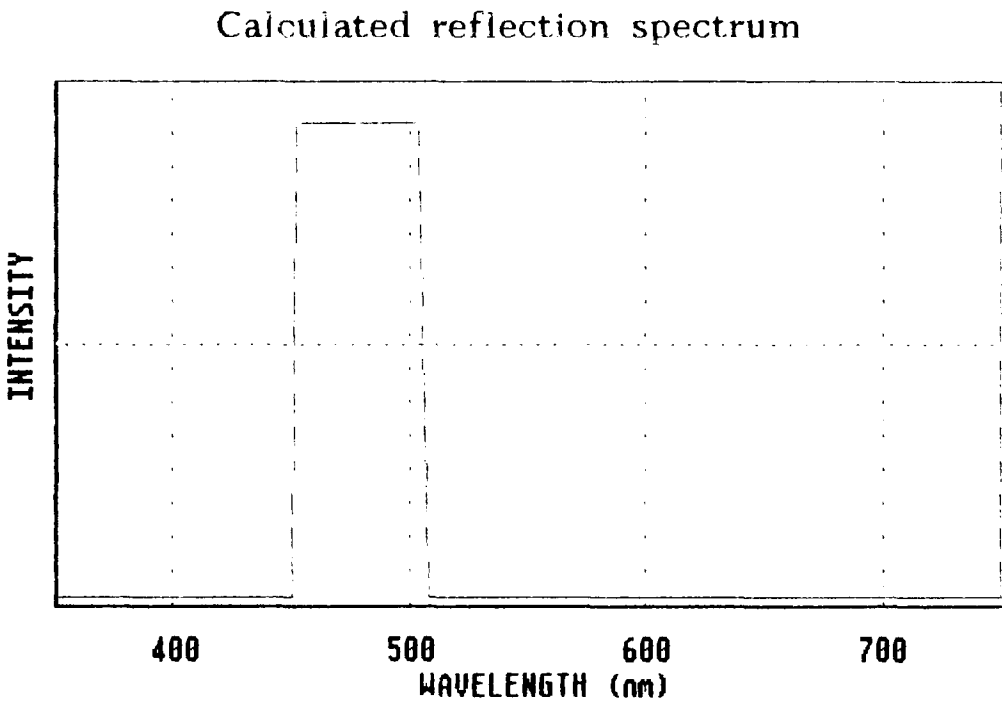
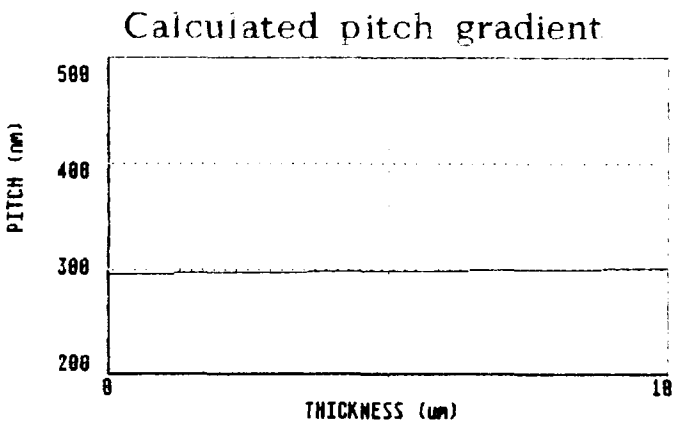
$$I_{\text{ref}} = I_{\text{inc}} \left(\frac{1 - (n_o/n_e)^{8d/\lambda}}{1 + (n_o/n_e)^{8d/\lambda}} \right)$$

Where d is the sample thickness and I_{ref} and I_{inc} are the reflected and incident intensities, respectively.

FIGURE 35. Calculated spectra in presence of a temperature gradient

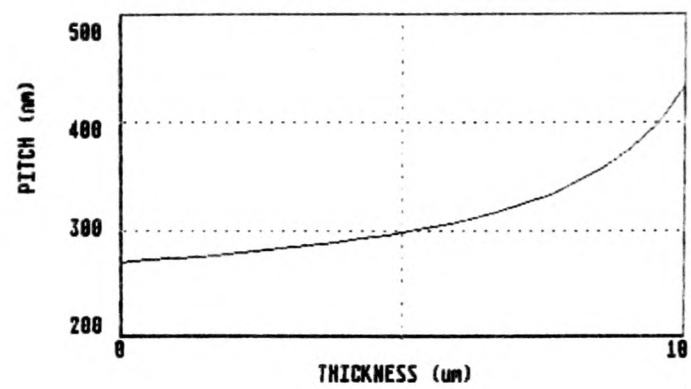
All plots calculated for mixture 2.

Temperature difference
across thickness:
 $22.6^{\circ} - 22.7^{\circ}$

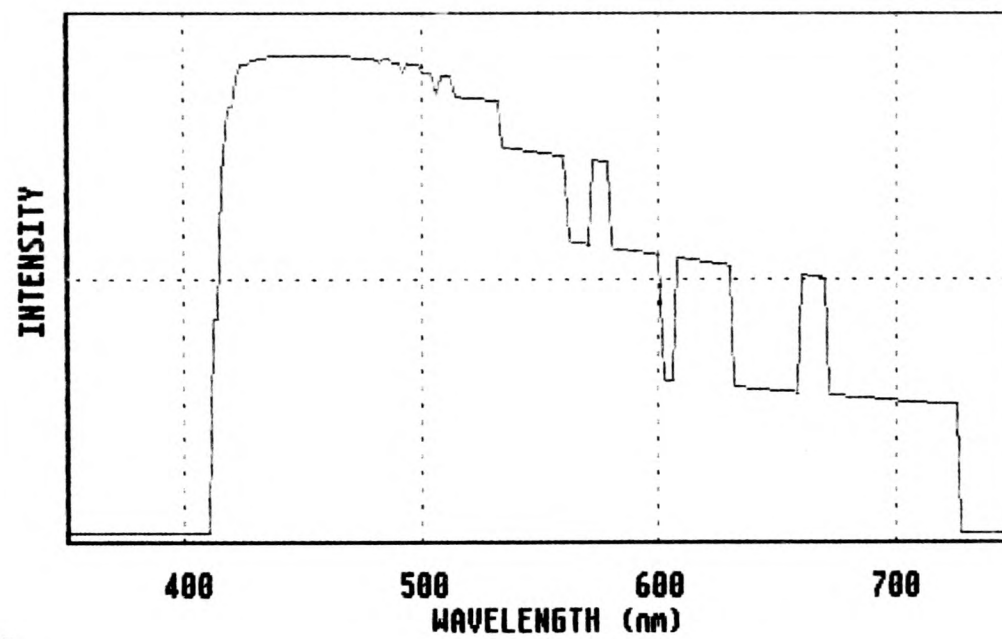


Temperature difference
across thickness:
 $21.8^{\circ} - 23.5^{\circ}$

Calculated pitch gradient

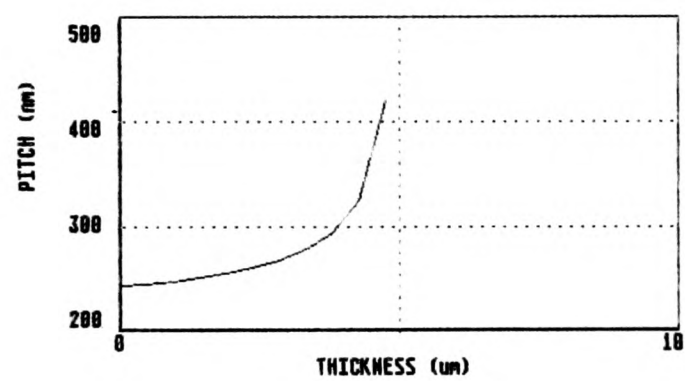


Calculated reflection spectrum

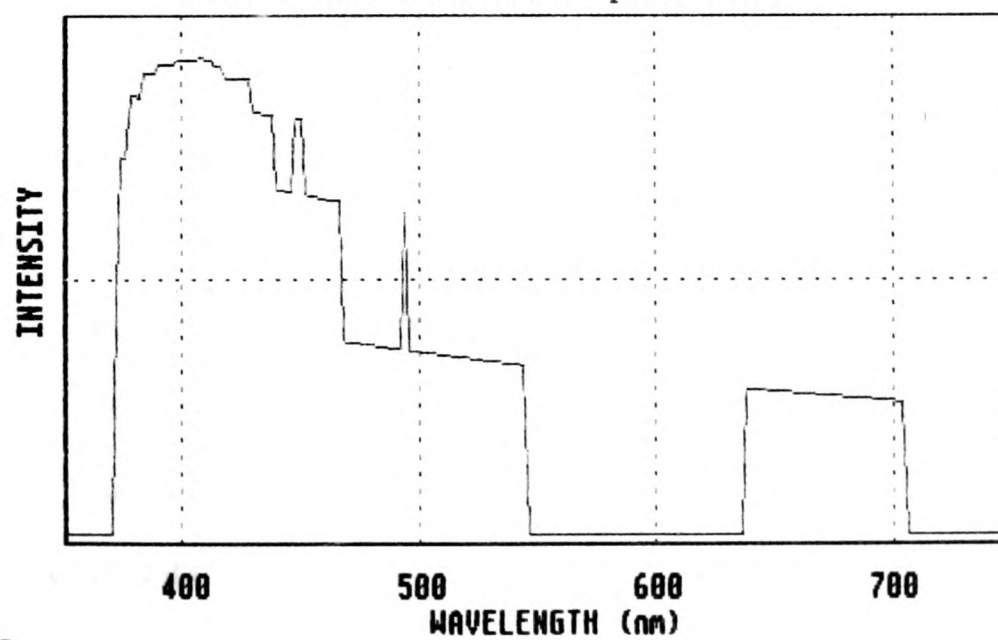


Temperature difference
across thickness:
 $16.8^{\circ} - 26.4^{\circ}$

Calculated pitch gradient



Calculated reflection spectrum



Using these equations a program was written for an Atari ST computer that could plot the pitch gradient and a reflection spectrum by inputting n_o , n_e , A , v , p_o and the temperature gradient. Even though these spectra are rather coarse, some useful comparisons may be made with the experimental spectra and the following conclusions were drawn:

The calculated spectra (see figure 35) show the same increases in bandwidth with temperature (pitch) gradient as the experimental spectra, but double peaks are not formed. This may be explained by considering that in the experiment the two opposite surfaces of the liquid crystal layer are at pitches corresponding to the temperatures of those surfaces, but that the pitch is not continuous through the layer for high gradients. Instead some discontinuity forms in the centre of the layer. This may be induced by a flow/undulative instability which isn't taken into account in the model.

The major errors in the experiment arise from the measurement of temperatures using the nickel gauges, since small changes in contact resistances can have a significant effect on the calculated temperature values. Steady state thermal analysis indicated that the temperature gradients were generally lower than calculated from the nickel gauge, but this may be due to uncertainty in the thickness and conductivity of the conductive paste between the lower slide and the heat sink. Table 3 offers a comparison of these figures together with a summary of the results.

3:4.3. Conclusions

1. At low temperature/pitch gradients a continuous change in pitch through the layer is observed.
2. At higher gradients corresponding to pitch changes of $>5 \times 10^6$ nm/m no selective reflection is observed which appears to correspond to a break up of the Grandjean texture.
3. Low $d\lambda/dT$ materials (e.g. mixture 4) retain Grandjean ordered

TABLE 3. Results of temperature gradient experiment.

Mixture 1

Temp. Grad./° C	Peak Wavelength/nm	Bandwidth /nm	Pitch Gradient/nm	Peak Intensity
22.5 - 22.7	468	63	33	39
22.3 - 23.0	435	115	113	41
22.0 - 23.3	394	233	242	40
22.2 - 23.9	353	>200	213	38

Mixture 2

22.9 - 23.0	478	60	3	64
22.8 - 23.2	477	87	13	60
21.8 - 23.5	467	127	103	60
21.6 - 23.8	461	213	139	59
21.3 - 24.1	454	>350	207	58
20.9 - 24.3	459	>350	209	59
20.4 - 24.7	448	140	213	57
19.3 - 25.3	437	-	219	59
19.0 - 26.0	433	-	224	58
16.8 - 26.4	441	130	227	56

Temp. Grad./° C	Peak Wavelength/nm	Bandwidth /nm	Pitch Gradient/nm	Peak Intensity

Mixture 3

22.7 - 23.4	504	55	26	77
22.6 - 23.7	507	64	41	76
22.4 - 24.2	516	77	70	75
22.3 - 25.0	500	95	95	75
21.9 - 25.9	506	170	177	74
21.1 - 26.6	501	220	201	74
20.5 - 27.1	507	250	205	74
19.5 - 28.2	493	230	211	76
18.5 - 30.0	494	180	218	72

Mixture 4

30.8 - 31.0	562	52	1	54.5
30.5 - 32.0	562	52	8	55.4
30.2 - 33.0	562	52	13	55.2
29.4 - 34.7	561	53	25	55.2
28.6 - 37.3	561	54	40	55.5
27.2 - 39.7	561	58	56	56
25.7 - 42.3	564	63	70	56
25.1 - 45.5	564	66	77	56
23.5 - 49.7	575	70	103	55

textures even in the presence of very large temperature gradients ($\sim 10^6$ °C/m).

4. Although low $d\lambda/dT$ mixtures are relatively unaffected by temperature gradients across the layer, their low temperature resolution limits their use for mapping heat transfer isotherms.

5. Alternative methods for determining heat transfer coefficients greater than $\sim 1000 \text{ Wm}^{-2}\text{K}^{-1}$ using liquid crystals are explored in the following section.

3:5. IMPROVEMENTS IN METHOD

The colour changes in cholesteric liquid crystals have been used very effectively to map surface temperatures in low heat transfer environments. During the course of this work detailed heat transfer mapping around pedestals was carried out by Dr. P. Ireland and Col. A. Byerley. In addition surface roughness was investigated by Z. Wang. All these tests were carried out at the Osney Laboratories of the Dept. of Engineering Science at Oxford. These tests demonstrated the wide range of uses of the liquid crystal method at low heat transfer rates and motivated the present work to extend their working range. Some of the techniques developed are discussed below.

3:5.1 Improvements to Existing Method

Prior to the present study PVA was used as the cholesteric ink binder. This is a relatively soft polymer and does not give a very durable encapsulated film. It has been found to blow off at high wind speeds. Therefore a range of alternative binders were investigated, which were found to have smoother and harder surfaces than PVA. Reflectance experiments were carried out to compare the optical performance of these binders, using the same liquid crystal mixture. The films did not all dry to the same thickness, and were therefore likely to affect the shape of the microcapsules. The binder materials used in the evaluation were (a). #32 from F. Davies, (b). Jon Cryll 80 and (c). Mowlilith, supplied by Harlow Chemicals. The Mowlilith gave generally the best results, producing an even, hard encapsulated layer. Some of the Jon Cryll materials degraded the liquid crystal.

3:5.2 New Methods

In order to overcome the time constant and temperature gradient problems which limit the working range of the technique the following methods were devised, developed and evaluated.

3:5.2.1 Cholesteric to Isotropic Transition Method

As shown earlier the selective reflection may be retained in the presence of a large temperature gradient provided that the pitch is relatively insensitive, as is the case for materials without an underlying smectic phase, or for temperatures well above the smectic to cholesteric transition. Furthermore the cholesteric to isotropic transition may be used as an isotherm, since the material can change from a single bright colour to a colourless, isotropic liquid at a well defined first order transition (Ch-I).

A simple test was conducted using this method, with encapsulated liquid crystals, in a low heat transfer environment, and the results are shown in the photographic sequence of plate 6. The sequence shows hot gas passing around a pedestal and the isotherm clearly brings out the horse shoe vortex structure of the heat transfer at the surface. An additional feature of the method, shown in the final frame, is that on cooling the system incorporates a thermal memory. This arises because as the liquid crystal cools from the isotropic phase it forms a texture which is significantly less reflective and appears dark. Plate 7 shows photomicrographs of two regions of the same encapsulated material in which one area has been heated into the isotropic and cooled while the other has not. The visible difference is quite distinct, the latter retaining a well ordered structure whilst the former is more disordered and less bright. This corresponds to a drop in the reflected intensity of about 30% and also explains why most heat transfer tests give better results if the surface is heated through the colour play rather than cooled through it. The

PLATE 6. Sequence of photographs demonstrating the cholesteric to isotropic phase change method of surface temperature method. The encapsulated material has been applied to the surface around a pedestal and subjected to hot gas flow from left to right. When the gas flow is turned off the layer exhibits a thermal memory of the region which has exceeded the phase change temperature (final frame).

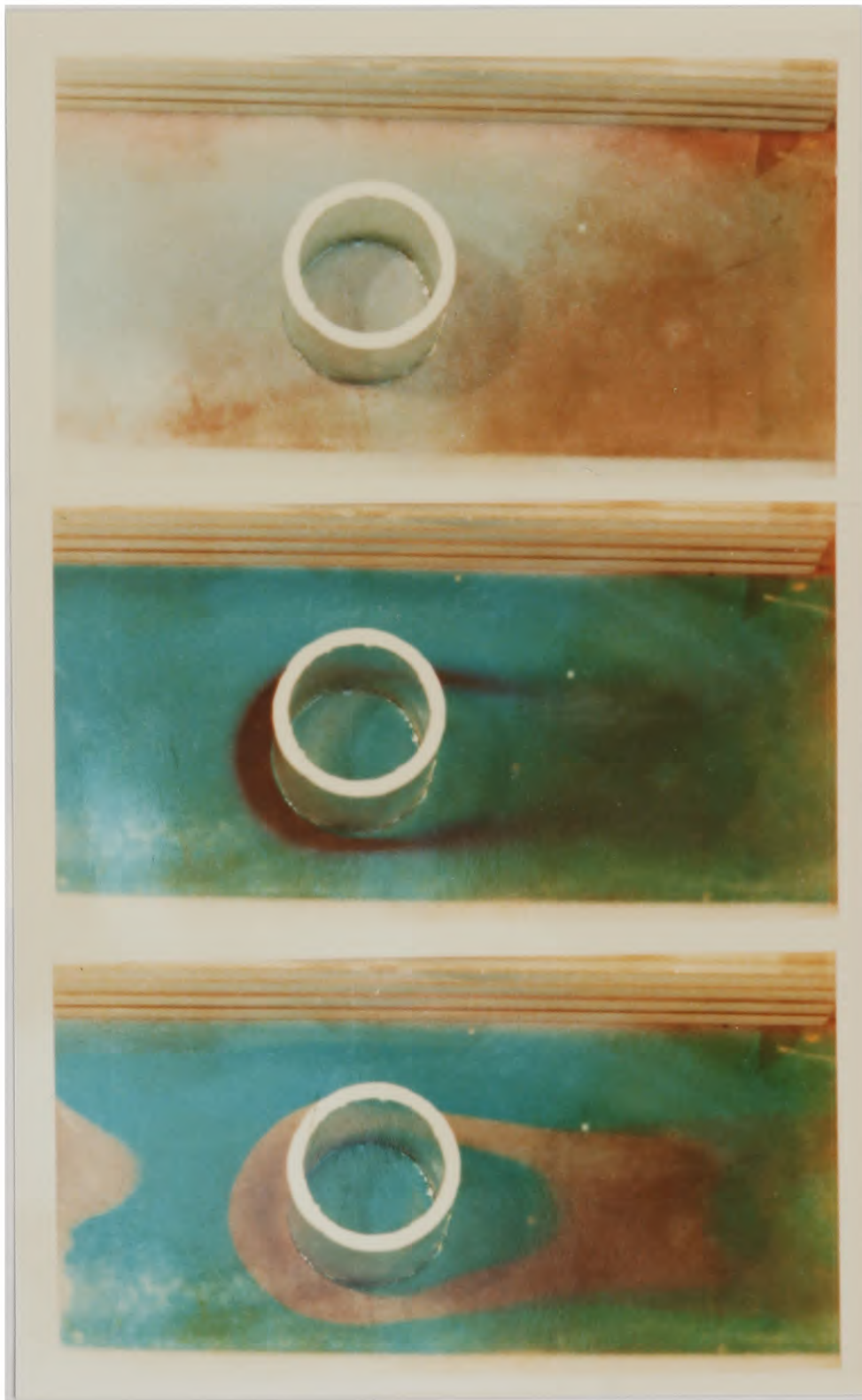
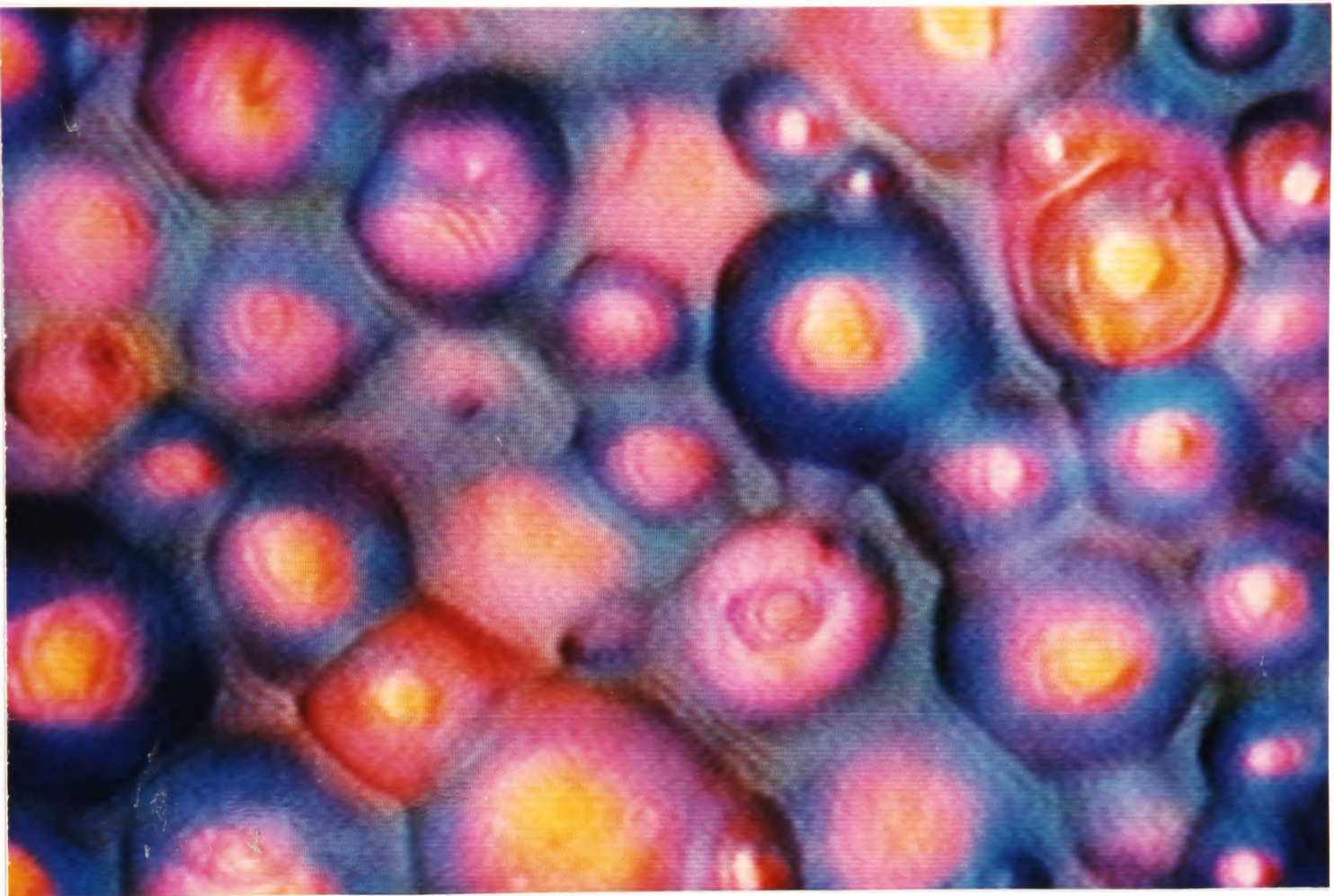
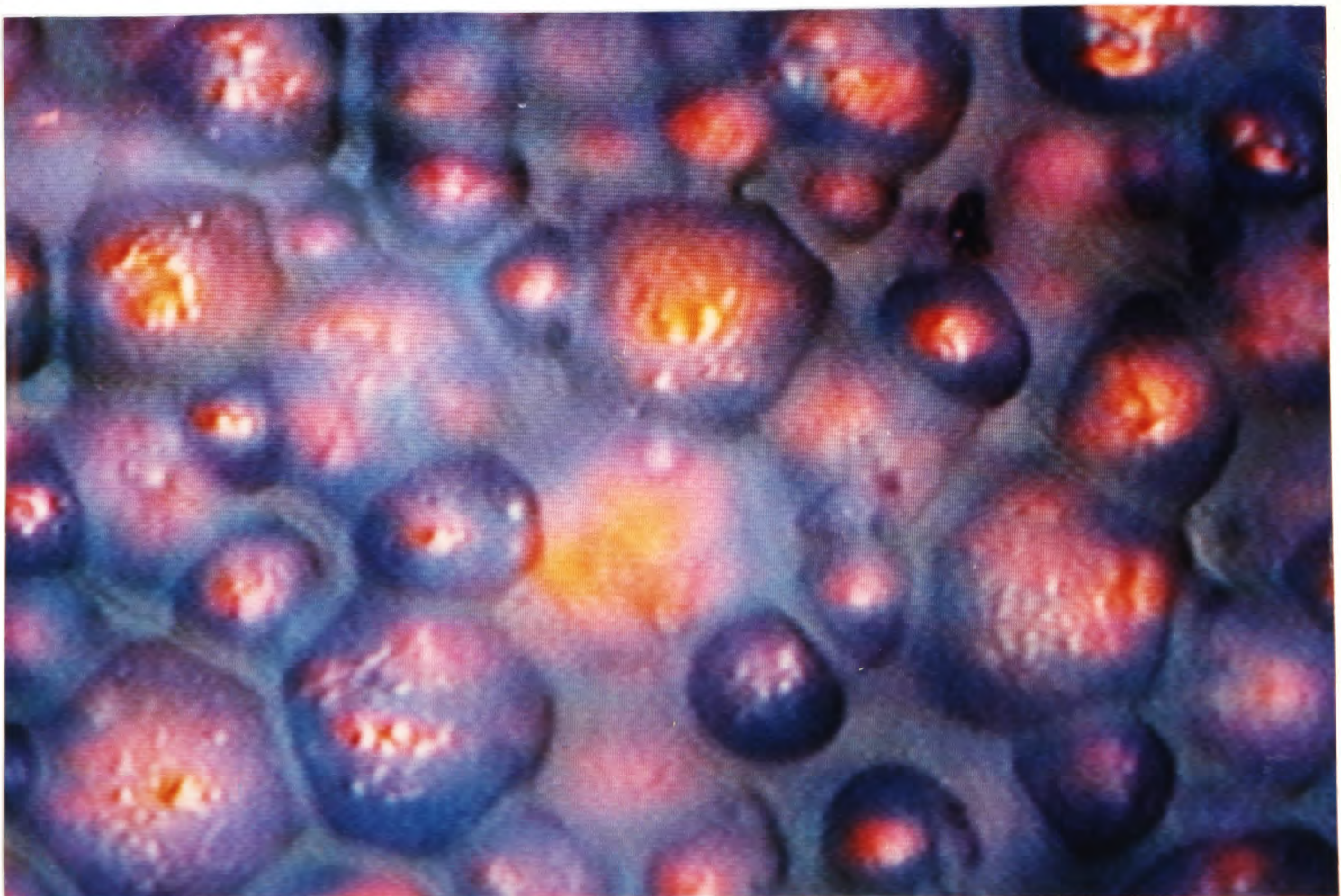


PLATE 7. (a) and (b). Microscopic comparison of encapsulated cholesteric liquid crystal material (a) before and (b) after being heated into the isotropic phase. This is the basis of the thermal memory effect as the heated material cools to become less ordered (greater percentage of focal conic texture) and so the reflected colour is much less bright than the unheated sample. Average capsule diameter approx. 10 μ m.

a.



b.

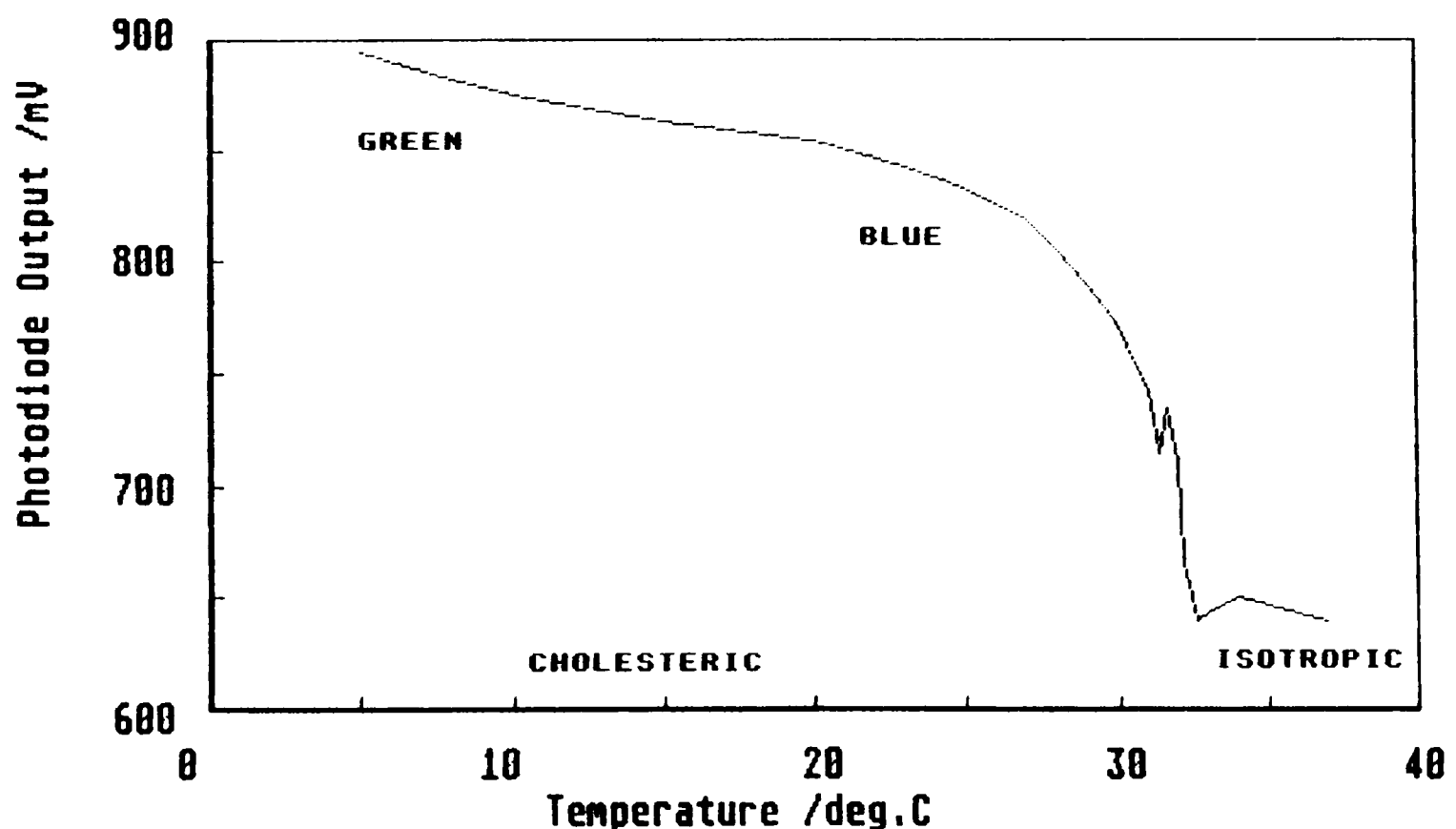


boundary between the two states is stable, but may be reset by cooling the material into a smectic phase well below the operating temperature. Reheating resets the brightly reflecting Grandjean texture.

Since the method showed promise for solving the temperature gradient problem further work was carried out to establish, (a).the thermal response time of the cholesteric to isotropic transition detector, and (b).whether multi-event detection was feasible using this system.

(a). The thermal response time was measured using the same heat pulse apparatus as before. A $\sim 10\mu\text{m}$ thick encapsulated layer was silk screen printed onto an ITO gauge as before. In this case, however, the change in reflectance from the cholesteric to isotropic phases was used as the isotherm and this was measured by the photodiode. Figure 36 shows a typical equilibrium photodiode calibration for the material used.

FIGURE 36. Calibration of the photodiode output for encapsulated cholesteric to isotropic phase change material.



The time constant equation used for the cholesteric colour method is not valid in this case, as the measured property is not being driven toward equilibrium by some force. It is purely a single measured event at the phase transition. The pulse test was repeated, however, using a mixture named TCC451, and the actual response times of the method were compared as a function of pulse power. The mixture had a cholesteric to isotropic transition at 31.6°C. The response times were of the order of milliseconds. The main factor that would usually be considered to cause a thermal lag at a phase transition is the latent heat of the material for that transition. A simple model was considered, where it was assumed that the temperature of the heated surface rises linearly *at the phase transition*, and that the phase change line then moves through the layer thickness at some speed. The time for this line to cross the layer thickness is then calculated. The details of this calculation have been included in Appendix D. Using a latent heat of 4000 J/kg and a rate of temperature rise of 1000 °/s results in a time of 3.4ms for the phase transition time to pass through the 24µm thick layer. The thickness was measured with a z-axis microscope. This time is almost identical to the measured response times, so the latent heat may be considered to be the major cause of lag at the transition. Hence in wind tunnel tests with high heat transfer rates, where the latent heat may be supplied very quickly, it is expected that the method would demonstrate very high response times. So far the method has been able to measure heat transfer rates up to 100,000 W/m², indicating the major improvement in response time and ability to withstand temperature gradients of this method over the colour change method.

(b). In order to establish the feasibility of combining two or more encapsulated mixtures into a single film, and thereby achieve a multi event technique, encapsulated samples of E44+40%CB15, (Ch-I at 48°C), and E7+35%CB15, (Ch-I at 29°C), were mixed and deposited as a single layer.

Preliminary tests showed that two isotherms were possible but the contrast between them was poor. Optimisation of the colour combinations could yield acceptable results. It may also be possible to combine this method with the birefringence method (described in the next section) to achieve two temperature indications.

The resolution of the method is limited by the temperature spread of the transition. This spread, or biphasic region, was of the order of 0.5°C for the materials mentioned above.

The ideal materials are those with a narrow temperature range for the Ch-I and with an underlying S-Ch transition well below the operating temperature to enable colour resetting. The best materials evaluated in terms of these requirements were derived from the BDH chiral nematics (TM74/75). Using the partially racemised analogues of these materials, a reasonably temperature insensitive pitch can also be achieved over a wide (~ 10 to 20°C) operating temperature range.

In conclusion, the use of the phase change method enables a considerable extension of the use of cholesteric liquid crystals in heat transfer measurement and wind tunnel tests carried out at Oxford have employed the technique to record heat transfer rates of up to $100,000\text{ W/m}^2$.

3:5.2.2. Birefringence Colour Method

As was mentioned earlier the physical properties of liquid crystals are anisotropic, and this includes the refractive index.

For example, light travelling with its electric vector vibrating perpendicular to the optic axis in an aligned nematic layer, which is a uniaxial positive material, will travel faster than light with its electric vector vibrating parallel to the optic axis, hence the materials are birefringent. Many commercially available nematic liquid crystals, such as 4-n-alkyl-4'-cyanobiphenyl, do not have absorption bands at visible

frequencies, so both waves will be transmitted, but will experience different refractive indices, n_o for the first case and n_e for the second. The birefringence (Δn) of the material is given by $(n_e - n_o)$.

If an aligned nematic layer is placed at 45° between crossed polarisers colours are visible because the layer acts as a retardation wave plate. The incident light is polarised at 45° to the optic axis. It may then be considered as two components parallel and perpendicular to the optic axis. These two travel at different velocities through the layer, so that when they emerge there is a phase difference between them. This is true for all wavelengths, but when the retardation is 2π there is no phase difference between the two components. In this case the two components remain unaffected by the layer and the resultant will be absorbed by the second polariser. All other wavelengths experience some retardation and become elliptically polarised to some extent when they recombine. Hence some portion of this light will be transmitted through the second polariser. The transmitted colour is the complimentary colour to that which was totally absorbed.

The layer acts as a wave plate for wavelengths which satisfy:

$$\text{retardation, } \Delta\Phi = (2\pi/\lambda) \cdot d \cdot (n_e - n_o) = 2\pi$$

Δn does not vary very greatly with frequency over the optical range.

For liquid crystals the birefringence also varies with temperature. Hence the transmitted colours vary and may be calibrated. A typical variation of birefringence is shown in figure 37, which shows that Δn changes increasingly quickly approaching the nematic to isotropic transition.

This is the basis of a temperature measurement method which is made practical by the use of a porous plastic membrane called celgard.^{C4} Celgard is a 25 μ m thick porous material which, when soaked in a nematic liquid crystal becomes fairly transparent. The absorbed nematic material is strongly aligned in the celgard, so this thin aligned layer may be applied to a test surface and viewed between crossed polarisers. Plate 8 shows an SEM

FIGURE 37. Variation of birefringence with temperature for a nematic liquid crystal (K15) at various wavelengths.

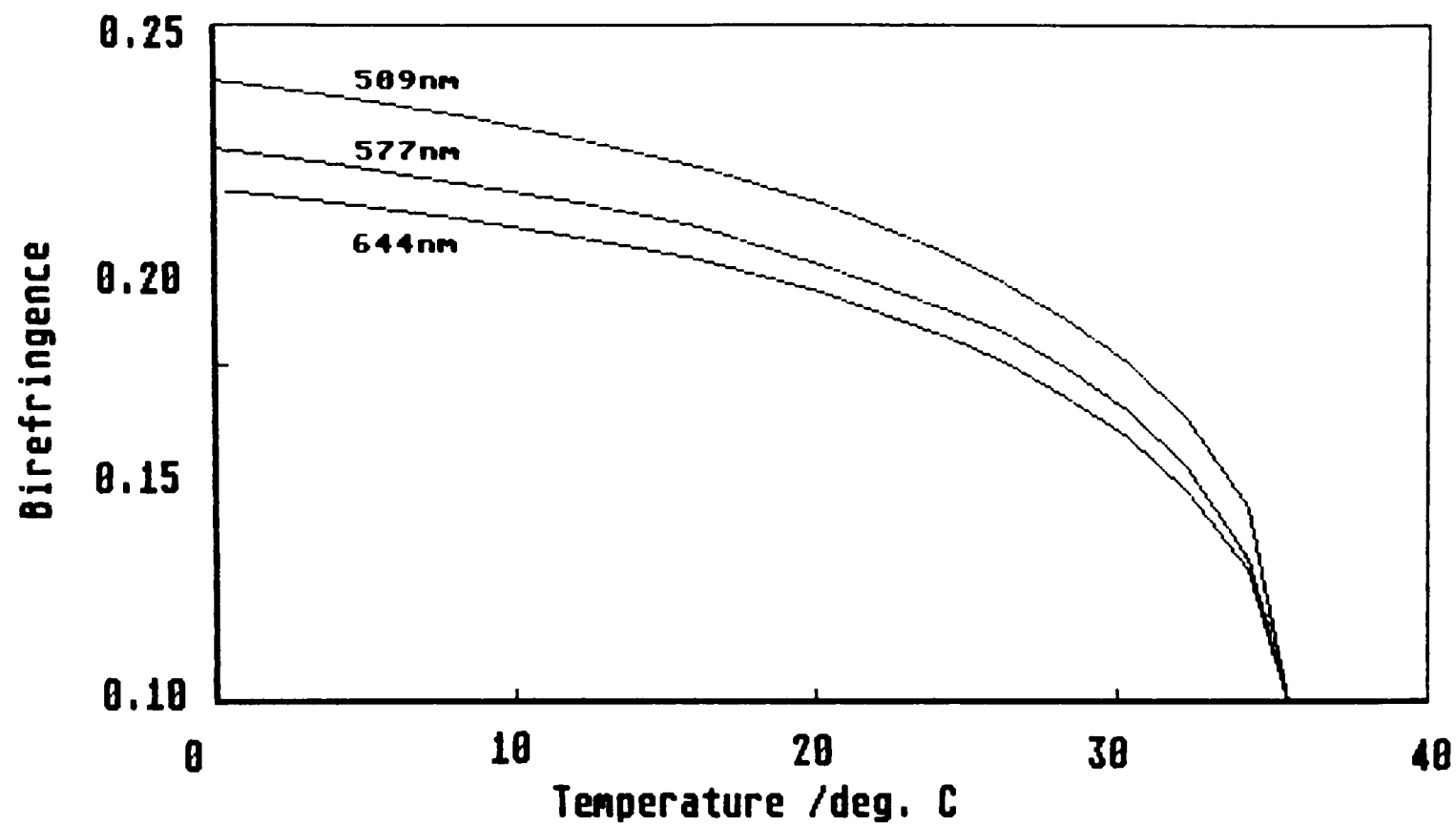


FIGURE 38. Transmission spectrum at different temperatures for K15 in celgard.

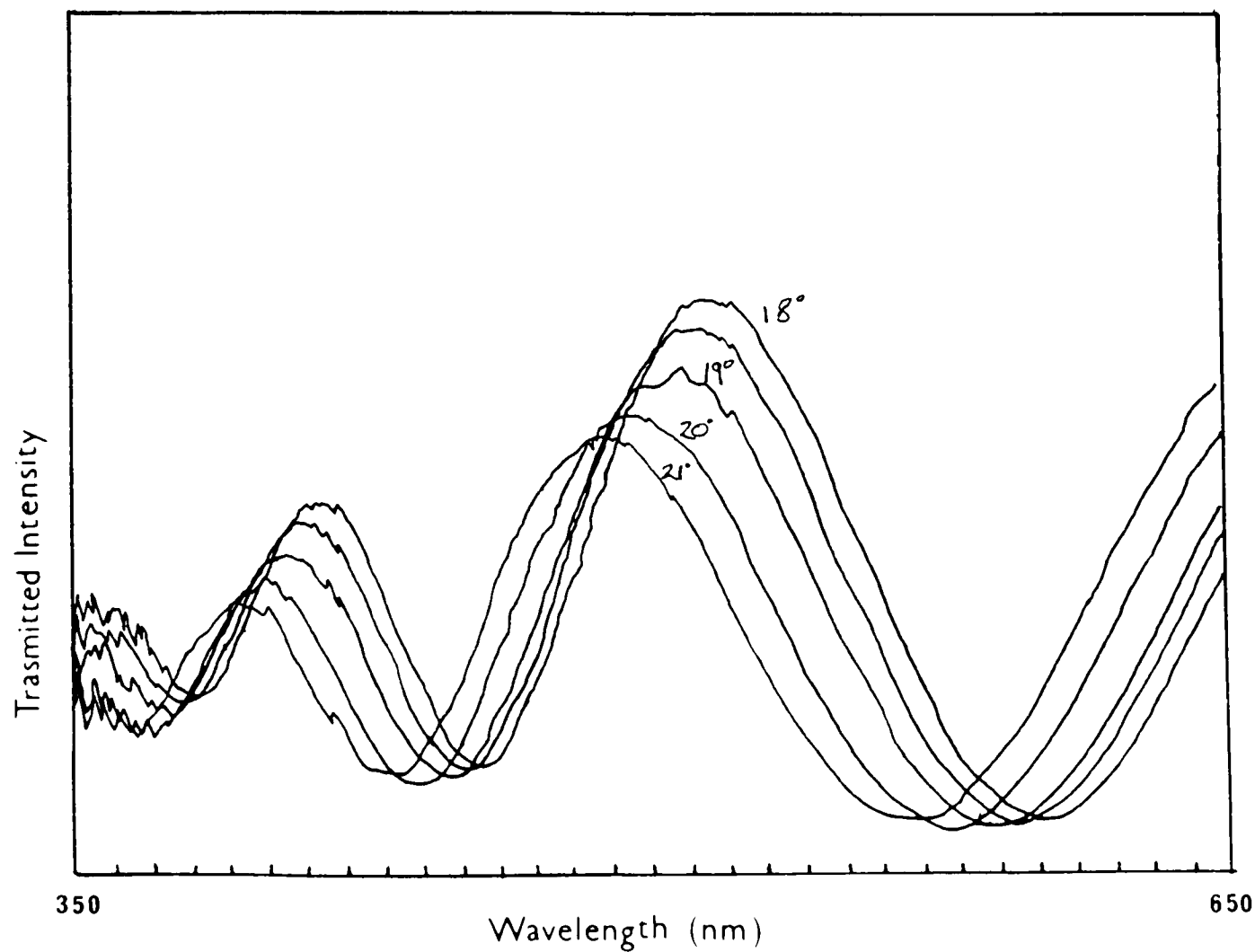
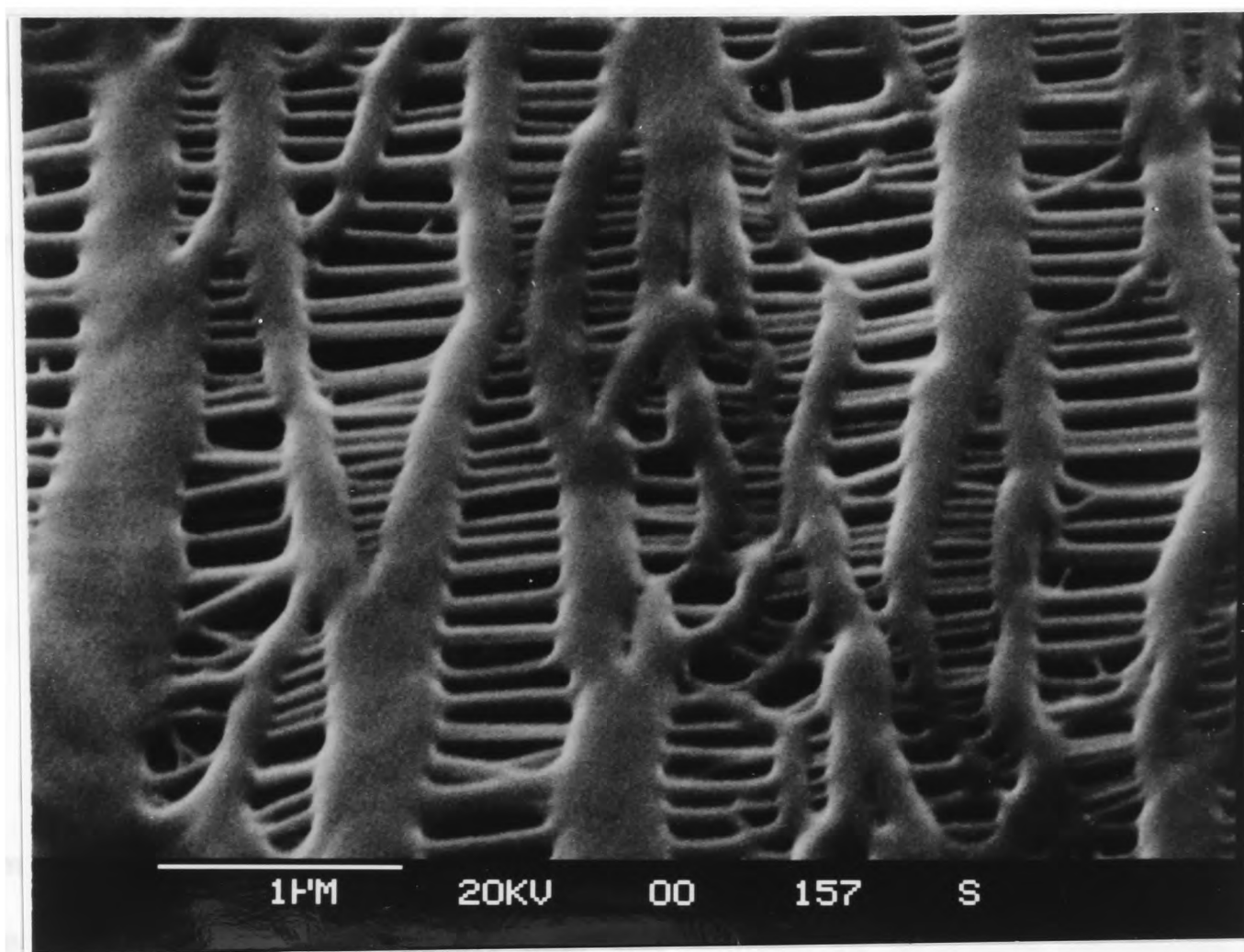


PLATE 8. Scanning electron microscope photograph of the structure of Celgard 2500.



photograph of the celgard structure.

One of the optically transparent liquid crystal binder materials, such as Mowilith, was used to adhere the celgard layer to the test surface, and ensured uniform thermal contact without degrading the liquid crystal.

The transmission spectrum, and its variation with temperature, as measured by the author, are shown in figures 38 and 39, respectively. A number of peaks occur in figure 38 corresponding to the transmitted intensity maxima which are given by the expression:

$$\frac{I}{I_0} = \sin^2 \left(\frac{d \cdot 2\pi \cdot \Delta n}{\lambda} \right)$$

Similar colour changes are possible in reflection. A number of peaks are present at any one temperature, and these are integrated by the eye, depending on their relative intensities, to appear as a colour. The peak wavelengths change very rapidly near the nematic to isotropic transition as the birefringence falls to zero. The temperatures obtainable are only limited by the melting temperature of the celgard material, which occurs at about 160°C.

The colour peaks do have some angle dependence, which must be taken into account if curved models are used. The colours should ideally be calibrated under the test lighting conditions. Figure 40 shows the peak colour variation with viewing angle at a number of different temperatures.

A simple test was carried out using this method, to visualise heat transfer around a pedestal subjected to a hot flow. Plate 9 shows the results of this test, which clearly demonstrates the bright, distinct colours which are possible. The colour changes are of course completely reversible and can be made to occur over a wide temperature range by a suitable choice of material. It is also possible to use this method in conjunction with the previous

FIGURE 39. Variation of reflection peaks with temperature for K15 in celgard.

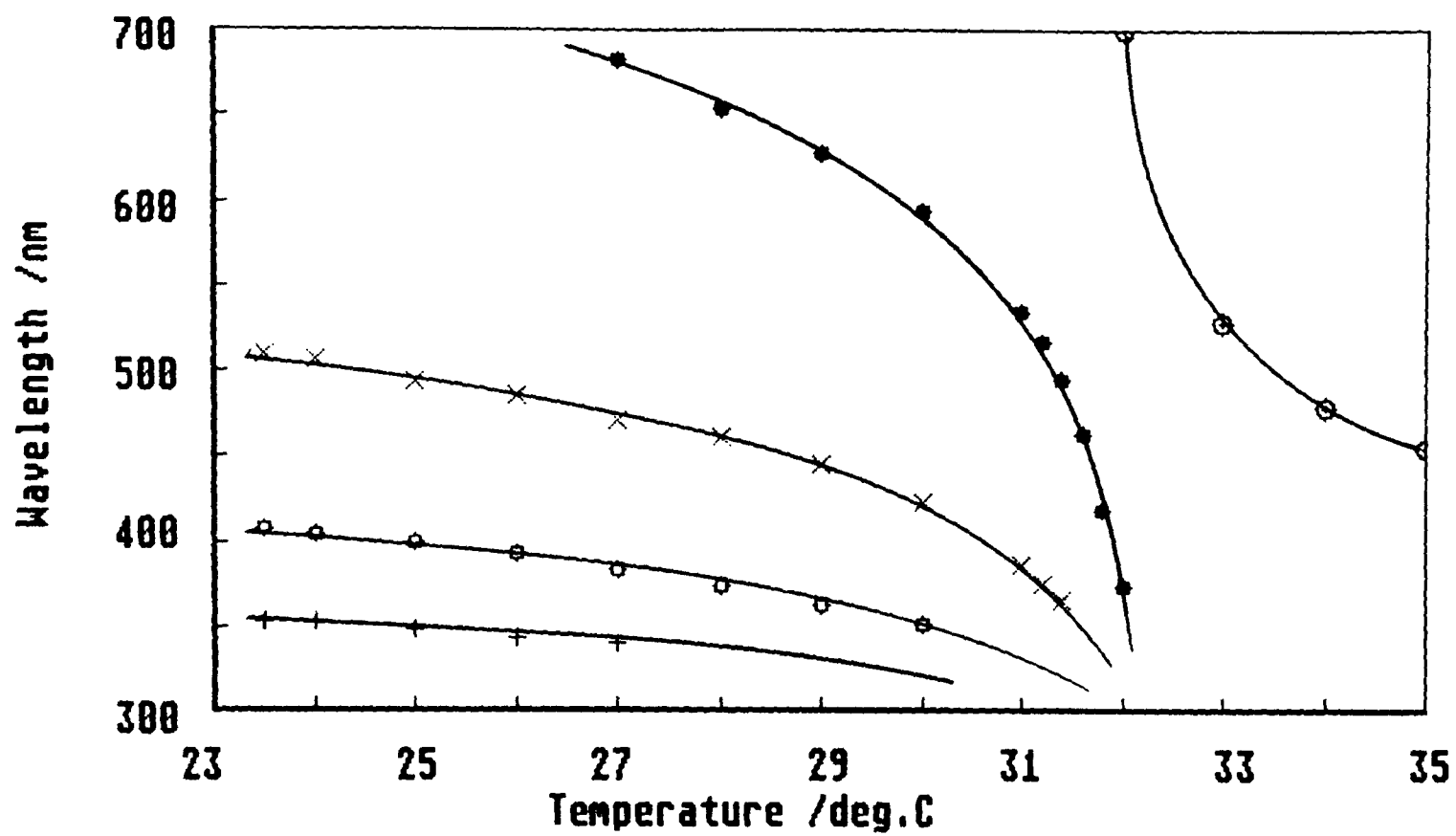


FIGURE 40. Viewing angle dependance of the transmitted wavelength through K15 in celgard between crossed polarisers.

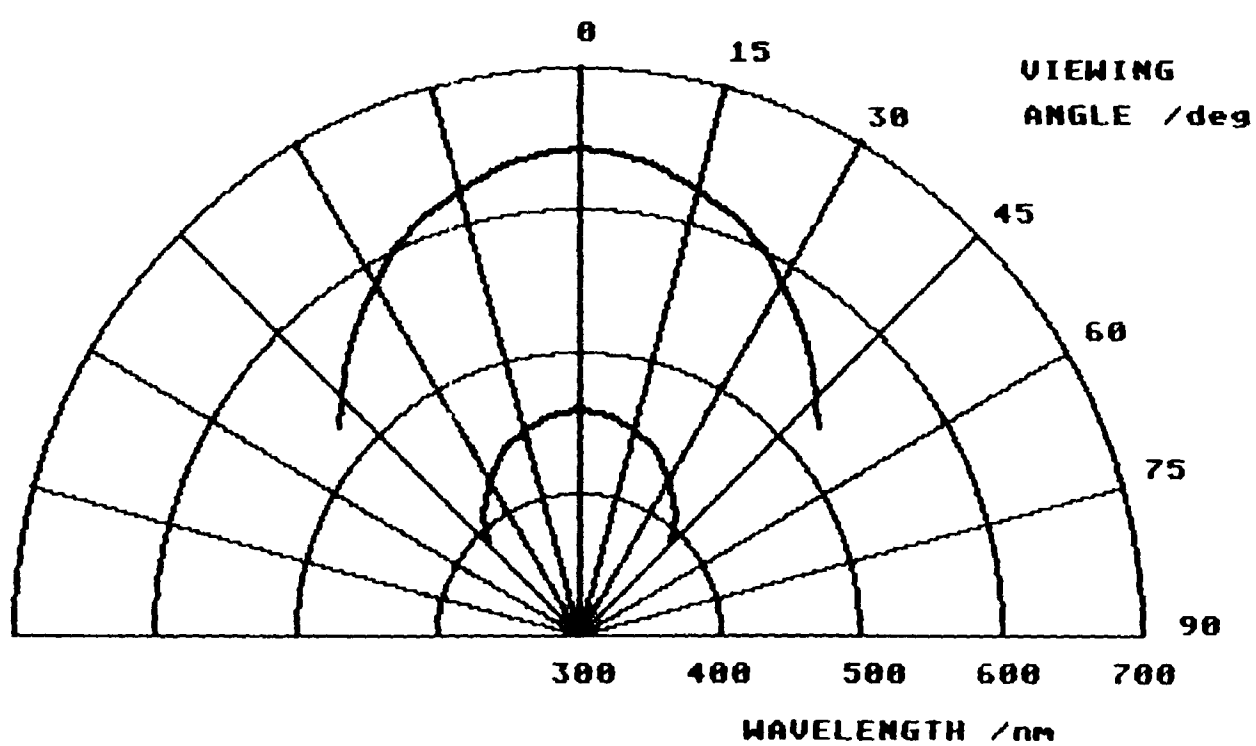


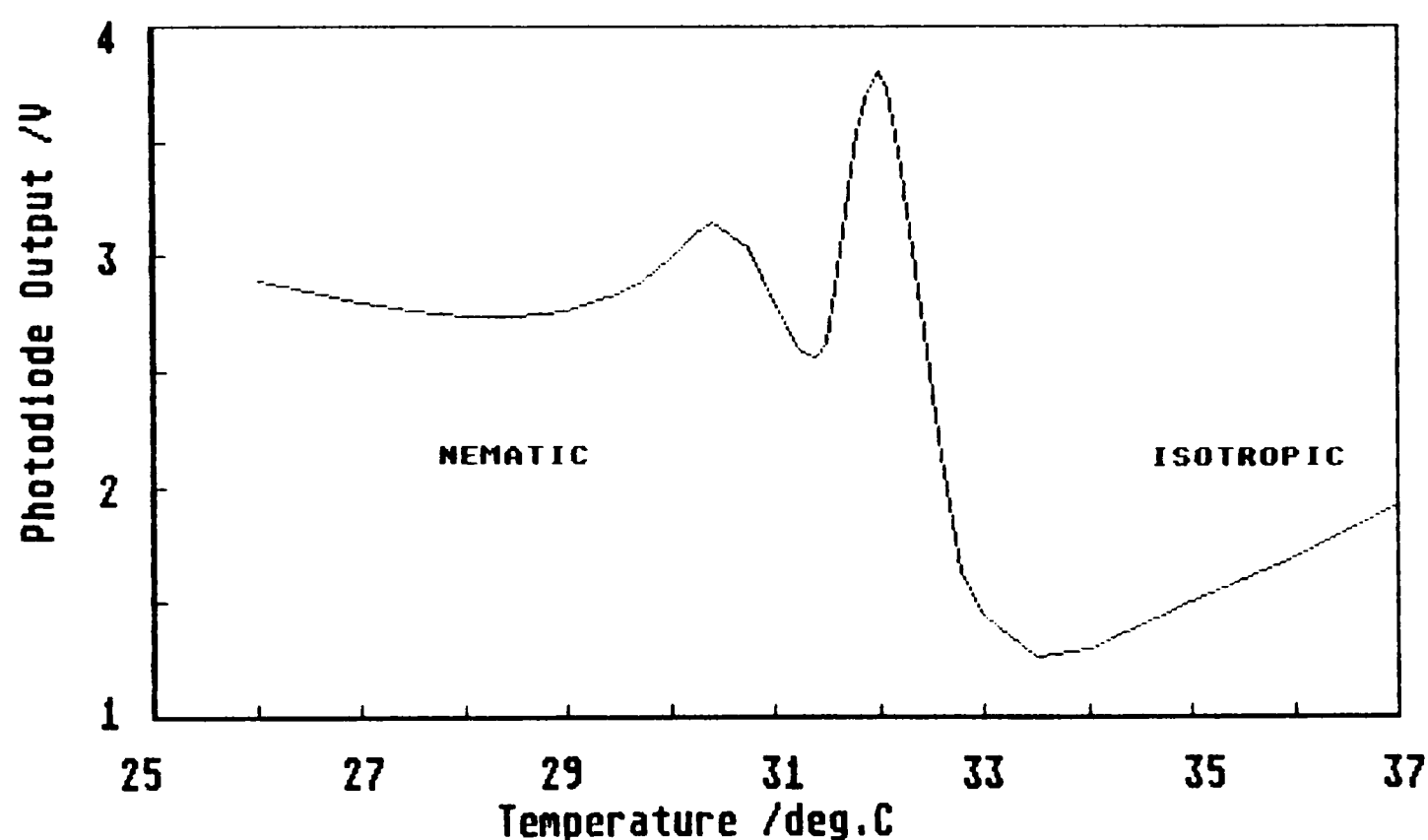
PLATE 9. Sequence of photographs demonstrating the nematic birefringence method of measuring surface temperature. The nematic is held in a celgard layer adhered to the surface. The sequence shows the surface temperature pattern around a pedestal subjected to hot gas flow from left to right.



method, in order to measure distant high and low temperatures. The cholesteric to isotropic transition material (silk screen printed on top of the celgard) would be used to measure the low temperature. This material would then go isotropic and so not effect the retardation changes experienced by light at a higher temperature due to the nematic layer.

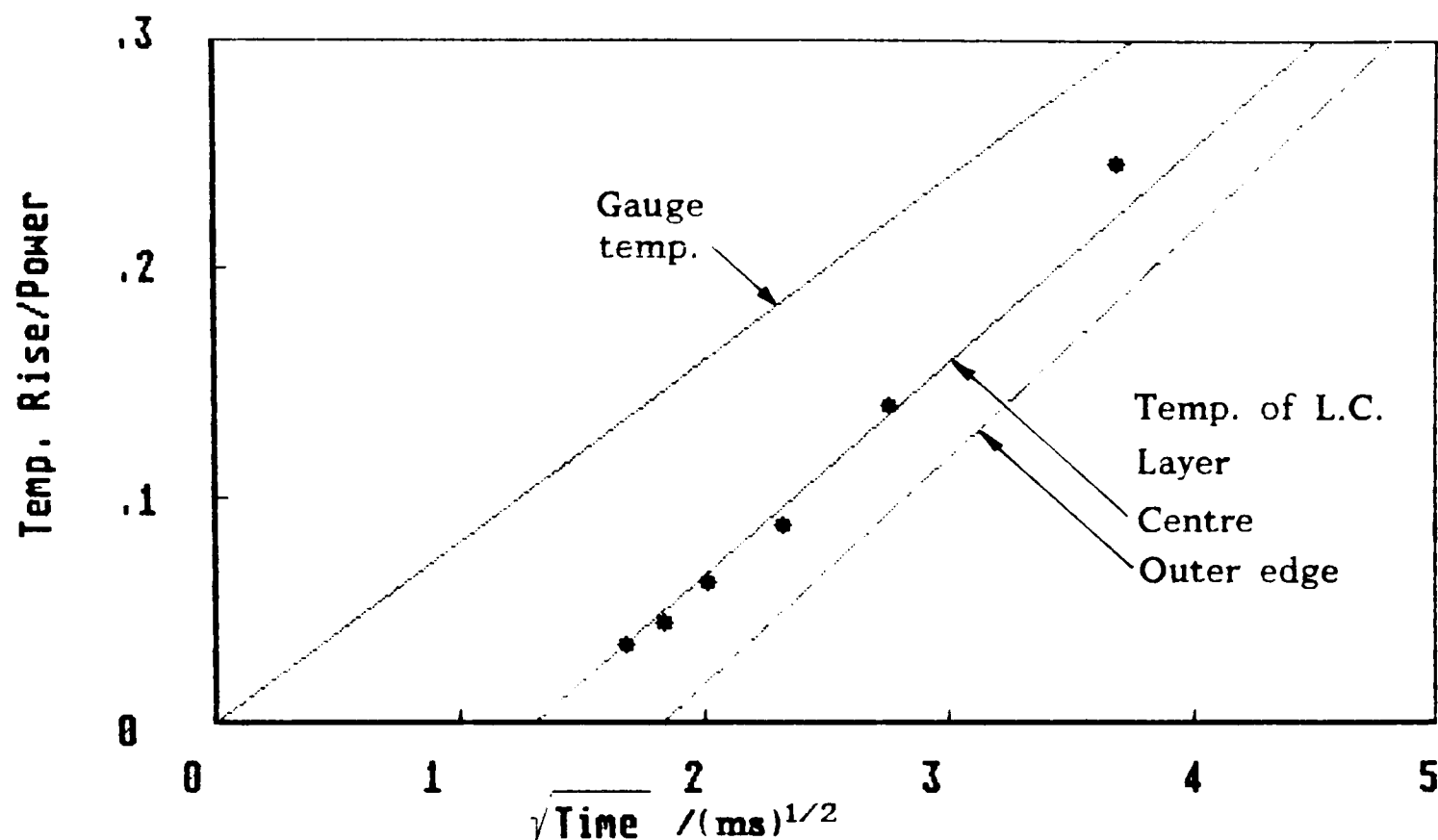
The heat pulse measurement method was again applied in an attempt to measure an intrinsic response time for this method. This was measured with respect to the colour that gave the maximum photodiode output (i.e. not at the phase transition). Figure 41 shows the equilibrium photodiode output for K15 in Celgard as a function of temperature.

FIGURE 41. Equilibrium photodiode calibration for the birefringence method. (K15 in Celgard)



The ambient temperature was set at 5°C below this colour change. It was expected that this response time would be extremely small as the birefringence should change instantaneously with temperature.

FIGURE 42. Temperature rise over power against square root of time for birefringence method. Points are experimental results for K15 in Celgard. This indicates that the only significant lag is thermal conduction.



The thermal conduction times for the centre and far edge of the 35 μm thick celgard layer (including thickness of glue to stick layer to surface) to heat by 5°C were calculated and plotted together with the measured total response times in figure 41. There is some uncertainty about where to set the reference position for such a thick layer, but it is clear that the intrinsic response time of the method is insignificant when compared to the thermal conduction time.

A patent has been filed by the Secretary of State for Defence on this method, with P.Bonnett, T.V.Jones, D.G.McDonnell and E.P.Raynes as inventors. Patent application number 88 04177.

3:5.3. Comparison of Methods

As the time constant calculation procedures were different between these methods they were assessed comparatively by the technique below.

The experiment compared the response of the selective reflection colour of the encapsulated liquid crystal with the birefringent colours of the K15, in which both materials were applied to ITO gauges on glass substrates, and subjected to the same current pulse. The reflection response was recorded with a high speed camera operating at 400 frames per second, and is shown in Plate 10. In the photograph the cholesteric colour change is invisible, although it was just visible by eye in the original test. However it was clear that the celgard birefringence colours were still considerably clearer and brighter, even in the presence of the large temperature change and temperature gradients produced in this test.

3:5.4. Conclusions

1. The preferred method for low heat transfer rate tests is the use of the colour changes in a cholesteric liquid crystal as the material is easy to apply to a variety of surfaces, easy to interpret and provides very good surface temperature visualisation.

2. The phase change technique offers a x10 improvement in measureable heat transfer and also incorporates thermal memory.

3. The birefringence method has the best intrinsic response time, but further work is required to (a).reduce its thermal mass by using a thinner celgard layer to reduce its thermal conduction time, and (b).develop a reflective system for greater flexibility in use. Promising initial tests using celgard in reflective mode have been carried out, but further optimisation is required. If these advances are successful the birefringence method would become the most versatile and accurate of the three.

PLATE 10. High speed photographic sequence (400 frames per second) comparing the birefringence colours in a nematic layer in celgard with the selective reflection colours from an encapsulated cholesteric liquid crystal when both are in contact with an ITO layer subjected to a heating pulse of the same power.



3:6. PARAMETER MEASUREMENTS

This chapter describes the methods used to measure the relevant liquid crystal physical parameters used in this study, which include viscosity, thermal product, density, specific heat capacity and thermal conductivity.

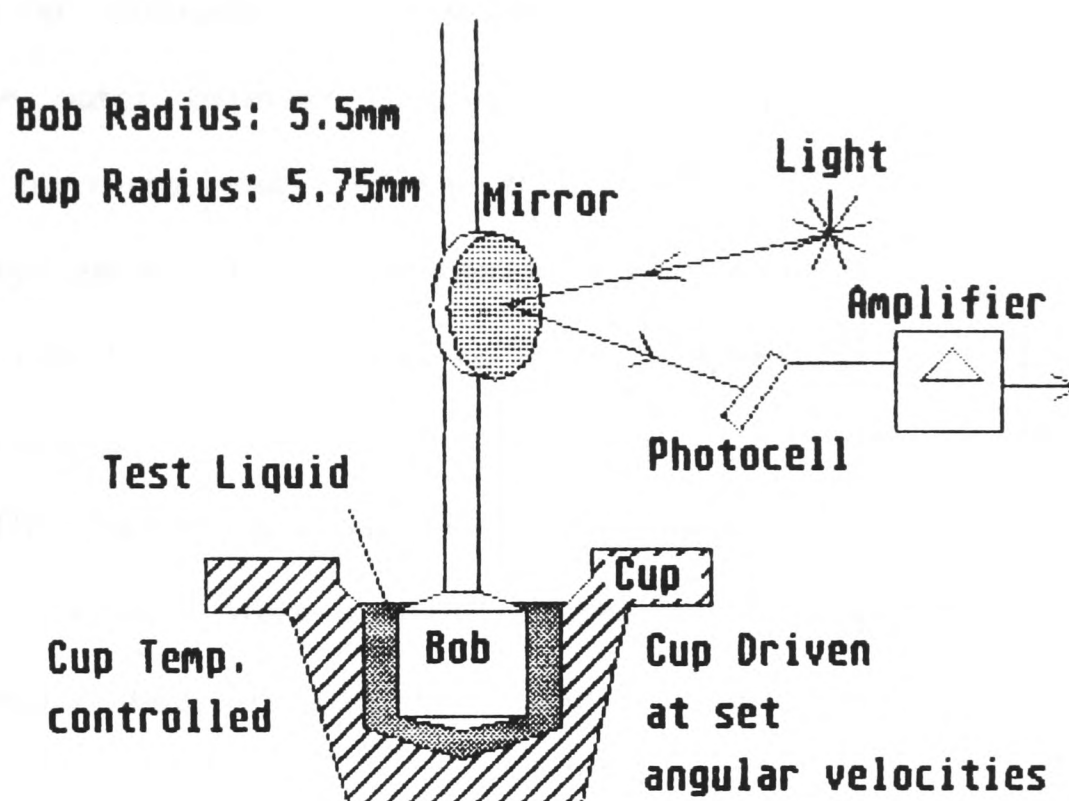
3:6.1. Viscosity

The viscosities of liquid crystals are much more complex than those of isotropic liquids. Leslie^{13,14} has defined five independent viscosity coefficients which arise in these anisotropic fluids, but in practice these are not straightforward to measure and full data exists for few materials. Nevertheless a useful guide to how these scale in different materials can be obtained by measuring bulk viscosities.

However even the bulk viscosity is difficult to measure directly in samples of smectic and cholesteric phases since their flow behaviour is highly non-Newtonian, i.e. the viscosity is dependent on the shear. Therefore to enable a comparison of these materials, measurements were made in the isotropic phase and the values reported in tables 1 and 2 (in chapter 3:3) obtained by extrapolation of the linear plots of $\ln(\text{viscosity})$ verses $1/(\text{absolute temperature})$. Although this does not give an absolute determination since it ignores any discontinuity at the transition, it does provide a useful comparison between materials assuming that the activation energies in both liquid crystal and isotropic phases are the same. More detailed viscosity studies on liquid crystals are presented in chapter 4:3.

Viscosities were measured on a Contraves Low Shear 30 concentric cylinder viscometer, in which one cylinder is driven at a known angular velocity. The liquid transmits the torque between the cylinders and the force on the second cylinder is measured. The cylinder height is 1, and the liquid is held within $r=a$ to $r=b$. See figure 43.

FIGURE 43. Concentric cylinder arrangement used in the Contraves Low Shear 30 viscometer.



If the liquid at r has the angular velocity ω , the velocity gradient is given by:

$$\frac{d(\omega r)}{dr} = \omega + r \left(\frac{d\omega}{dr} \right)$$

$$\text{Viscous force at } r = A \cdot \eta \cdot r \left(\frac{d\omega}{dr} \right) = 2 \cdot \pi \cdot r^2 \cdot \eta \cdot \left(\frac{d\omega}{dr} \right) \cdot l$$

$$\text{Torque, } T = \text{force} \times \text{length} = 2 \cdot \pi \cdot r^3 \cdot l \cdot \eta \cdot \left(\frac{d\omega}{dr} \right)$$

$$\text{Therefore } \int_a^b \frac{T}{r^3} dr = 2 \cdot \pi \cdot l \cdot \eta \cdot \int_0^{\omega_0} d\omega$$

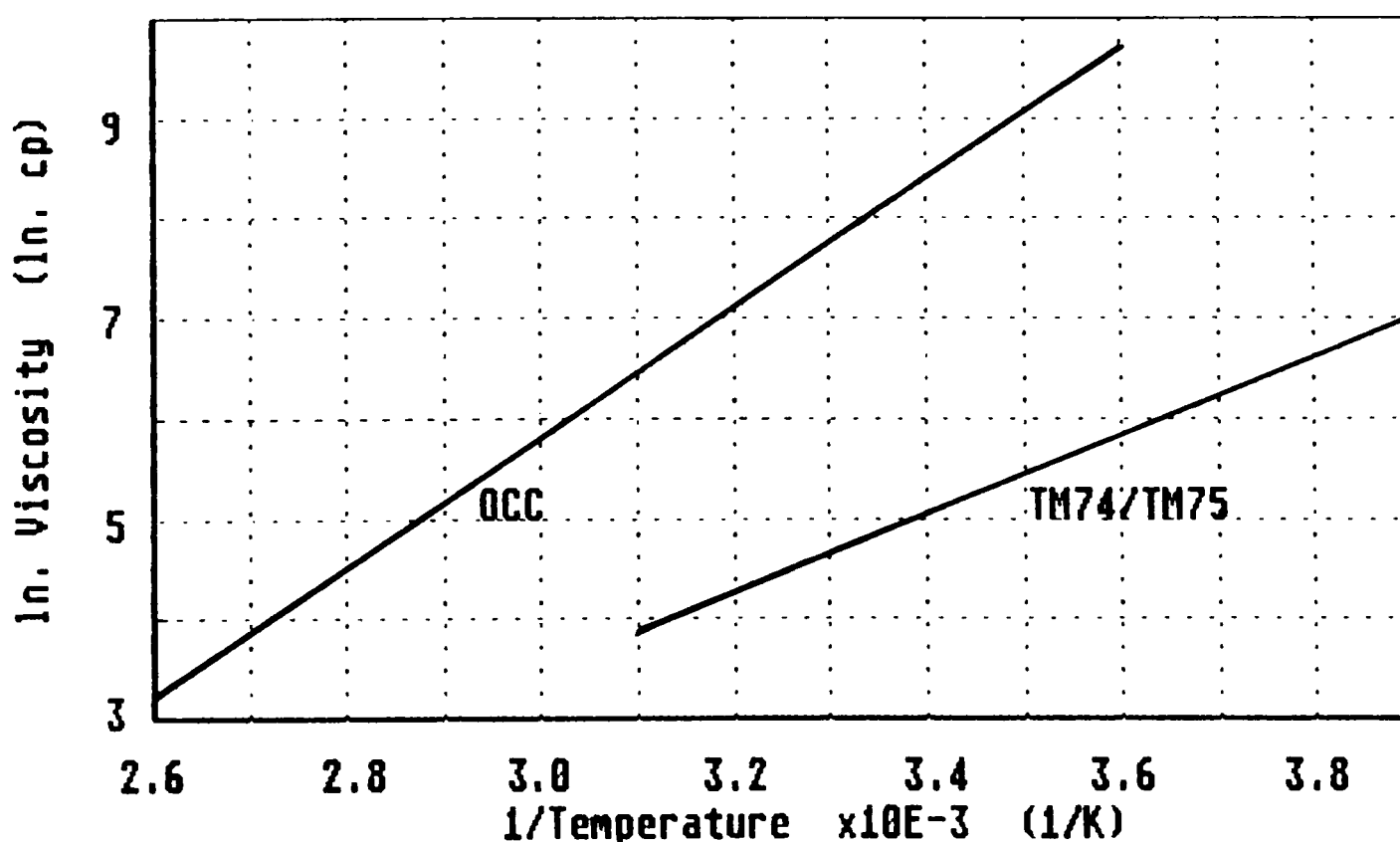
$$\text{Therefore } T = 4 \cdot \pi \cdot l \cdot \eta \cdot \omega_0 \cdot \frac{a^2 \cdot b^2}{(b^2 - a^2)}$$

Since the cylinder geometries are known, the viscosity may be calculated for the measured values of torque and angular velocity. This can be carried out at different temperatures by circulating a thermostated fluid through the outer cylinder. Liquid viscosities have an Arrhenius type of dependence on temperature, of the form $\eta = \eta_0 \cdot \exp.(-E/kT)$. Hence plots of $\log.(\text{viscosity})$ against $1/\text{absolute temperature}$ were plotted to produce straight lines which could be extrapolated to lower temperatures.

Results

The results obtained on the materials studied are plotted in figure 44, and these results were used in the thermal time constant plot. The same method was also used to study the effects of the aerosil additive on viscosity, see chapter 4:3.

FIGURE 44. Ln.Viscosity verses $1/\text{absolute temperature}$: Results for TM74 and TM75 mixtures and cholesteryl oleyl carbonate. (i.e. the TM74/75 mixture which has a Sm-Ch transition at $1/T = 3.5 \times 10^{-3}$ ($\sim 13^\circ\text{C}$) has a viscosity of $\exp.5.5$ ($\sim 244\text{cp}$), and so on.)



3:6.2. Density

(i). Neat Liquid Crystal Material

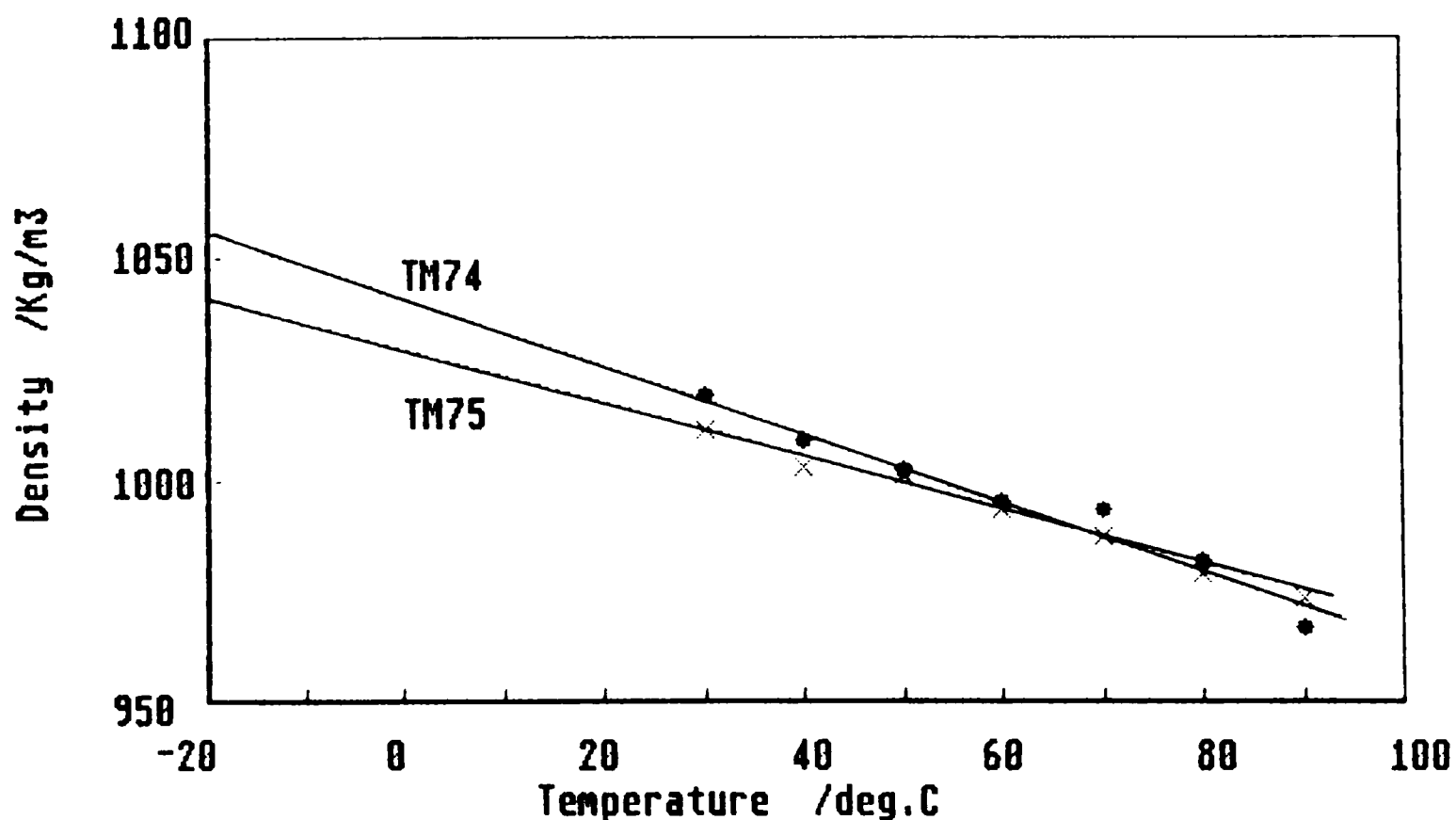
The density of the fluid was measured using an Anton-Paar densitometer, model DMA60. A u-shaped tube of known volume was electromagnetically vibrated. This may be thought of as analagous to a tuning fork. The resonant frequency is dependent on the density of the material filling the tube. This may be compared to the resonant frequency when the tube is filled with a material of known density, to find the density of the test material. The tube was temperature controlled, enabling the density as a function of temperature to be determined. The densitometer carried out the required calcuilations and output the result directiy.

Results

Density as a function of temperature for TM74A and TM75A is shown in figure 45. At 25°C the values are:

	TM74A	TM75A
density/kg.m ⁻³	1025	1017

FIGURE 45. Density verses temperature : results for TM74 and TM75



(ii). Encapsulated Liquid Crystal

A more direct assessment of the density of the encapsulated material was enabled by the availability of large samples ($\sim 8\text{cm}^3$). These were weighed and immersed in water, noting the displaced volume.

Results

Average value : 1040 kg.m^{-3}

3:6.3. Specific Heat Capacity

Specific heat capacity measurements were made using a differential scanning calorimeter. (Mettler DSC30 with a TC10A Processor) The device contained two small trays in contact with two thermocouples. Into one a known mass of a test material was placed, whilst the other was used as a reference. The amount of heat required to take each sample through a certain temperature rise could then be measured. The specific heat capacity was calculated from:

$$q = m.c.\Delta\vartheta$$

If the same amount of heat is applied to both samples then:

$$q_1 = m_1.c_1.\Delta\vartheta_1 = m_2.c_2.\Delta\vartheta_2$$

$$\text{Hence } c_1 = c_2.(m_2/m_1).(\Delta\vartheta_2./\Delta\vartheta_1)$$

$\Delta\vartheta_1$ and $\Delta\vartheta_2$ are measured by the thermocouples in contact with the trays. c_2 and m_2 refer to the specific heat and mass of the (empty) reference tray which are known.

Figure 46 shows a typical temperature scan. The specific heat capacity peaks as the material goes through phase transitions, but otherwise is fairly constant.

FIGURE 46. Typical differential scanning calorimeter output for a chiral nematic liquid crystal. Major phase changes are clearly indicated, as well as the almost constant specific heat capacity in the liquid crystal phase.

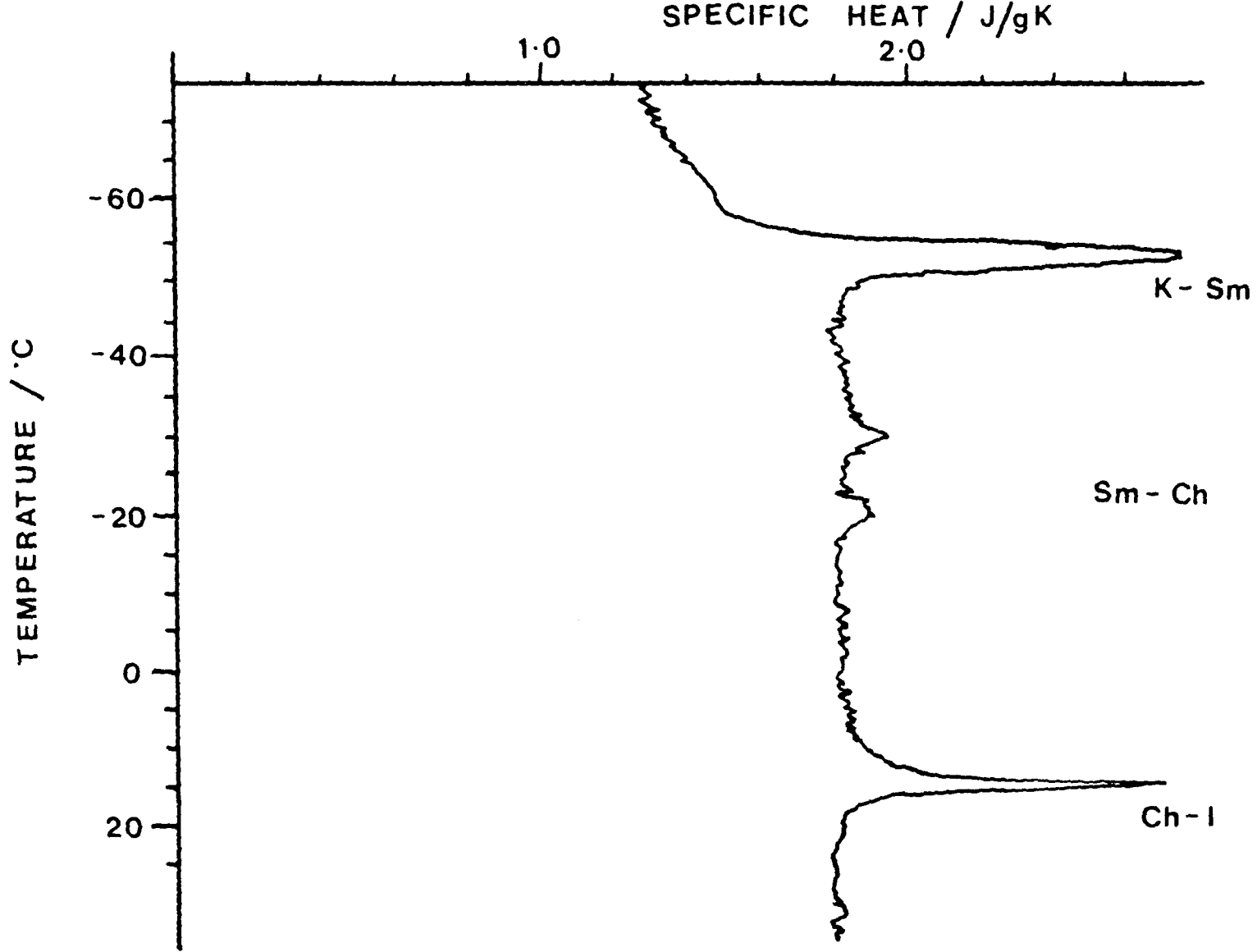
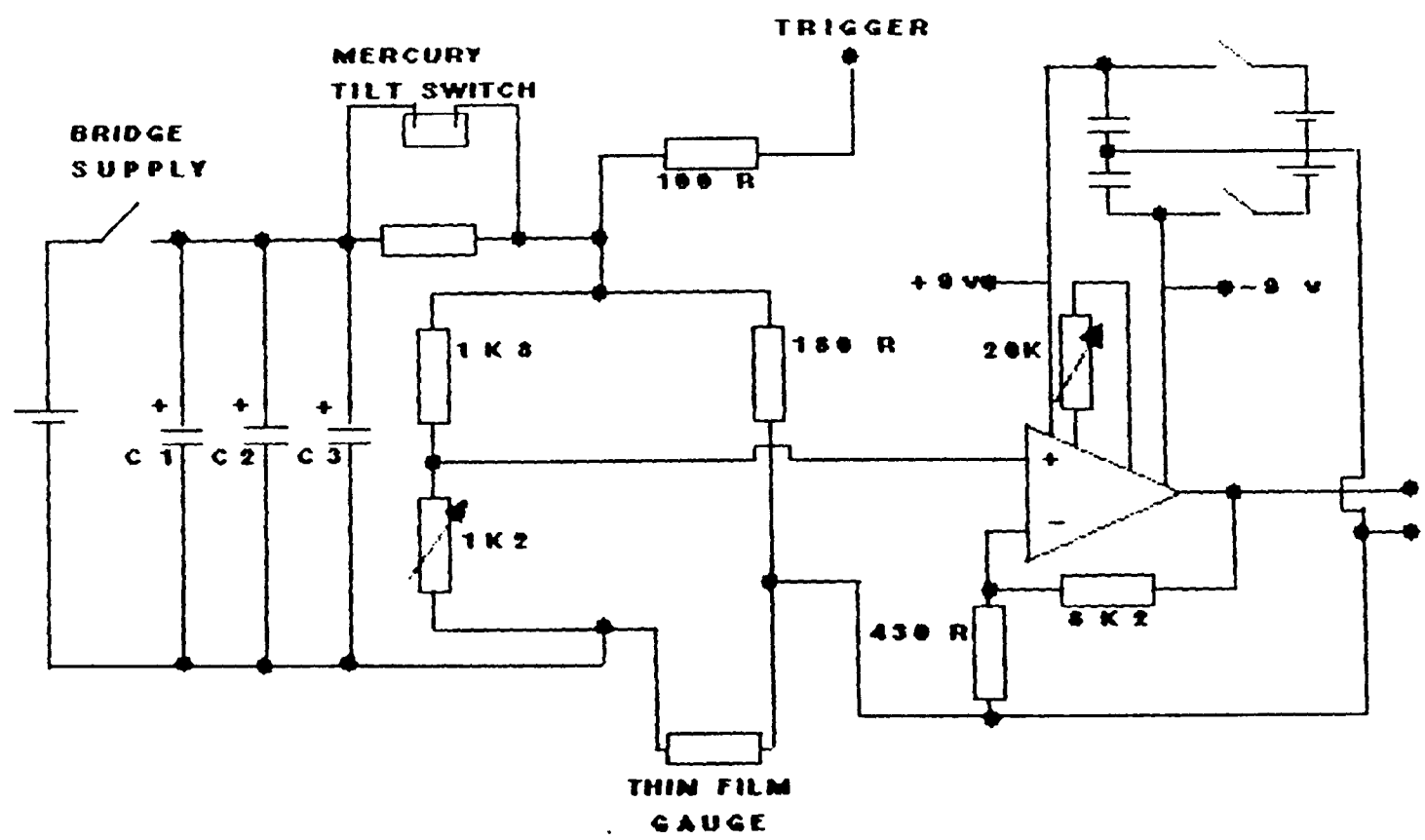


FIGURE 47. Circuit diagram for the thermal product current pulsing experiment.



Results

at 25° C	TM74	TM75	TCC107	B354
specific heat/JKg ⁻¹ K ⁻¹	1870	1830	1700	1730

3:6.4. Thermal Product

The thermal conductivity and diffusivity data obtained for encapsulated and neat liquid crystal materials were used in thermal modeling calculations of liquid crystal layers on heated surfaces, and to determine the thermal time constants. Measuring the thermal conductivity of a liquid incorporates a high degree of error, therefore the density and the specific heat capacity were measured separately and a novel method developed at Oxford⁵¹ was used to measure the thermal product. The thermal product, $\sqrt{\rho c K}$, is the quantity of most importance in thermal analysis since it dictates the rate at which heat is transferred into a material. Again these measurements were carried out on a bulk sample, and gave some average value rather than attempting to resolve the anisotropic components since previous studies⁵² have shown that the degree of anisotropy in thermal conductivity values is not great.

The measurement equipment used is shown in figure 47. A thin platinum film resistance gauge, deposited onto a ceramic substrate, was subjected to a pulse of current. The gauge temperature rose parabolically over short times as it lost heat to the substrate. It may be assumed that there was negligible heat loss to the air. The heating rate was dependent on the thermal product of the substrate. When a liquid was applied over the gauge and the gauge was pulsed again the parabolic heating was at a lower rate as a consequence of the extra heat transferred from the gauge to the liquid. This extra heat loss was dependent on the thermal product of the liquid. This was the basis of the measurement, which required one test liquid of known thermal product to calibrate the substrate.

The gauge acted as one arm of a Wheatstone bridge, which was initially balanced. As the gauge heated its resistance increased and produced an output from the bridge, which was proportional to the gauge temperature change:

$$\Delta v = V.\alpha.\Delta T. = V.\frac{\Delta R}{R}$$

α = temp. coeff. of resistance

V = voltage across bridge

Δv = bridge output

ΔT = gauge temp. change

$$\text{but also } T = \frac{I^2.R.}{A\sqrt{\pi}. \sqrt{\rho c K}}$$

A = gauge area

This is the expression for one dimensional conduction , where the thermal product value refers to the substrate.

$$\text{Hence: } \sqrt{\rho c K} = \left(\frac{\sqrt{t}}{\Delta v} \right) \frac{\alpha.I^2.R.V}{A.\sqrt{\pi}}$$

The need to know the gauge area. A . α and I^2R can be eliminated by calibrating the instrument with a material of known thermal product.

$$\text{Thus: } \sqrt{\rho c K}_{\text{sub}} = \frac{\sqrt{\rho c K}_{\text{liquid}}}{\left(\frac{\Delta v_1}{\Delta v_2} \right) - 1}$$

From the ratio of the two heating curves (in air and with the known material), the thermal product of the substrate can be found and the same formula may be used to find the thermal product of the test liquid.

The measurements were recorded at short times enabling the 1-D approximation of heat conduction through the sample to be used with good accuracy. This was confirmed from a plot of Δv against $\sqrt{t}$. which remained linear over the first 5ms. Beyond this time lateral conduction becomes significant which would greatly complicate the calculations. Regression analysis

was carried out on plots of v_{air} against v_{liquid} at short times to produce a good value for the ratio of $\Delta v_1/\Delta v_2$.

Circuit Details

The gauge was pulsed by tripping a bank of three capacitors, which were charged by two 9volt batteries, to achieve a step function current pulse. A mercury tilt switch was used to eliminate any bounce on switching. The bridge sensitivity was maximised by making R_1 and the gauge resistance as close as possible, since

$$\Delta v = V \cdot \frac{R_1 \cdot \Delta R_g}{(R_1 + R_2)^2}$$

Only a small amount of material was required on the gauge, as the penetration depth over the times studied was shown by Schultz and Jones³¹ to be quite low. It has been shown that the depth at which the temperature is 1% of the surface value may be given by:

$$d \approx 4 \cdot \sqrt{kt}$$

$$\text{If } t = 5 \times 10^{-3} \text{ s and } k = 1 \times 10^{-7} \text{ m}^2\text{s}^{-1}$$

then d is only $\approx 90\mu\text{m}$ or 0.09mm .

Data was recorded by feeding the bridge output traces to a digital storage oscilloscope.

Both neat and encapsulated materials were tested. The latter were allowed to dry completely before pulsing.

Glycerin was used as a standard of known thermal product. The value used was $920 \text{ Jm}^{-2}\text{K}^{-1}\text{s}^{-1/2}$.

All measurements were taken at room temperature, however the temperature of the sample rises considerably during the pulse. Hence the technique assumes that the thermal product is essentially constant over the operating temperature range.

Results

<u>Material</u>	<u>$\sqrt{\rho c K}$ /Jm⁻²K⁻¹s^{-1/2}</u>	<u>Error</u>
Encap. L.C.	620	+/- 60
Binder	630	+/- 60
TM74	550	+/- 60
TM75	680	+/- 60

Despite the large spread in the results it is clear that the thermal product values are of the same order as those for perspex ($\sqrt{\rho c K} \sim 600$). This has the benefit that the encapsulated liquid crystal layers which are used on perspex models will have little effect on the heat flow into or out of the model. The thermal product results have been used to calculate the thermal conductivities and diffusivities given in table 4.

It was decided to measure the thermal conductivity independently, for two reasons. Firstly, to improve the accuracy of the figures given in table 4, and secondly, so that conductivity as a function of temperature could be found.

3:6.5. Thermal Conductivity

The technique chosen was a steady state method involving the use of concentric cylinders⁵³, since this apparatus was relatively straightforward to produce and gave a direct measurement of the thermal conductivity at controlled temperatures.

After a few prototypes were tried, the final design, shown in figures 48 and 49, was constructed. The tubes were made of copper, so that their thermal resistance was insignificant compared to the liquid crystal. A heater coil in a ceramic spacer ran through the central copper tube. Thermocouples were connected to the inner and outer tubes using high conductivity silver loaded adhesive. The tubes were held apart using plastic

TABLE 4. Results of thermal parameter measurements for liquid crystalline materials used in this thesis.

Parameter	TM74	TM75	Encap. Material	Est.% Error	Units
Density	1019	1011	1040	1	Kg m ⁻³
Spec. Heat	1870	1830	1730	1	J Kg ⁻¹ K ⁻¹
Thermal Prod.	550	680	620	10	J m ⁻² K ⁻¹ s ^{-1/2}
Conductivity	0.16	0.25	0.21	20	W m ⁻¹ K ⁻¹
Diffusivity, x10 ⁻⁷	0.84	1.35	1.2	20	m ² s ⁻¹

FIGURE 48. Design of concentric tube apparatus used to determine thermal conductivity of liquid crystals.

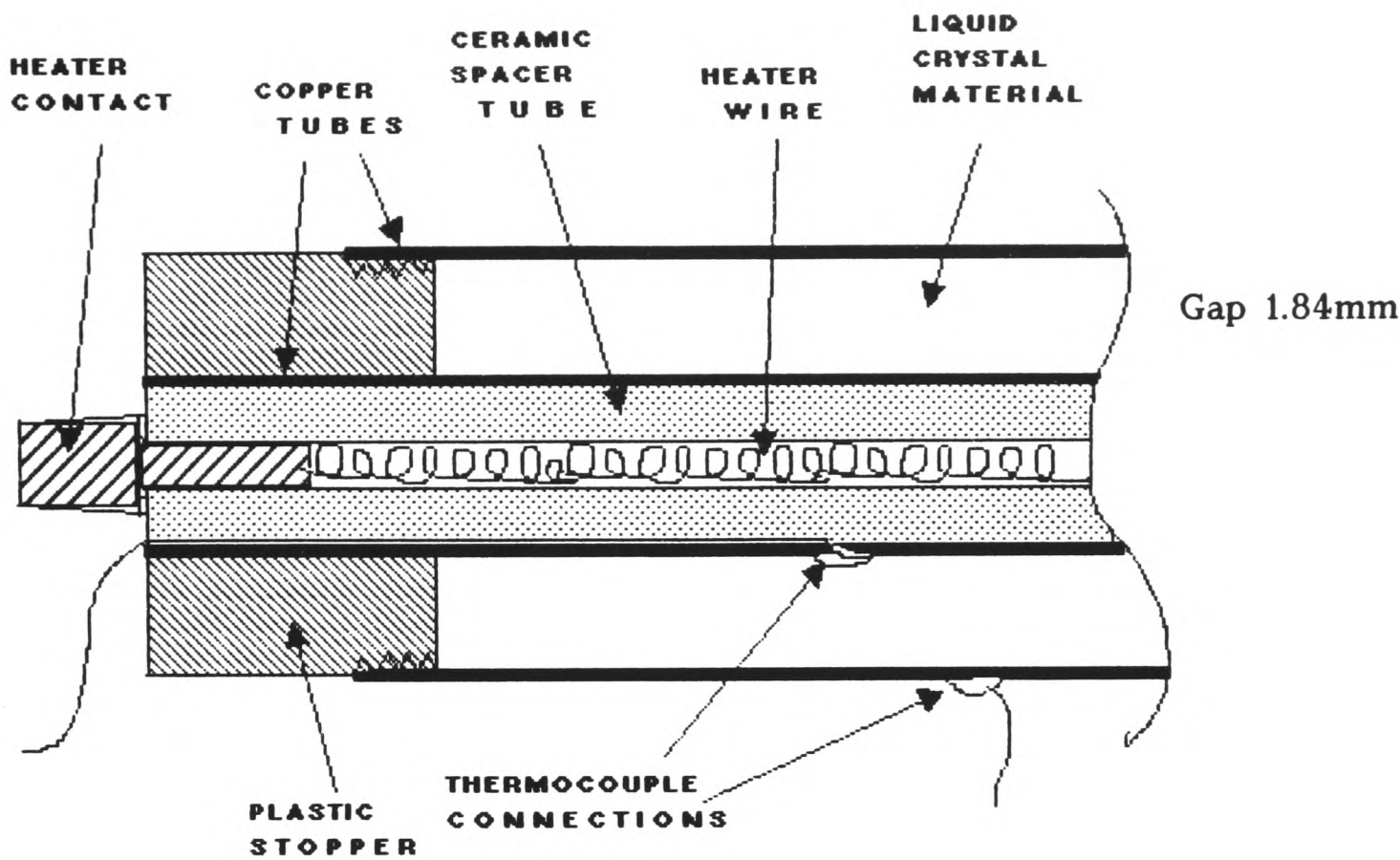
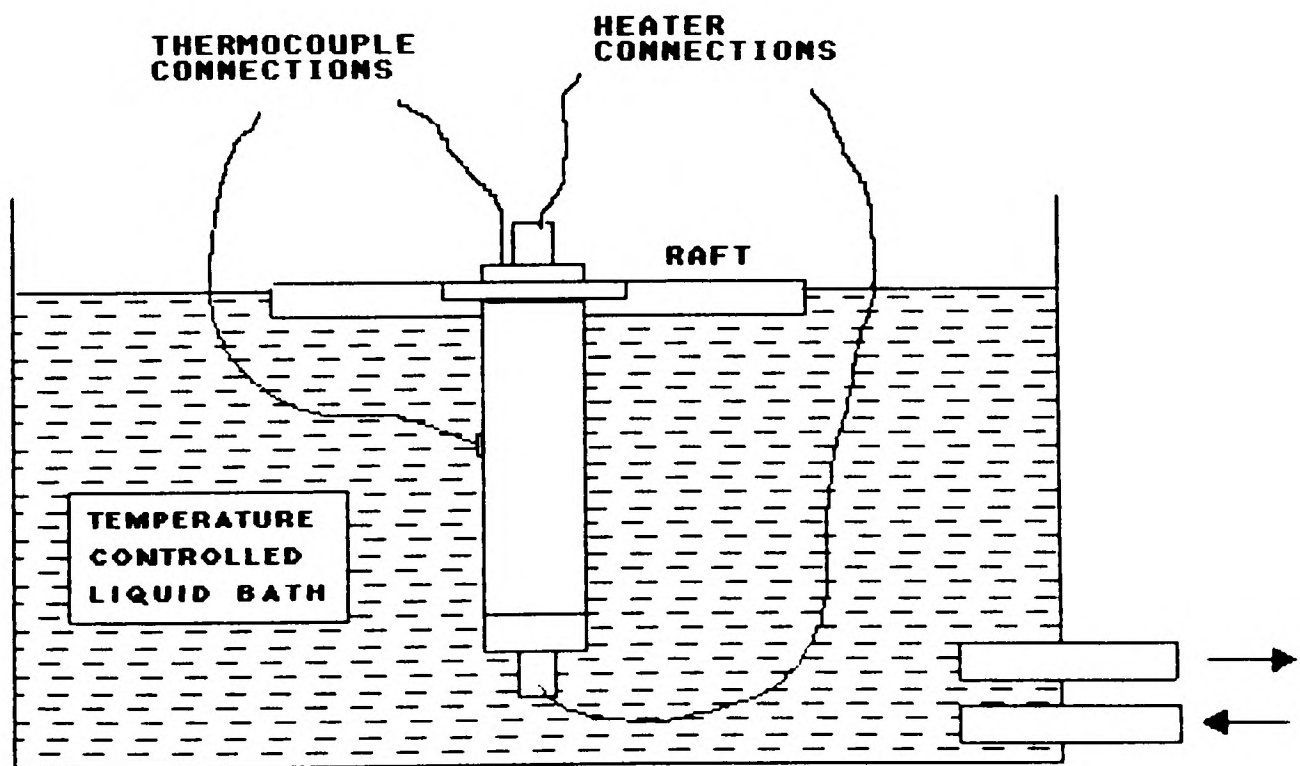


FIGURE 49. Temperature control of the concentric tube arrangement.



stoppers at each end, which also prevented high heat loss from the ends. The tubes were then immersed in a temperature controlled liquid bath, so that a wide range of temperature gradients could be reached quite quickly.

The thermal conductivity was calculated as follows, assuming radial heat flow:

$$q = \frac{dQ}{dt} = -K.A. \frac{dT}{dr} \quad \begin{array}{l} \text{Area, } A = 2.\pi.r.l \\ l = \text{tube length} \\ r = \text{tube radius} \end{array}$$

$$\text{therefore : } dT = -\left(\frac{q}{2.\pi.r.l.K}\right).dr$$

Once equilibrium is reached, q is independent of r , so we may integrate between r_1 and r_2 (i.e. over the liquid crystal gap).

$$\text{therefore } T = -\left(\frac{q}{2.\pi.l.K}\right).\ln.r + c$$

$$T_1 = -\left(\frac{q}{2.\pi.l.K}\right).\ln.r_1 + c \quad , \quad T_2 = -\left(\frac{q}{2.\pi.l.K}\right).\ln.r_2 + c$$

$$\text{So } T_1 - T_2 = \left(\frac{q}{2.\pi.l.K}\right).\ln.\left(\frac{r_2}{r_1}\right)$$

$$\text{or } K.(T_1 - T_2) = \left(\frac{V.I}{2.\pi.l}\right).\ln.\left(\frac{r_2}{r_1}\right)$$

as the heat is supplied electrically.

For our apparatus, $l = 84\text{mm}$, $r_1 = 3.16\text{mm}$, $r_2 = 5.00\text{mm}$

therefore $K(T_1 - T_2) = V.I. \times 0.8693$

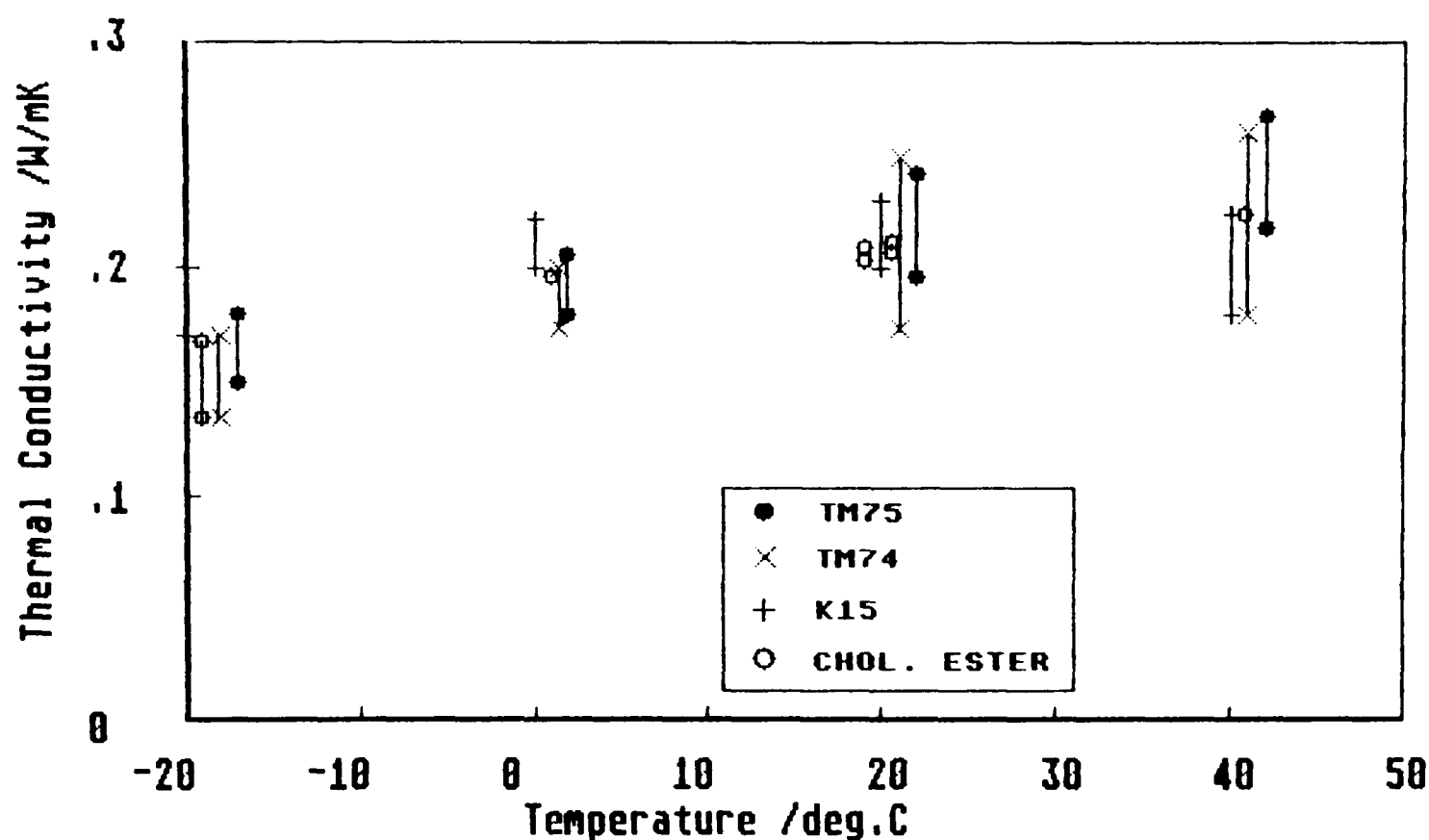
T_1 and T_2 were measured directly with thermocouples, so K could easily be found. There was no attempt to align the liquid crystal inside the tubes, so a bulk or average value of thermal conductivity was being measured. As well as liquid crystal samples, test measurements were also done using air,

distilled water and toluene. The liquid crystal materials used were TM74, TM75, K15 and a cholesteryl ester.

Results.

The results are plotted against temperature in figure 50. The room temperature values are listed below.

FIGURE 50. Results of thermal conductivity measurements on liquid crystals as a function of temperature.



<u>Material</u>	<u>K/Wm⁻¹K⁻¹ (spread)</u>	<u>Accepted value</u>
Air	0.070 +/- 0.010	0.025
Toluene	0.165 +/- 0.017	0.135
Water	0.525 +/- 0.1	0.594
K15	0.220 +/- 0.02	
TM74	0.19 +/- 0.03	
TM75	0.22 +/- 0.02	
Chol. Ester	0.21 +/- 0.005	

It is encouraging that the TM74/75 values compare quite well with the values obtained from the thermal product measurements. However the trend with temperature is opposite to that previously reported for liquid crystals^{54,55,56}. Also it has been observed that thermal conductivity in liquids scales with the density⁵⁷. Hence the possible errors present in this method will be examined in detail.

Errors

(A).Measurements.

(i).Temperatures - measured to within $\pm 0.1^\circ\text{C}$

Hence ΔT could be $\pm 0.2^\circ\text{C}$, giving an error of 4% in ΔT if $\Delta T=5^\circ$

(ii) .V.I. - measured directly with multimeter - error $\approx 0.3\%$

(iii).Tube dimensions - radii measured with micrometer to $\pm 0.02\text{mm}$ length measured to $\pm 0.5\text{mm}$. Error 3% in geometry term $(1/2\pi l) \cdot \ln(r_2/r_1)$.

(B).System Errors.

(i).Effect of radiation - this was determined to be negligible, as the measured value for K_{air} was the same for $\Delta T=2.7^\circ\text{C}$ as $\Delta T=10.5^\circ\text{C}$.

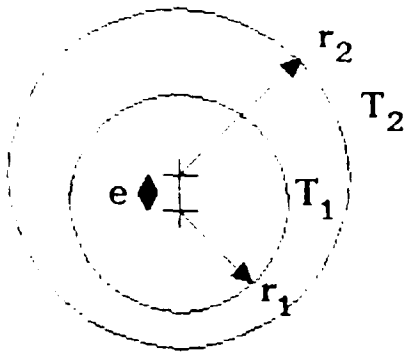
(ii) .Lateral Conduction - along the tube and out of the ends.

The tube length was much greater than the radius, and plastic ends were used to minimise lateral conduction, so it is assumed that this is not a source of significant error.

(iii).Contamination - by the fluid in the temperature controlled bath.

The extent of this factor is unknown, but the tubes were well cleaned between runs, and the tubes properly sealed.

(iv).Eccentricity of the inner cylinder - this can be accounted for with a shape factor, S , so that $q = K.S.\Delta T$.



$$S = 2.\pi.l./\cosh^{-1}\left(\frac{r_1^2+r_2^2-e^2}{2.r_1.r_2}\right)$$

$$\text{So } q = \frac{K.2.\pi.l.(T_1 - T_2)}{\cosh^{-1}\left(\frac{r_1^2+r_2^2-e^2}{2.r_1.r_2}\right)}$$

Note that $\cosh^{-1}.x = \ln.(x + \sqrt{x^2 - 1})$

We would expect the eccentricity, e , to be 0.5mm at most. This would reduce the measured value of K by about 3.6%.

(v) .Convection effects - due to the temperature gradient and possible thermo-mechanical coupling effects. Note that the spread in the results for the viscous cholesteryl ester are much less than for the other materials, and that the spread for all materials increases with temperature. Both these factors indicate that convection may be playing a part between the tubes.

The most significant factors appear to be the temperature measurement, eccentricity, and convection effects, the latter of which is probably the cause of the erroneous measured temperature dependence of the thermal conductivity. The estimated accuracy of the results is about 10%.

In summary, although the method provides a useful indication of thermal conductivity, because of experimental uncertainties it is no more reliable than the previously described dynamic technique. The values obtained were used in the calculation of the temperature rise in a pulsed liquid crystal layer in order to determine the material time constant.(chapter 3:3).

4. SHEAR STRESS

4.1. SHEAR STRESS

A knowledge of the drag over surfaces is extremely important in aerodynamic design since this affects vehicle characteristics and performance. Drag is the resistance to motion on a body caused by the fluid it is passing through, and is composed of shear drag and pressure forces (form drag) acting on the body. In this section only the shear stress is considered. This is a result of the viscosity of the fluid, and is a function of the fluid velocity, body geometry and surface finish. Minimisation of this drag can lead to improved performance and significantly reduced fuel costs, and so drag reduction is the subject of much military and civil aerodynamic research. Up to 50% of the fuel burned by commercial aircraft is used to overcome viscous drag⁵⁸, hence even small reductions in drag may significantly reduce fuel costs. The improvement would also enable higher speed and longer range to be achieved.

Adequate methods of measuring shear stress over aerodynamic surfaces are vital if improvements are to be assessed. Present methods, reviewed later in this chapter, are not entirely satisfactory and there is a clear requirement for a reliable surface shear stress mapping technique. A method utilising liquid crystals has been developed which may fulfill this requirement, and this is described and evaluated in a later section. A general introduction to the subject of surface shear stress and shear stress measurement will be given in this section.

Two distinct regimes of flow are known to exist, laminar and turbulent. There follows a brief description of the properties of each type of flow.

4:1.1. Laminar Flow

This occurs at low flow rates when the flow is ordered and without turbulence. In the region near a boundary surface a boundary layer develops due to the condition of no slip at the boundary. The shear force at any point is proportional to the velocity gradient at that point and has a finite value at the surface, see figure 51. The boundary layer is defined as the region where the fluid velocity increases from zero at the boundary to 99% of its free stream value at the edge of the boundary layer. For a smooth flat plate, (figure 51), the thickness of the boundary layer increases with distance. The plate exerts a retarding force on the fluid at the plate so that the layers decelerate and retard the motion of layers further from the plate. Hence the boundary layer grows and the velocity gradient drops (see figure 51.b.), reducing the shear force at the surface. When a certain thickness is reached the boundary layer becomes unstable and there is a transition to turbulent flow.

The shear stress is proportional to the velocity gradient. The maximum shear, at the wall, is given by:

$$\tau_o = \eta \cdot dv/dy \quad (\text{N/m}^2)$$

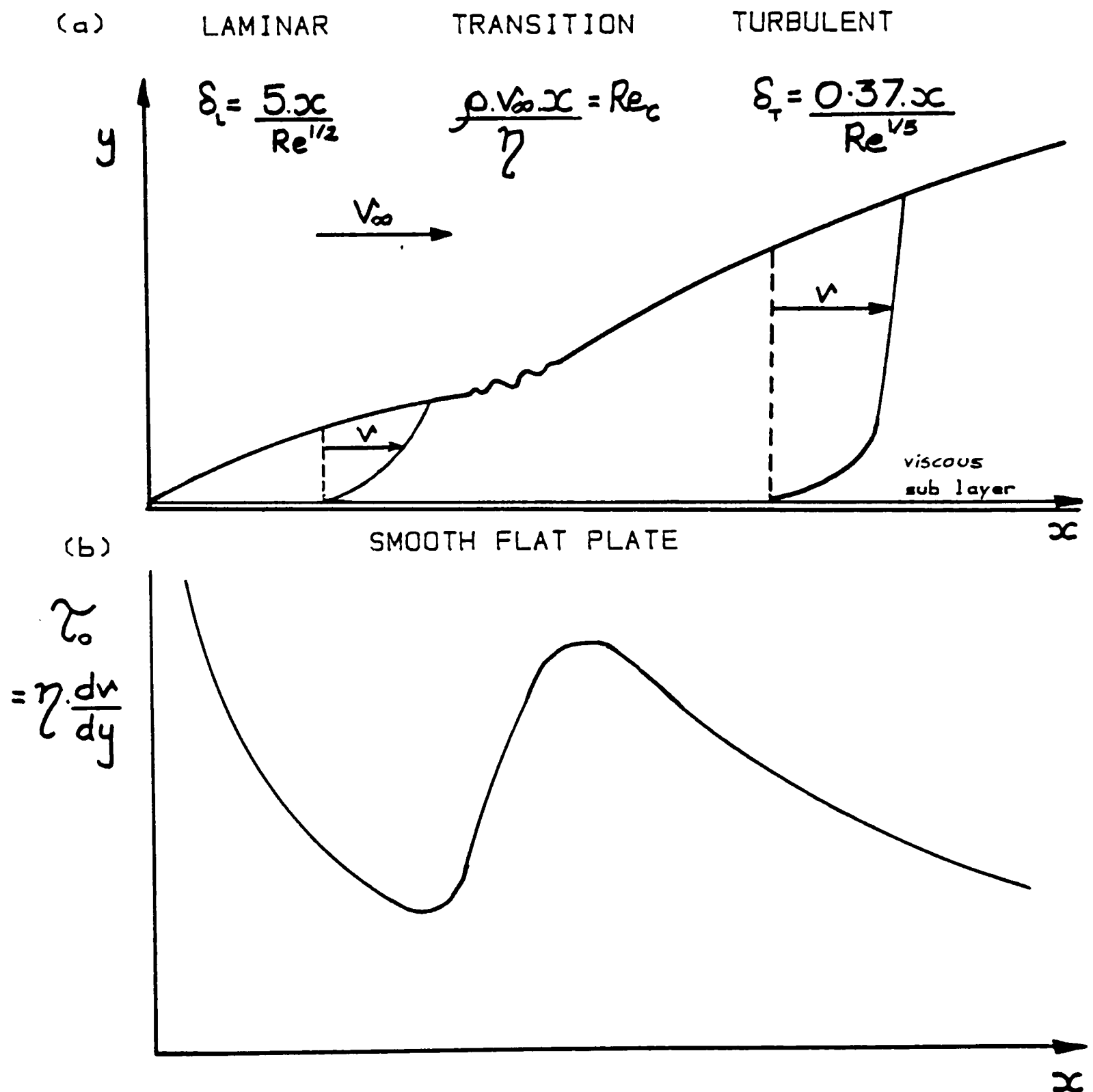
where η is the fluid viscosity (N.s./m^2) and dv/dy is the velocity gradient.

A skin friction coefficient may also be defined from:

$$\tau_o = C_f \cdot \rho \cdot v^2 / 2 \quad - - - 4.1 - -$$

where v is the free stream velocity and ρ is the fluid density. C_f is called the skin friction coefficient. Although many equations in aerodynamics have a basis in theory, because of their complexity many of the coefficients involved are determined empirically. Hence it has been found that the following equation

FIGURE 51. Schematic of (a) laminar and turbulent boundary layer thickness as a function of distance along a smooth flat plate, showing velocity profiles and (b) associated shear stress distribution.



δ = boundary layer thickness η = fluid viscosity

ρ = fluid density V_∞ = free stream flow velocity

τ_o = surface shear stress Re = Reynolds number

Re_c = critical Reynolds number = 5×10^5 for smooth flat plate.

is a good approximation for cases of laminar flow over a smooth, flat plate:

$$C_f = 0.686 \times Re^{-1/2}$$

where Re is the Reynolds number. This is defined as:

$$Re = v.d.\rho./\eta = v.d./\nu$$

where d is a characteristic distance of the system (usually the distance of the point of interest from the leading edge of the plate), and ν is the kinematic viscosity, and is given by :

$$\nu = \eta/\rho \quad (m^2/s)$$

The Reynolds number is a dimensionless quantity and is a measure of the relative magnitudes of inertial to viscous forces occurring in flow, as ρv^2 can represent inertial force per unit area and $\eta.v./d$ can represent shear stress due to the action of viscosity.

This equation may be combined with the definition of surface shear 4.1, to give:

$$\tau_o = (70415 \times \nu \times d)^{-1/2} \times 0.4425 \times v^2 \quad - - - 4.2 - -$$

Laminar flow occurs at low Reynolds numbers. In general there is a transition to turbulent flow at a critical Reynolds number, which is dependent on the surface roughness and geometry. For example in smooth pipe flow turbulence usually occurs when the Reynolds number exceeds 2000, and for flow over a flat plate the transition occurs when the Reynolds number reaches approximately 5×10^5 .

4:1.2. Turbulent Flow

High levels of surface shear are encountered when turbulent flow is present. The flow is disordered, with small areas of the fluid moving in random directions which only have an average velocity in the direction of flow. As such it is much harder to analyse this type of flow, but generally it is considered that there is larger momentum transfer in the fluid causing a large viscous shear force. (This also causes higher thermal transport between the fluid and the surface.) Turbulent flow consists of three zones, though they are not sharply defined and merge into one another.

A very thin layer next to a boundary is called the viscous sub-layer. This is a region of relatively smooth flow which gives a high shear at the surface. There follows a region where the velocity profile obeys the 'law of the wall'. In this region the flow is turbulent, with a high degree of mixing. The velocity profile increases in a logarithmic manner and then forms a fairly flat profile, causing a wide boundary layer - see figure 51. At greater distance from the wall the logarithmic profile breaks down and a third region exists, called the velocity-defect law region.

A relationship has been arrived at between the skin friction coefficient and Reynolds number for flow over a smooth, flat plate:

$$C_f = 0.059 \times Re^{-1/5}$$

and, combining this with 4.1 and using the density and viscosity values for air gives:

$$\tau_o = (70415 \times \nu \times d)^{-1/5} \times 0.03806 \times v^2 \quad - - - 4.3 - -$$

4.1.3. Measurement of Velocity

Flow velocity may be measured by means of the Pitot/static tube (see figure 52). This is a device for measuring the stagnation and static pressures, which may be measured differentially and the fluid velocity deduced from the result using the Bernoulli equation:

$$\frac{P_1}{\rho} + \frac{u_1^2}{2} + gh_1 = \frac{P_2}{\rho} + \frac{u_2^2}{2} + gh_2$$

This states that the total head (due to pressure, velocity and gravity) is constant along a streamline.

If the streamline is horizontal then $h_1 = h_2$, and if $u_1 = 0$ (i.e. at stagnation of the flow) then

$$P_1 - P_2 = 1/2 \cdot \rho \cdot u_2^2$$

$$\text{So } u_2 = \sqrt{\frac{2 \cdot \Delta P}{\rho}}$$

where ΔP is the pressure difference between the stagnation and static values. The Bernoulli equation only applies accurately for incompressible flow, but this equation may be used to achieve good velocity estimates from pressure measurements in many circumstances.

Hot wire anemometry is also commonly used to determine the velocity profile near a surface, where the current required to maintain the temperature of a wire in a gas flow is measured and related to the mass flow rate of the gas.

Once the velocity distribution near a surface is known, the surface shear stress may be calculated from theoretical relationships between the velocity profile and the shear stress. The wind tunnel used was too small to allow easy access by external devices, so a more direct method was utilised.

FIGURE 52. Pitot/Static tube.

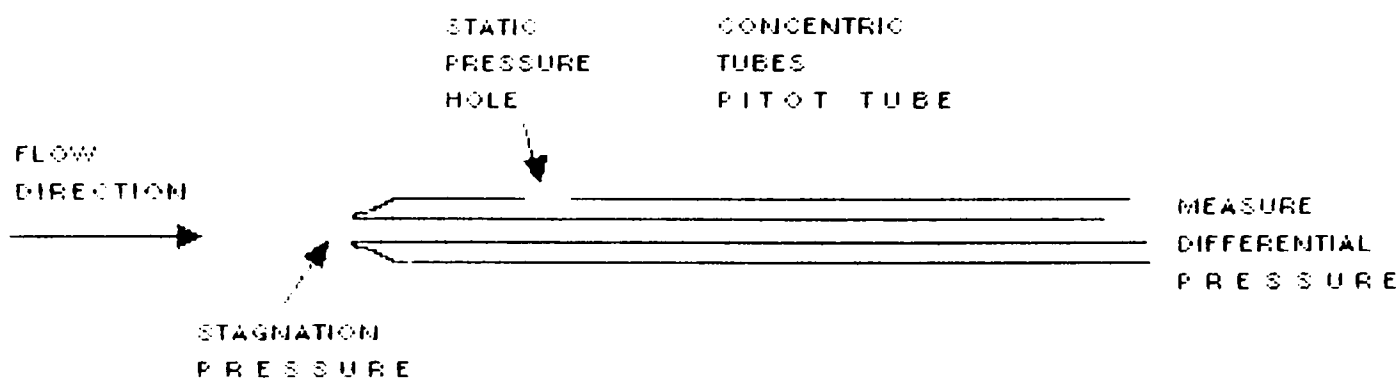


FIGURE 53. Static pressure tube.

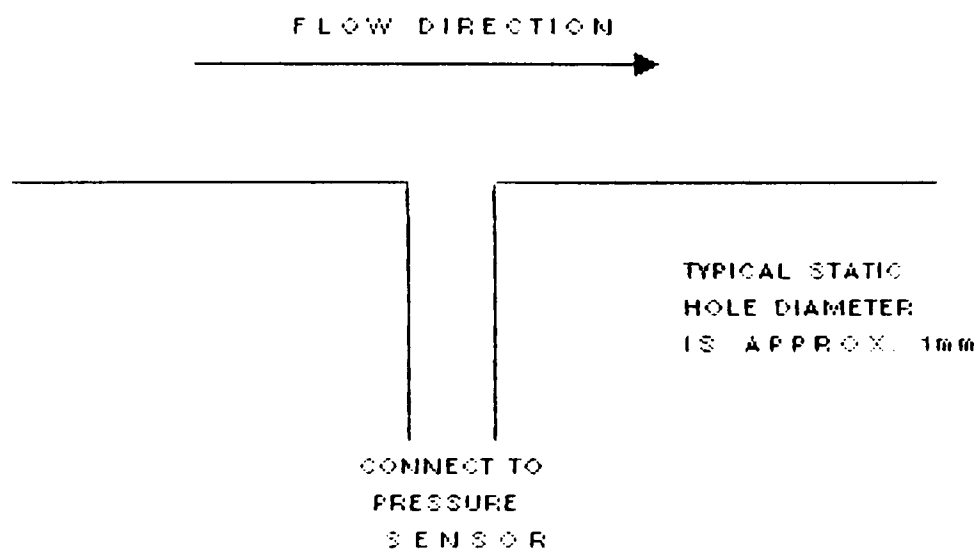
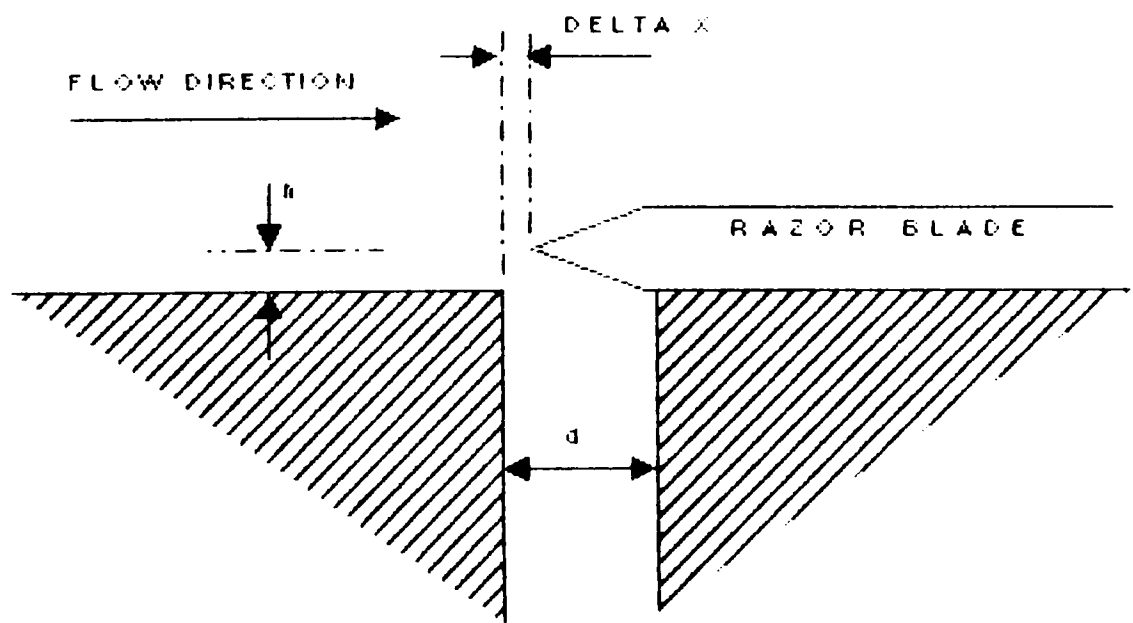


FIGURE 54. Preston tube.



4:1.4. Measurement of Surface Shear

The surface shear stress may be calculated from a knowledge of the flow velocity and a characteristic length, d . Figure 51.b. shows a typical variation of the shear stress for laminar and turbulent flow over a flat plate at one flow velocity. If the flow velocity were increased the level of shear would rise and the transition to turbulent flow would occur closer to the leading edge.

Surface shear stress measurement methods have been reviewed in many papers, e.g. ref. 59,60. There are many different methods which are more direct than inferring stress from velocity profiles. One of the most direct measurements is to measure the force on a surface element. A section of the surface, which must be perfectly flush, is connected in some way to a balance or transducer to measure the displacement of the surface element. This method can be very troublesome as the balance needs to be large enough to give a large signal, but small enough to give a meaningful local measurement. The element must have gaps around the edges in order to move, and these can disturb the flow or result in pressure leaks. An alternative is to use a small 'fence' submerged in the sublayer next to the wall. Measurements of static pressure directly in front of and behind the fence can then be used to infer the surface shear stress.

Other methods are based on the analogy between shear stress and heat or mass transfer. In the case of heat transfer a small resistive gauge may be evaporated or painted onto the surface. This is included in a Wheatstone bridge. The current required to keep the gauge temperature constant in flow conditions may then be related to the surface shear stress. These devices have a fast response time and do not interfere with the flow, but heat may be lost to the substrate as well as to the flowing fluid, so some allowance must be made for this. Electrochemical techniques are used

for the mass transfer analogy. An electrode is evaporated onto the surface. This is covered with suitable chemicals and the voltage to the electrode is maintained so that the rate of reaction is determined by the mass flow rate over the chemicals. The chemicals must be compatible with the model and fluid used, and the method is only useful in liquid flow. The velocity gradient may be calculated from the current supplied to the electrode. All these methods are fairly laborious, require special equipment and are by no means totally reliable. One of the more reliable methods is the Stanton/Preston tube, though it is still very longwinded and may take several hours to carry out a series of point measurements along a test surface. The Preston tube was the method used to calibrate the wind tunnel surface and duct described in the next section, so this technique is described in more detail below.

4:1.4.1. Preston Tube Measurements

Stanton first used a Pitot tube at the surface to measure shear. He calibrated the technique in laminar flow and in the viscous sub-layer in turbulent flow, and indicated that the shear stress was a function of the stagnation pressure minus the static pressure. The two pressures may be measured at the same static pressure tapping by using a razor blade adhered to the surface such that the blade tip protrudes over the hole. For the calibration of Stanton to hold the blade tip must be inside the thickness of the boundary layer.

The thickness of the boundary layer may be estimated from an equation due to Blasius⁶¹:

$$\delta = 5.d./Re^{1/2} \quad (\text{laminar flow})$$

$$\delta = 0.37.d./Re^{1/5} \quad (\text{turbulent flow})$$

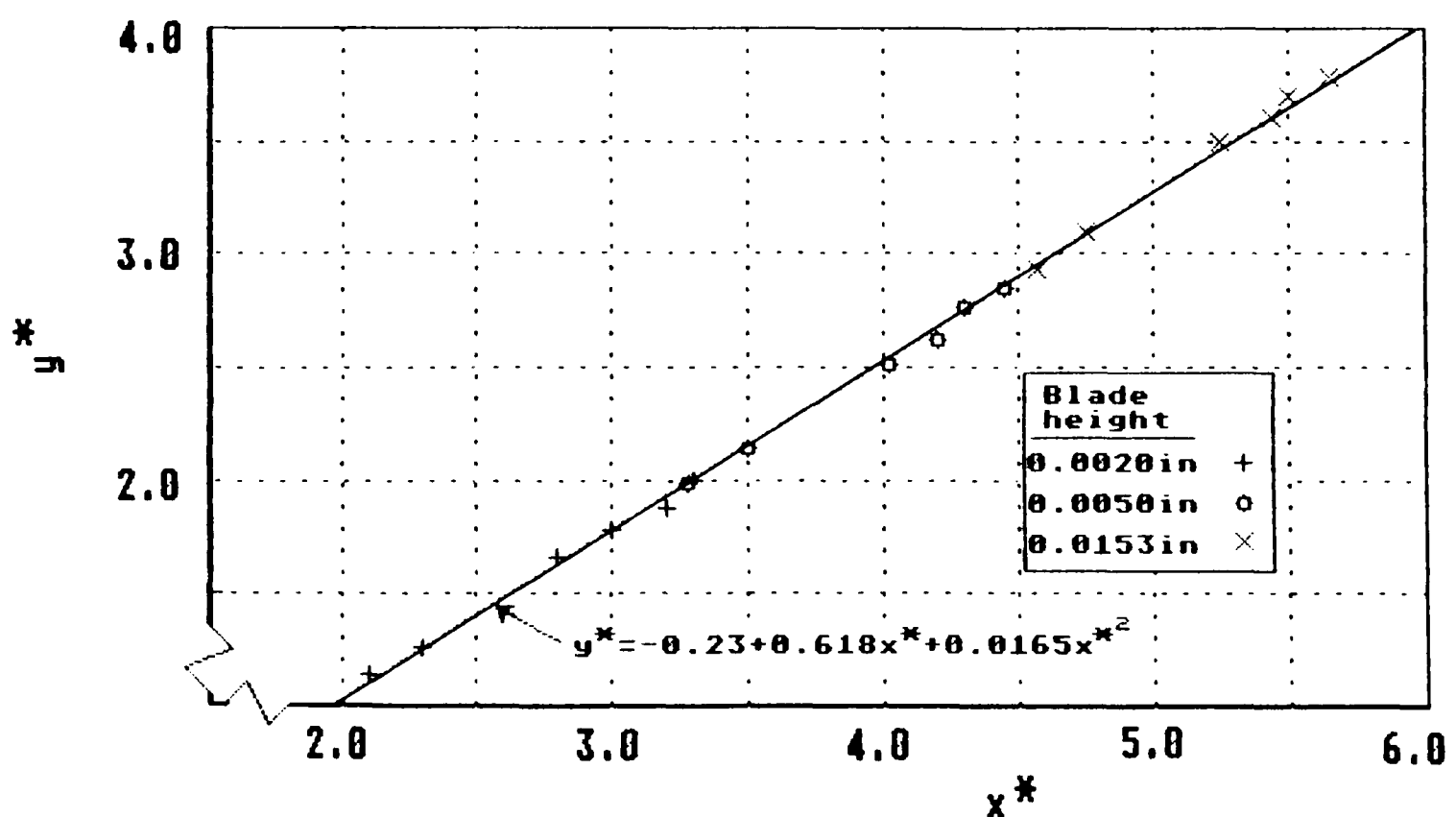
In the experiments described in the next sections this thickness was

of the order of a millimeter, so the tip of the blade was inside the boundary layer, as the height of the blade tip was only about 80 μ m.

Preston⁶² applied this idea to turbulent flow over smooth surfaces. This tube need not be submerged in the viscous sub-layer, but can enter the buffer layer into the turbulent flow. Prestons calibration curves indicated that this method, based on the law of the wall, was reasonable. Improved calibrations have been proposed by Patel⁶³ and East⁶⁴. The calibration due to East is the simplest and most widely applicable and so was used to calibrate the wind tunnel used in this thesis.

The method consists of making two measurements at each test point. The first measurement is to measure the static pressure at the surface with a static pressure tapping, see figure 53. A razor blade is then glued to the surface such that the taper of the blade protrudes over the hole, see figure 54. The pressure is measured again at the hole. The height of the tip of the blade was measured. The pressure difference is then expressed in non dimensional form and a calibration curve used to find τ_o .

FIGURE 55. Calibration of razor blade Preston tube due to East.



The calibration of East is reproduced in figure 55, which is a plot of

$$y^* = \log\left(\frac{\tau_o \cdot h^2 \cdot \rho}{\mu^2}\right) \quad \text{against} \quad x^* = \log\left(\frac{\Delta P \cdot h^2 \cdot \rho}{\mu^2}\right)$$

The surface shear stress, τ_o , can be found once the blade height, h , has been measured. This is a point measurement and assumes a prior knowledge of the flow direction. The geometry of the arrangement can have some effect on the results obtained. The blade can also have an effect on the flow so only one measurement may be made at a time. Hence measurements over a whole surface take some time to complete as the adhesive must be cleaned from the surface after each measurement at each location. To summarise the results of East for two dimensional flows:

- (i) the blade width should be at least 30 times greater than the blade height and the static hole diameter should be at least 3 times greater than the blade height.
- (ii) the greatest source of error arises from the tip of the blade not being at the edge of the static hole, i.e. Δx not being zero (see figure 54 .). The error in ΔP does not exceed 5% as long as $(\Delta x/h) < 0.5$.

4:2. THE USE OF LIQUID CRYSTALS IN SHEAR STRESS MEASUREMENT

Previous studies have been carried out on the effect of shear stress on liquid crystals, and although these have demonstrated some interesting optical effects they were in insufficient depth to enable an evaluation of their application in practical shear stress measurement^{12,65→71}.

The non-Newtonian flow characteristics of cholesteric liquid crystals makes interpretation of their response to shear complex. In the study to be described the shearing force has been applied perpendicular to the helical axis, resulting in a velocity gradient parallel to the helix, which is in effect similar to the previous studies mentioned above. Under these conditions the reflection spectrum is, in general, observed to shift towards the blue on shearing, and the reflected intensity drops with a broadening of the bandwidth. These effects could be consistent with a tilt distortion of the helix, but the previous work did not examine the effects of varying viewing or illumination angles, which would provide evidence for this model.

Workers at NASA have attempted to measure surface shear stress using liquid crystals by two methods. The first of these, which uses encapsulated liquid crystals, monitors surface temperature changes as a function of air flow rate at a point on the surface which is heated with a laser beam⁷². The analysis of the temperature data assumes a correlation between heat transfer and skin friction, and is analagous to the hot film probe method mentioned in section 4:1. Although this is a sound assumption and could be a reliable technique, it doesn't enable simultaneous whole surface assessment of shear. The second technique involves coating the surface with an evenly applied, thin layer of neat liquid crystal and observing the shear induced colour changes produced by the airflow^{58,73,74,75}. This method has been demonstrated in flight tests in which portions of the test aircraft have been coated with liquid crystal material and observed from a second aircraft. The primary objective was to determine the location of the laminar to

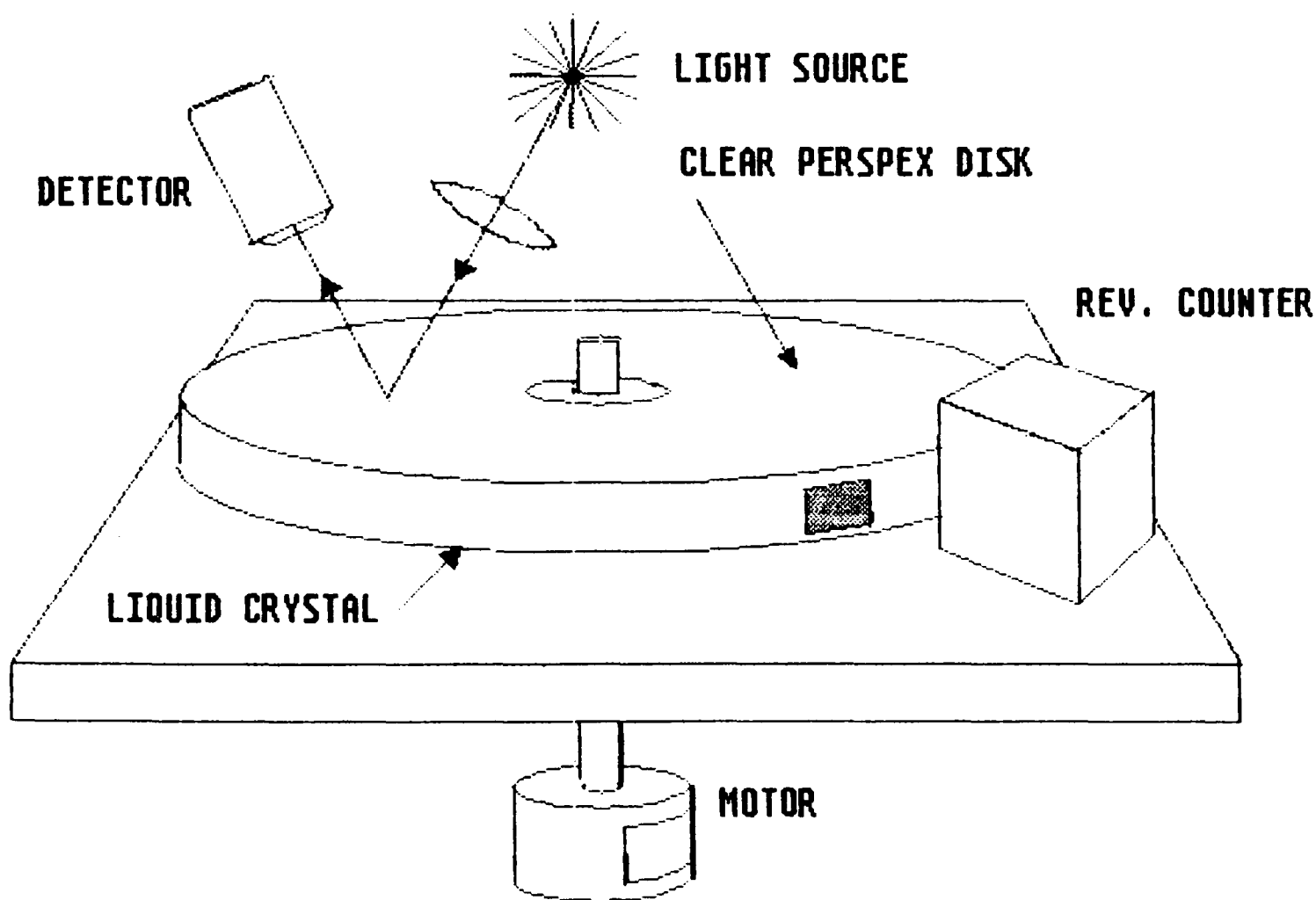
turbulent boundary layer transition, and good correlation with alternative methods has been claimed. More recent work⁷⁶ has emphasised the importance of temperature and lighting effects, which were ignored in the initial experiments.

In order to investigate these effects more thoroughly a program of work was initiated at Oxford under well controlled laboratory conditions, using rotating disks and wind tunnels.

4:2.1. Rotating Disk Experiment

The apparatus was constructed as shown in figure 56 and consisted of a blackened perspex base, with a motor driven clear perspex disk, 100mm in diameter and 5mm thick, resting on top of it. The angular velocity of the disk, which could reach a maximum of 7 rads. s^{-1} , was measured using a

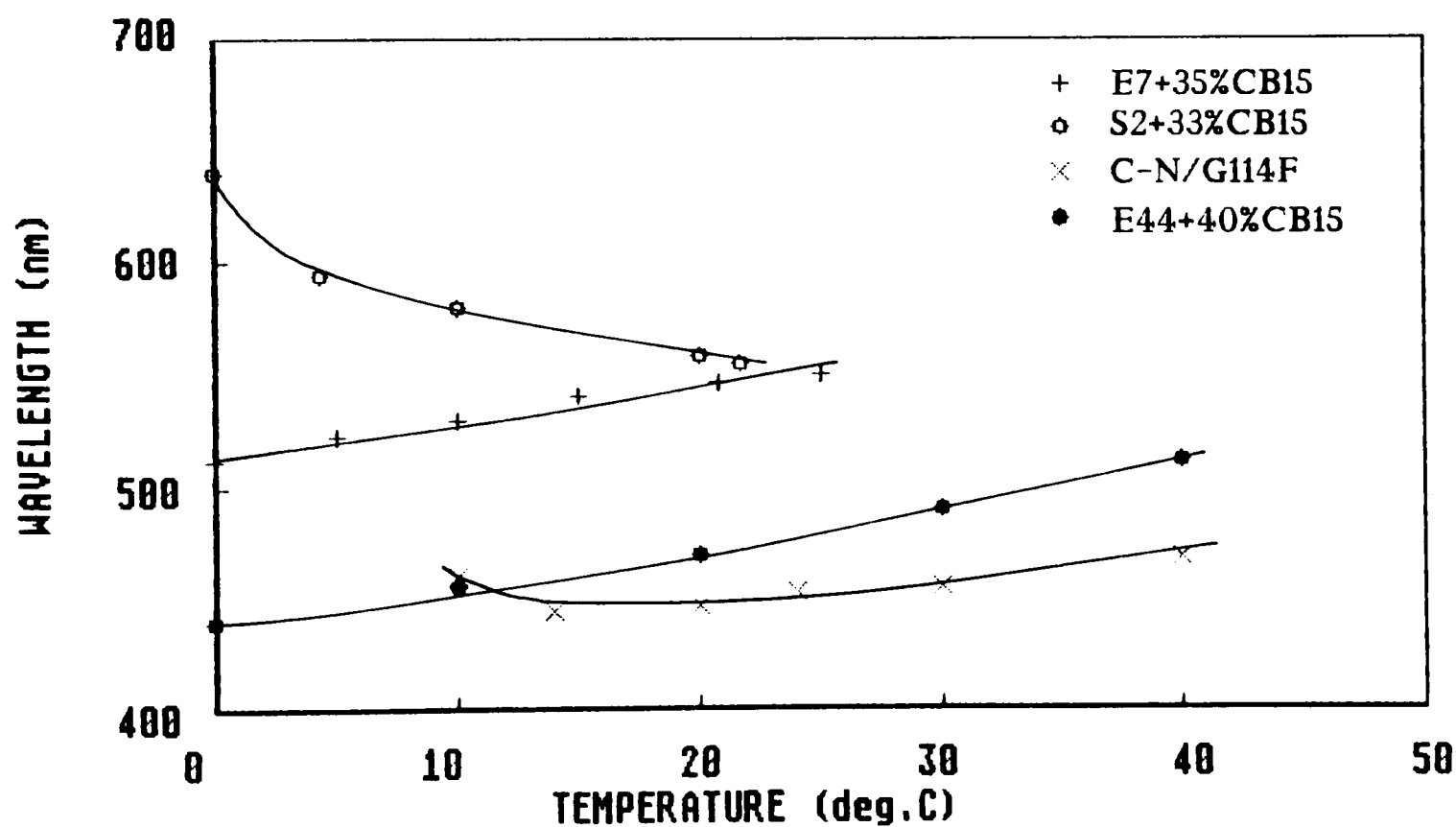
FIGURE 56. Rotating disk apparatus used to examine the effect of shear on liquid crystals.



photodiode arrangement on the edge of the disk. This enabled the shear rate to be calculated at a particular radius on the disk, using a liquid crystal thickness between the plates of 50 μ m.

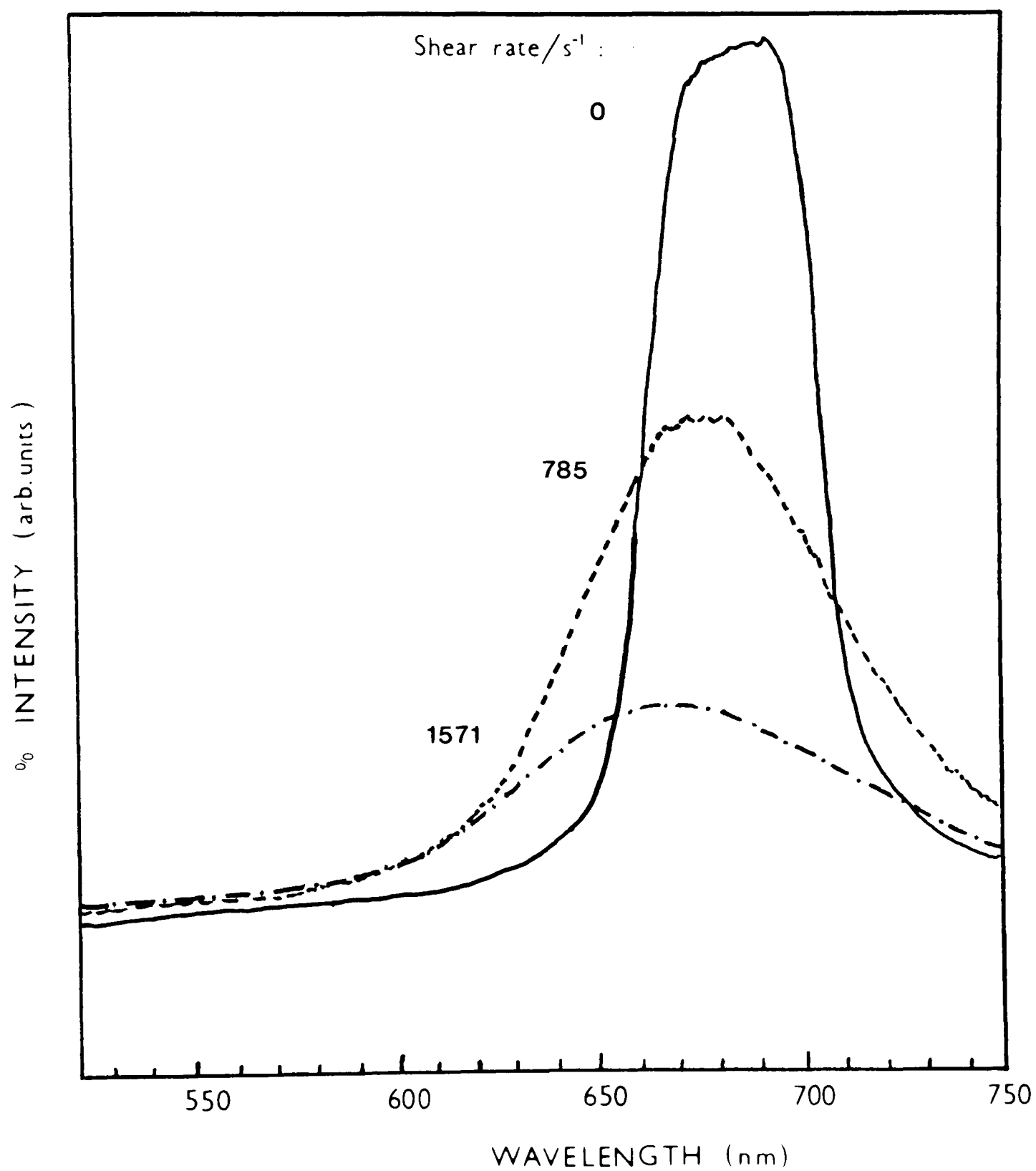
Temperature insensitive liquid crystals were used, namely S2+ 33% CB15, E44+40% CB15, E7+35% CB15, E9+35% CB15 and C-N/G114 F. The 'E' mixtures and S2 are eutectic mixtures based on the 4-n-alkyl and 4-n-alkyloxy 4'-cyanobiphenyls. CB15 is an acronym for S (+) 4 (2''-methylbutyl) 4'-cyanobiphenyl. Figure 57 shows the colour/temperature calibrations for these materials. These materials were supplied by BDH Ltd, except for C-N/G114 F. which is a cholesteryl ester and was supplied by Hallcrest.^{C3}

FIGURE 57. Wavelength verses temperature characteristics of the mixtures used to study shear stress on liquid crystals.



Experiments were conducted as a function of viewing and illumination angles. Diffuse illumination was also used, provided by an integrating hemisphere. Spectral measurements were made using a Rofin-Sinar RSO 6000 spectrum analyser and RSO 0200 control unit interfaced to a BBC 32K microcomputer, referenced to the Kodak white standard 6091.

FIGURE 58. Effect of shear on the reflection spectrum of S2+33%CB15. when sheared between rotating disks.



Results

In general the results for all four materials were similar, producing a blue shift and reduction in the reflected light intensity at normal viewing angles. A typical change in the reflection spectra is shown in figure 58 for S2+33%CB15, which shows all the previously reported trends. Spectral behaviour is shown in figures 59, 60 and 61 which show peak reflected wavelength, selective reflection bandwidth and reflected intensity as a function of shear rate, respectively. With reference to figure 61, the magnitude of the decrease in reflected intensity produced by shearing was found to be very dependant on whether the illumination was produced by a diffuse or point source. Diffuse illumination produced a smaller difference in the two intensities which is consistent with a change from a specular to a more diffuse reflection from a sheared to an unsheared sample. This also explains the increase in the bandwidth with shear (figure 60) which is a function of the flow induced breakup of the monodomain ordering.

FIGURE 59. Effect of shear stress on the peak reflected wavelength for three of the mixtures used in the rotating disk experiment.

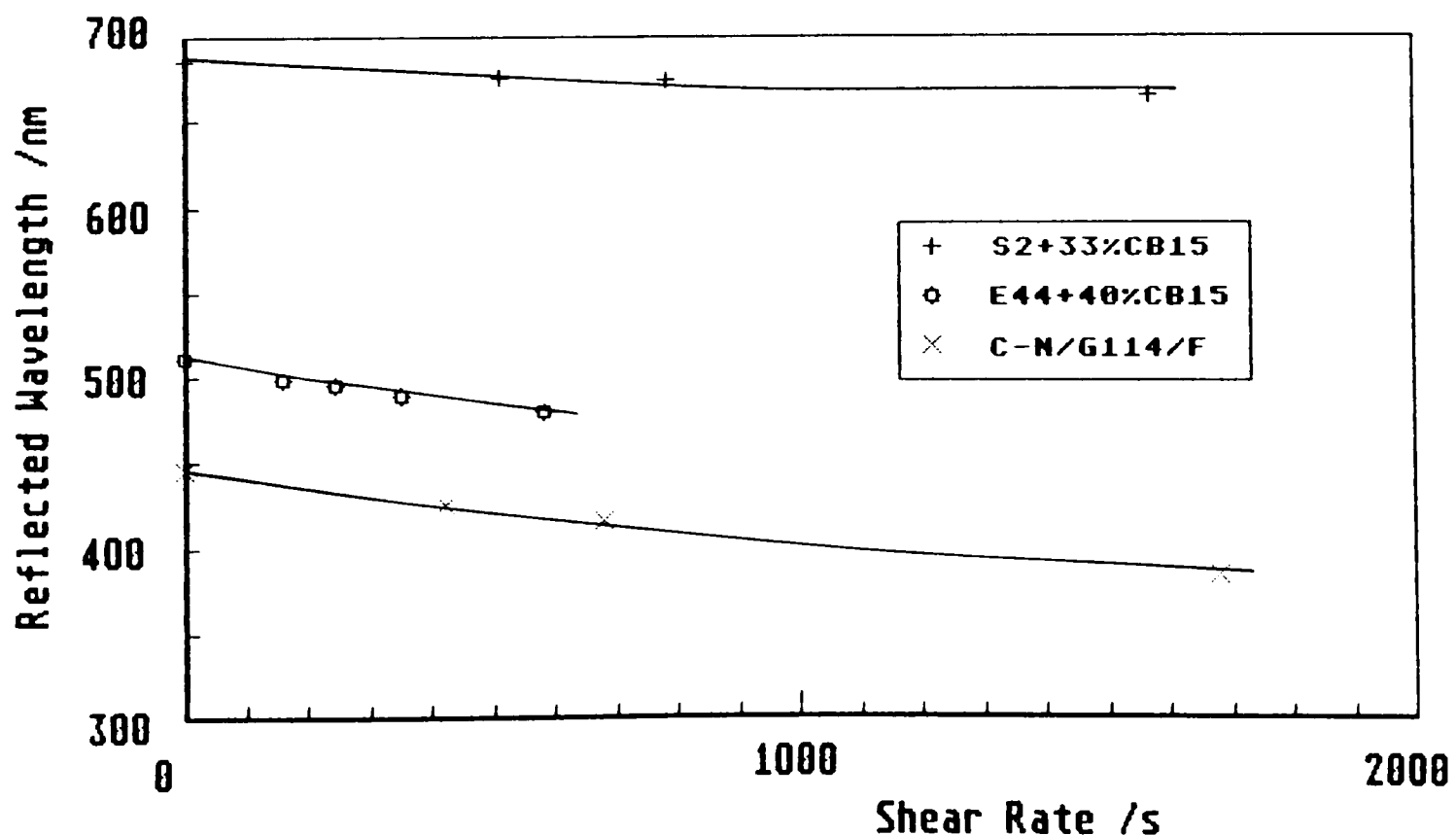


FIGURE 60. Dependence of the reflected bandwidth on shear rate applied to a liquid crystal layer in the rotating disk apparatus.

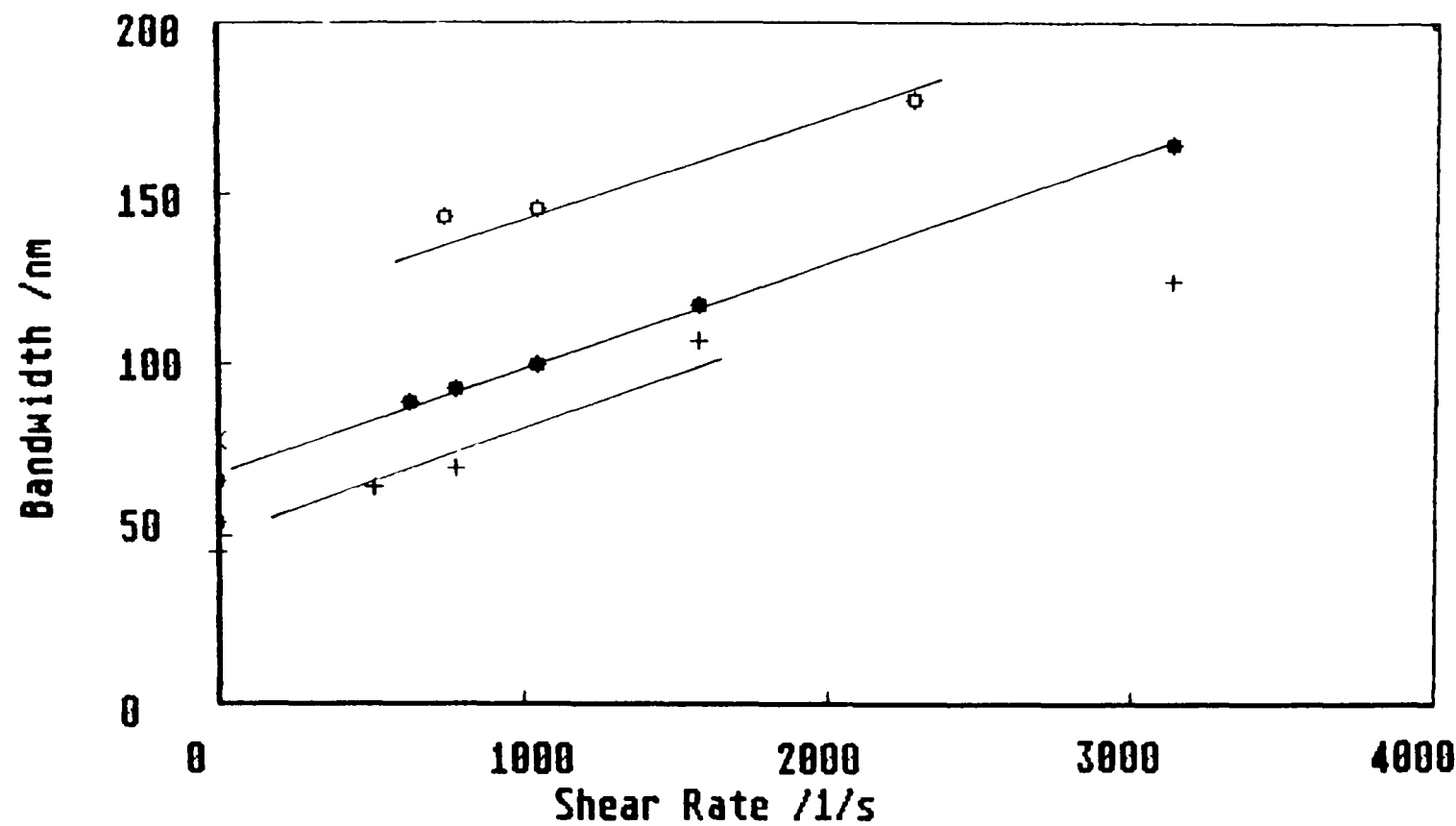
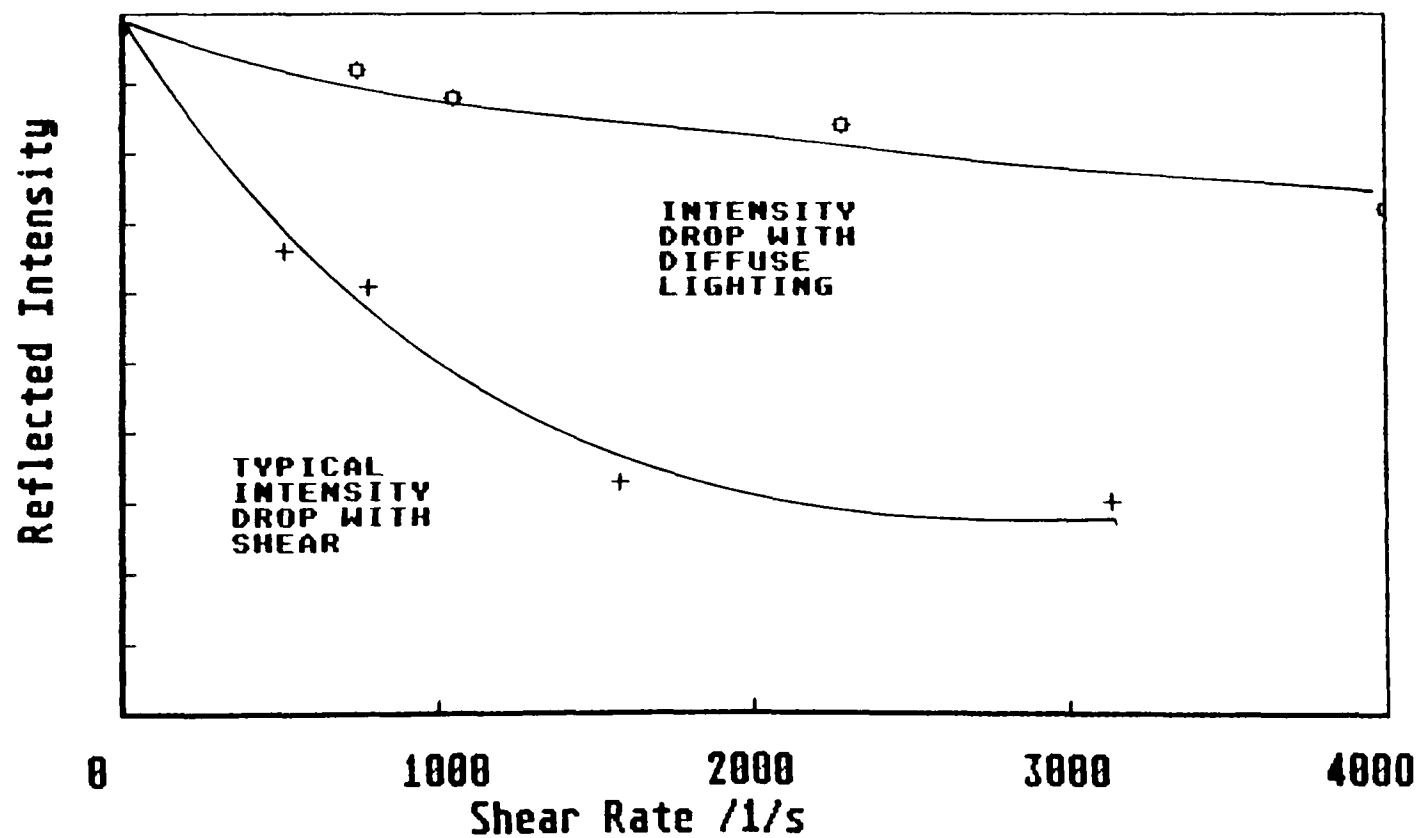


FIGURE 61. Dependence of the reflected intensity on shear rate of a liquid crystal layer in the rotating disk apparatus.



A qualitative interpretation of these effects can be made by envisaging a simple distortion of the helix under the action of a shear stress as shown in figure 62(a). Polar plots of the peak reflected wavelength as a function of viewing angle for sheared and unsheared samples are shown in figure 62(b), and are consistent with this picture. This is apparent to the observer viewing the rotating disk off axis, who sees a red shift in the half of the disk approaching him and a blue shift in the receding half. (see plate 11).

The reflected intensity as a function of viewing angle for diffuse illumination ($\phi_1 = \phi_2$) and point illumination were calculated from equation 2.3, given below:

$$\lambda = n.p.\cos.\frac{1}{2}\left(\sin^{-1}\frac{\sin.\phi_1}{n} + \sin^{-1}\frac{\sin.\phi_2}{n}\right)$$

For the simple case of the helical axis tilting at 45 degrees with respect to the surface plane, the calculated shifts in the peak reflected wavelength would be as shown in the table below.

Viewing Angle:	-60	-30	0	+30	+60
Point source: λ Shift:	↓58	↓73	↓57	↓20	↓5 nm
Diffuse: λ Shift:	↓6	↓81	↓57	↑18	↑78 nm

(based on an initial pitch of 344nm, with $\bar{n}=1.6$)

The arrows indicate a shift up in wavelength(to the red) or down (to the blue). The peak wavelength variations for diffuse and point illumination are shown in figure 63.

Although these figures take no account of bend or stretch distortions to the helix, they indicate that both red and blue shifts are seen only with diffuse illumination, while with perfect point illumination only blue shifts will be seen.

FIGURE 62 (a). Schematic of the effect of a shear stress on the cholesteric helical structure. **(b).** Polar plots comparing experimental results of the angle dependence of the reflected wavelength with and without shear present. Material used : E7+35%CB15, shear rate 1200 s^{-1} .

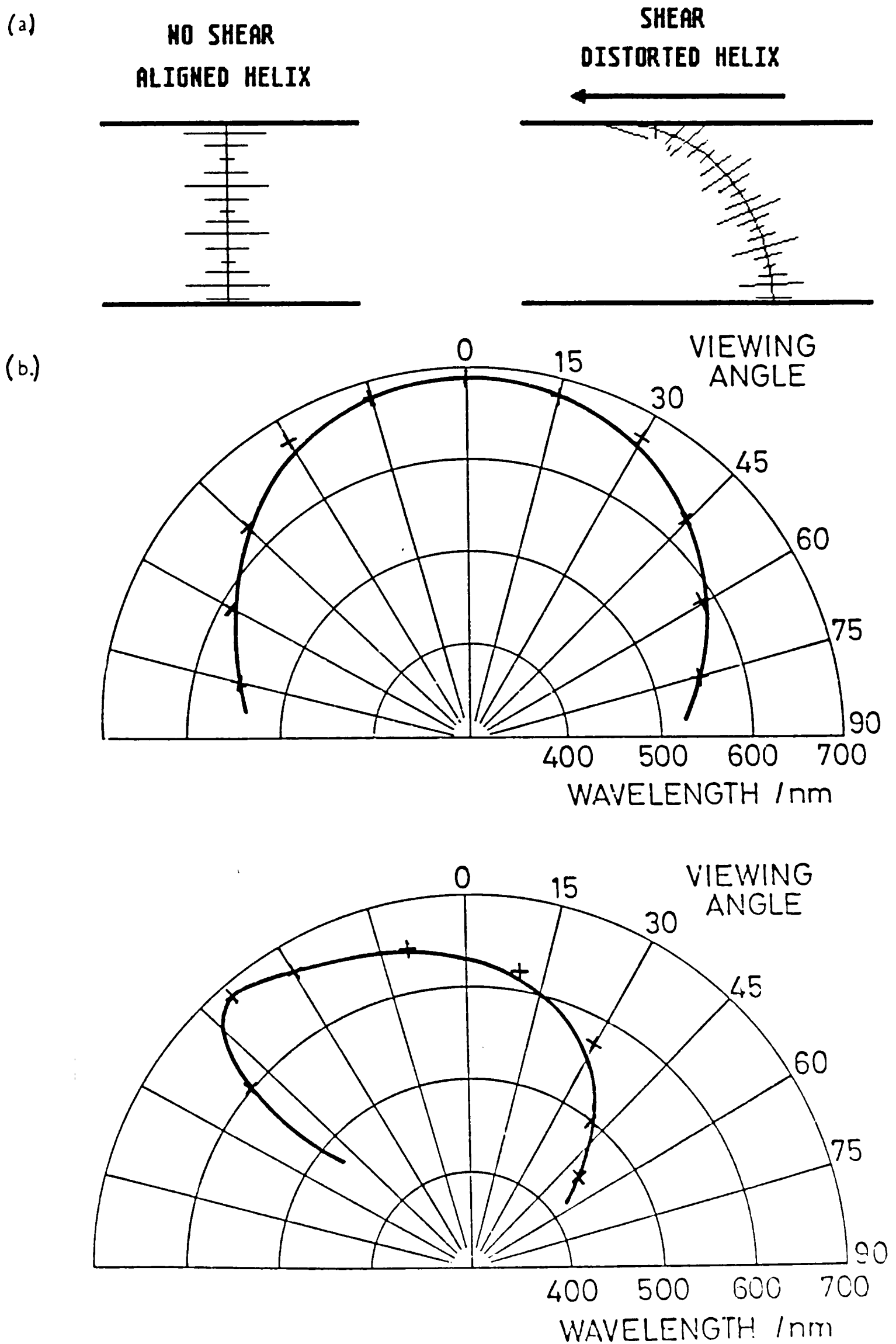
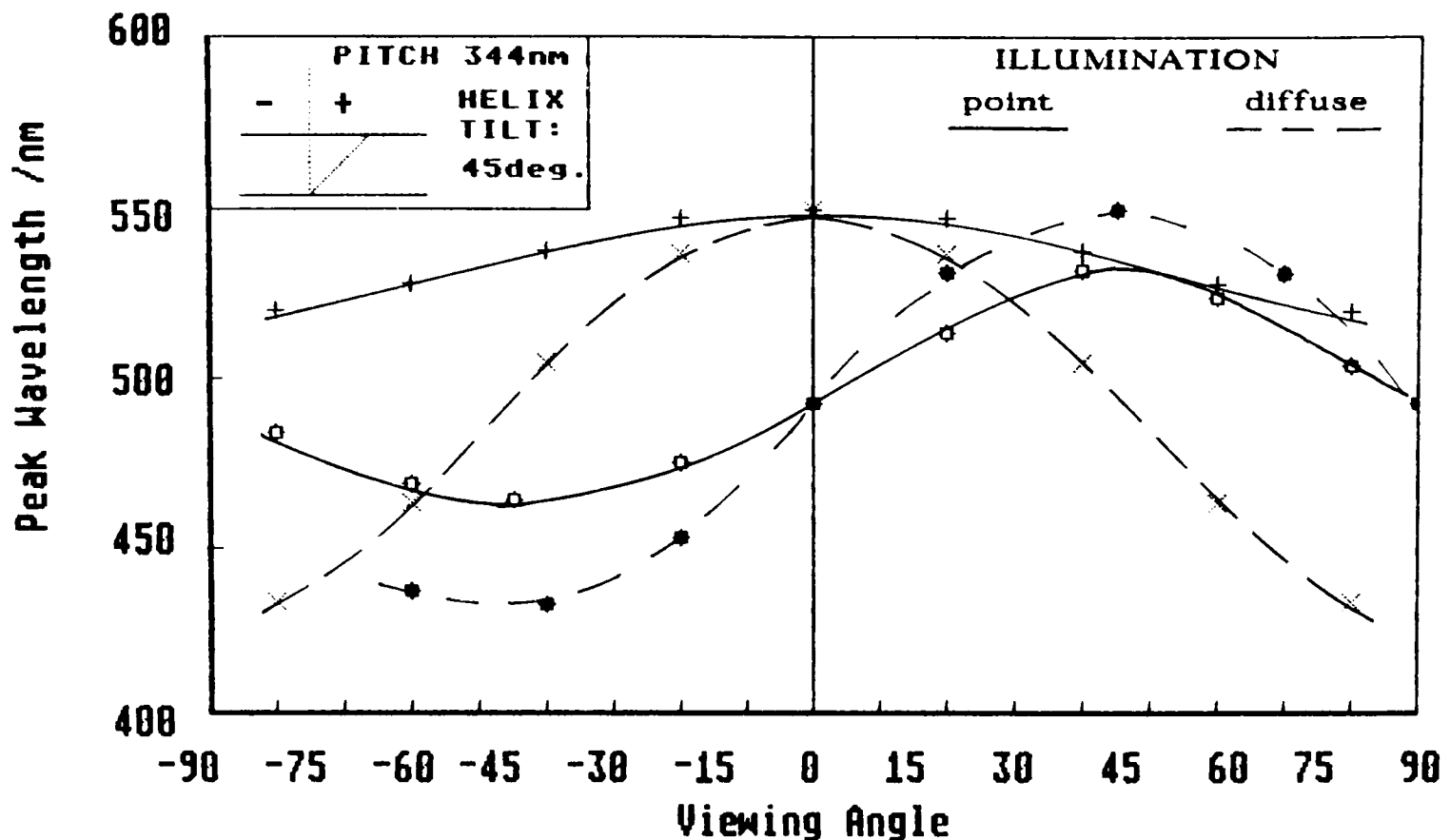


PLATE 11. Cholesteric liquid crystal under shear in the rotating disk apparatus. The varying angle dependence of the reflected colour is apparent as the right hand side of the disk, which is moving away from the viewer is clearly bluer than the left hand side. This may be explained by the distorted (tilted) helix model.



FIGURE 63. Simplified illustration of the effect of viewing angle changes on the reflected wavelength from a sheared cholesteric liquid crystal.

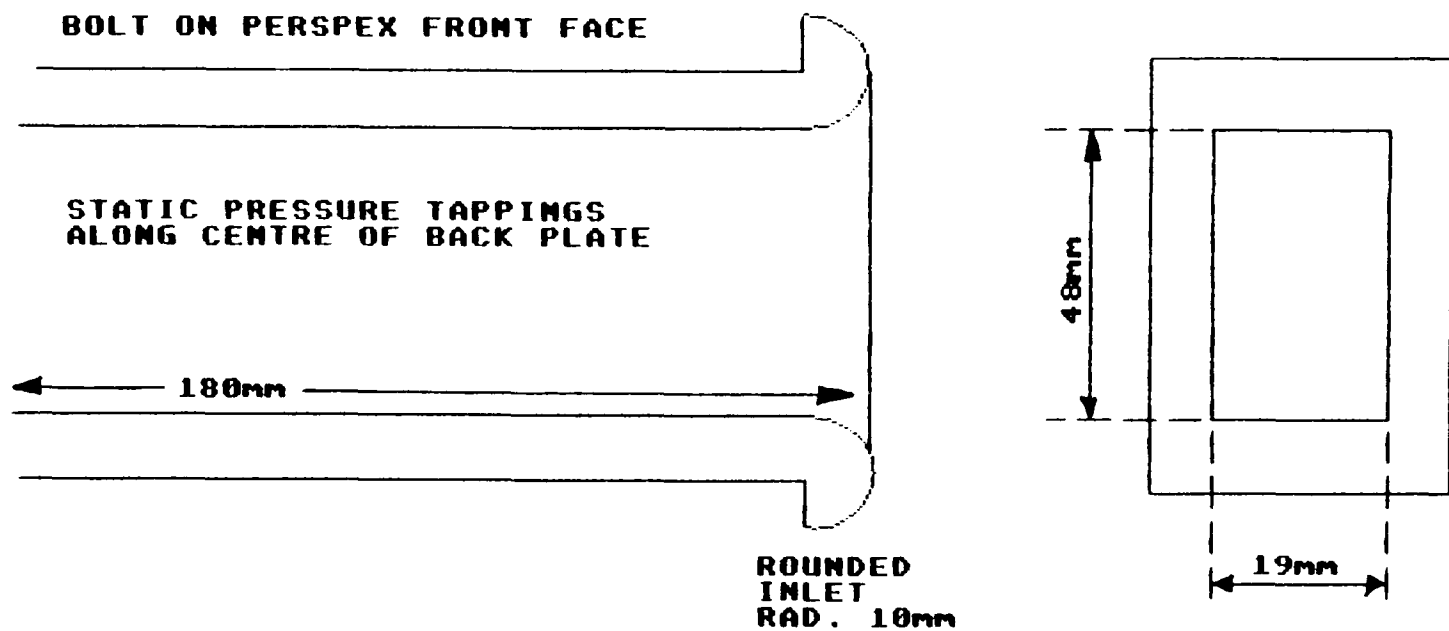


In practice the reflection spectra quickly degrades upon shearing, making accurate assignment of peak reflection colours difficult. In conclusion, although colour changes are observed and act as a general indication of shear stress, an accurate measurement of shear stress level based on these changes cannot be easily made due to degradation of the structure.

4:2.2. Wind Tunnel Experiment

The wind tunnel used to conduct shear stress experiments on the liquid crystals was constructed from perspex and is shown in figure 64. The duct shown in the figure was connected to a vacuum pump via a flow control valve, which allowed room temperature air to be drawn through the duct at controlled flow rates. The liquid crystal was applied either to the back wall of the channel or to a test surface inserted into it (see figure 65). The shear stress at points on the test surfaces was calibrated using Preston tubes.

FIGURE 64. Geometry of the inlet of the blow down wind tunnel used to conduct the shear stress studies on liquid crystals.



The four temperature insensitive materials used in the rotating disk experiment were used again in the wind tunnel. Their reflection spectra were recorded using the Rofin-Sinar spectrum analyser and video recordings were also made of the colour changes.

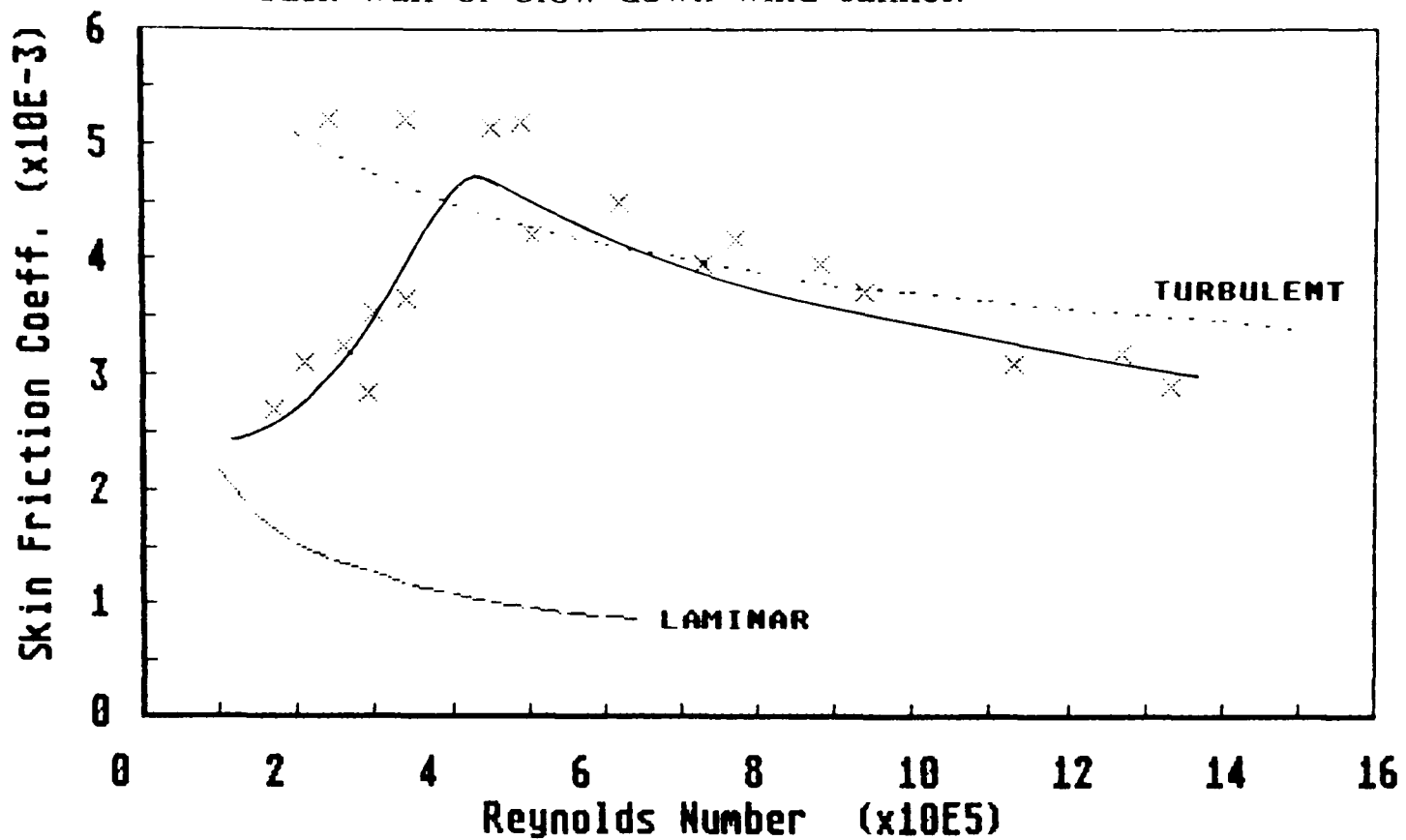
Preston Tube Calibrations

(a).Wind Tunnel Wall.

The channel wall was flat and smooth with a rounded entry section. Using Preston tubes, pressure readings were recorded at regular intervals along the wall as a function of flow rate. Equations 4.1 and 4.2 were used to calculate the laminar and turbulent lines which are shown with the results in figure 66.

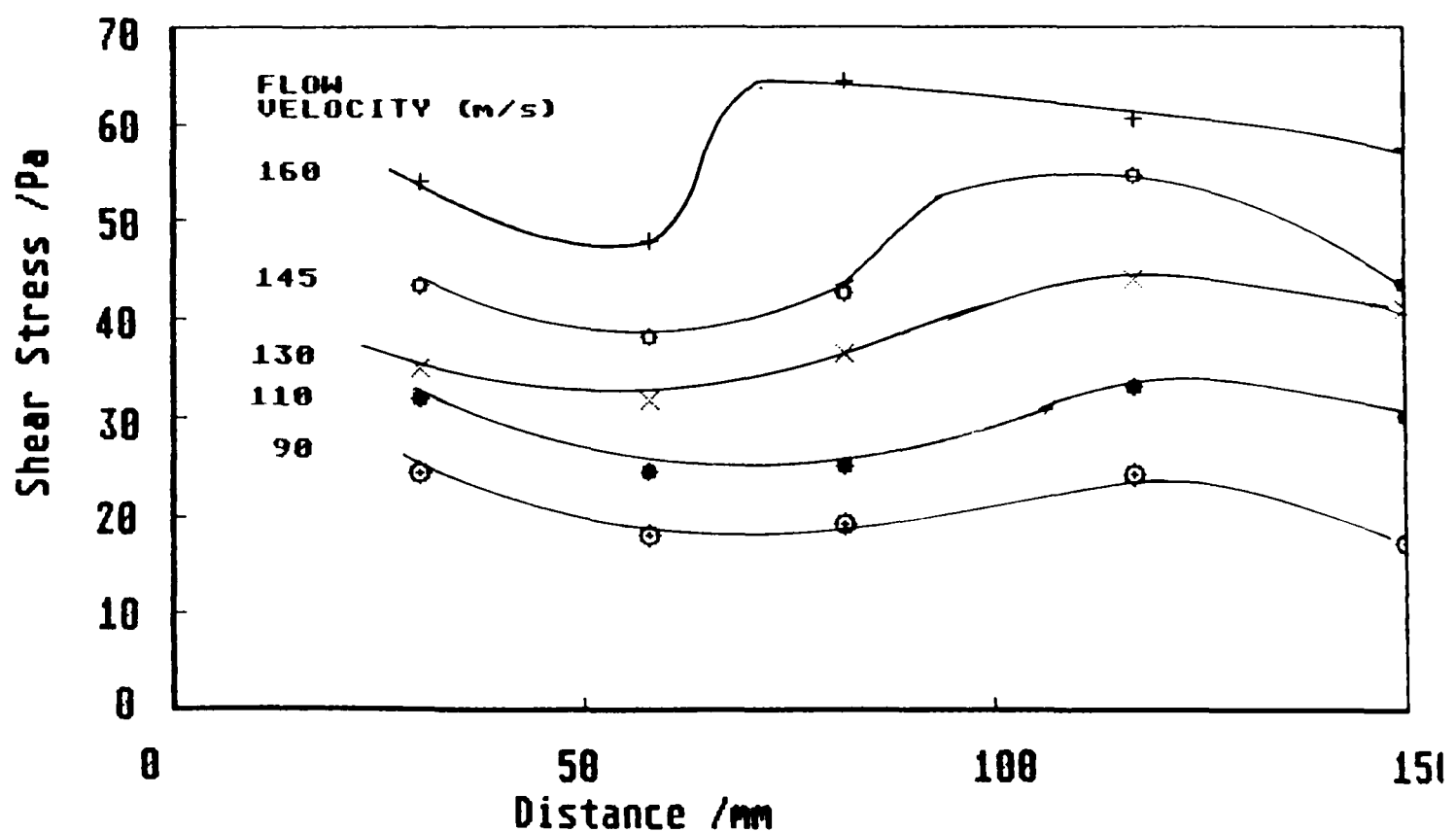
FIGURE 65. Geometry of aluminium blade used in the blow down wind tunnel.

FIGURE 66. Skin friction coefficient verses Reynolds number calibration for back wall of blow down wind tunnel.



The associated shear stress levels shown in figure 67 clearly show the laminar to turbulent boundary layer transition progressing towards the front of the channel with increasing flow rate, though the transition is not very sharp.

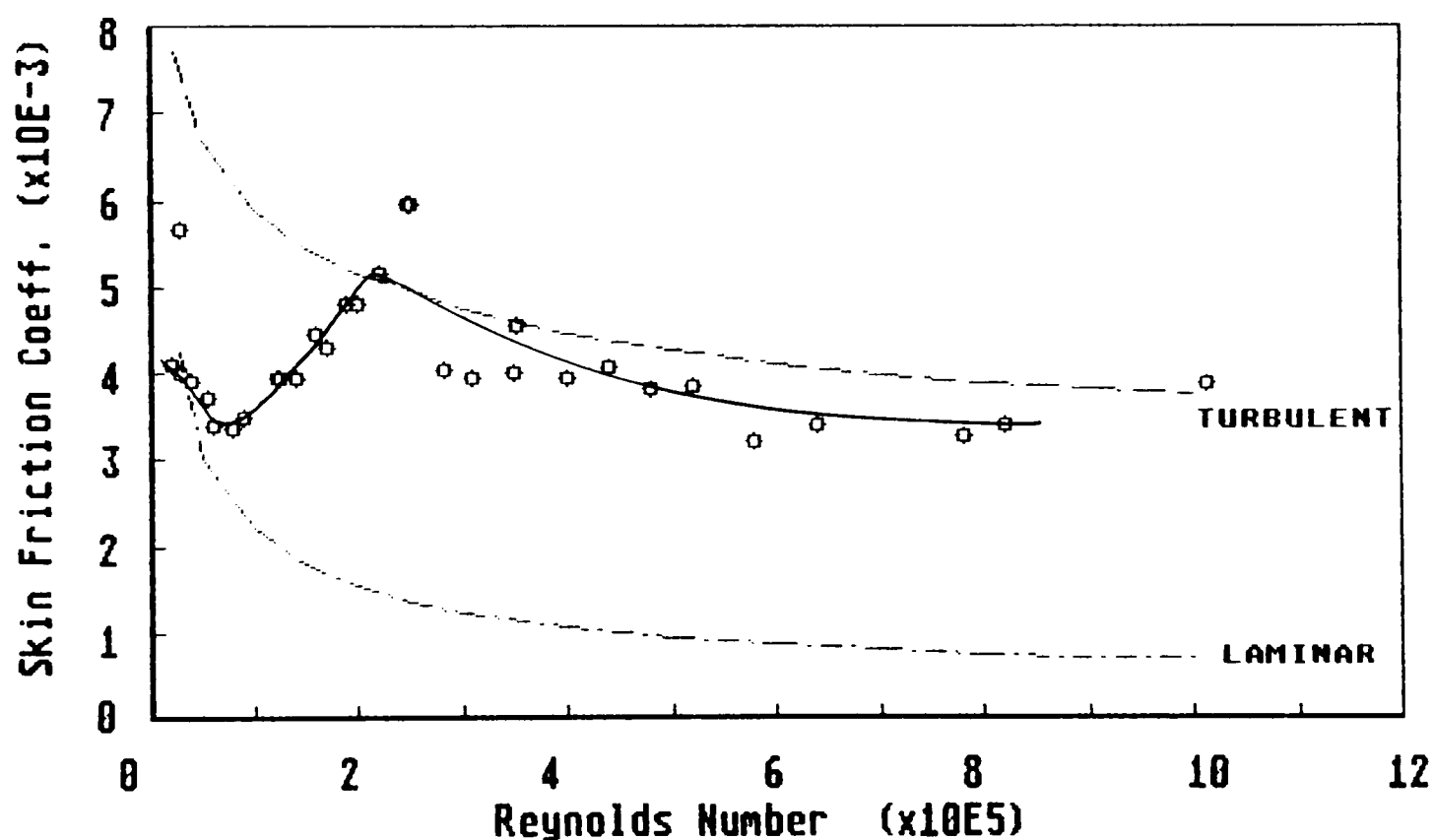
FIGURE 67. Shear stress level against distance along the back wall of the wind tunnel, at different flow rates.



(b). Blade Calibration.

An aluminium blade was used in an attempt to achieve a sharper transition and remove edge effects. The blade was secured in the centre of the wind tunnel and the shear stress on its surface calibrated with Preston tubes. The pressure tappings were attached to one side of the blade and fed out of the tunnel behind the blade. The number of tappings had to be limited in order to minimise the restriction to air flow in the channel. Care was taken in the positioning of the blade to avoid separation of the airflow at the tip. The shear stress calibration shown in figure 68 was obtained, which still did not exhibit very sharp transition, but gave a good indication of the shear stress level along the blade.

FIGURE 68. Skin friction coefficient verses Reynolds number calibration for the aluminium blade used in the wind tunnel.



Liquid Crystal Measurements

The liquid crystals were applied to the blackened test surfaces as thin (20-50 μ m) uniform layers with good initial alignment.

Application of a shear stress to the layer resulted in the same effects as were observed in the rotating disk experiment, namely a shift of the peak reflected wavelength to the blue, a decrease in reflected intensity and a widening of the reflection spectrum, though in this case there was the added complication that the material tended to flow away over the surface at high shear levels. Figure 69 shows a typical result for E44+40%CB15, viewed close to normal incidence. Illumination was provided by a bright tungsten lamp, but was not perfectly diffuse.

The colour changes were again viewing angle dependant, with red wavelength shifts being observed when looking into the flow and blue shifts when looking in the flow direction. In these tests the flow rate was ramped up from zero, but the reflection spectra degraded with increasing shear, and above 60Nm^{-2} accurate measurement became impossible. (see figure 70).

In the above tests the position of the boundary layer transition could not be accurately observed. If the flow rate was gradually increased a gradual colour change was observed, but the change was too diffuse to be useful. Therefore the test surface was exposed to a constant low level of flow and a probe was inserted to locally trip the flow from laminar to turbulent. This gave a clearly defined wedge, see plate 12. Moreover the trip probe was also used with the liquid crystal to differentiate regions of differing shear during flow. During laminar flow the probe would trip the flow to turbulent and produce a bluer wedge than the surroundings, while during turbulent flow the probe produced a separated wake, giving a red shift inside the wedge.

A final test was carried out in which a large increase in shear stress was suddenly applied to the liquid crystal layer in the wind tunnel. The results, shown in figure 71, show an initial large change in the peak reflection wavelength from A to B. If the flow rate is then held constant at this level the reflection colour degrades to C. Subsequent ramping of the flow rate

FIGURE 69. Effect of shear stress on the reflection spectrum of a cholesteric liquid crystal (E44+40%CB15). Result from wind tunnel.

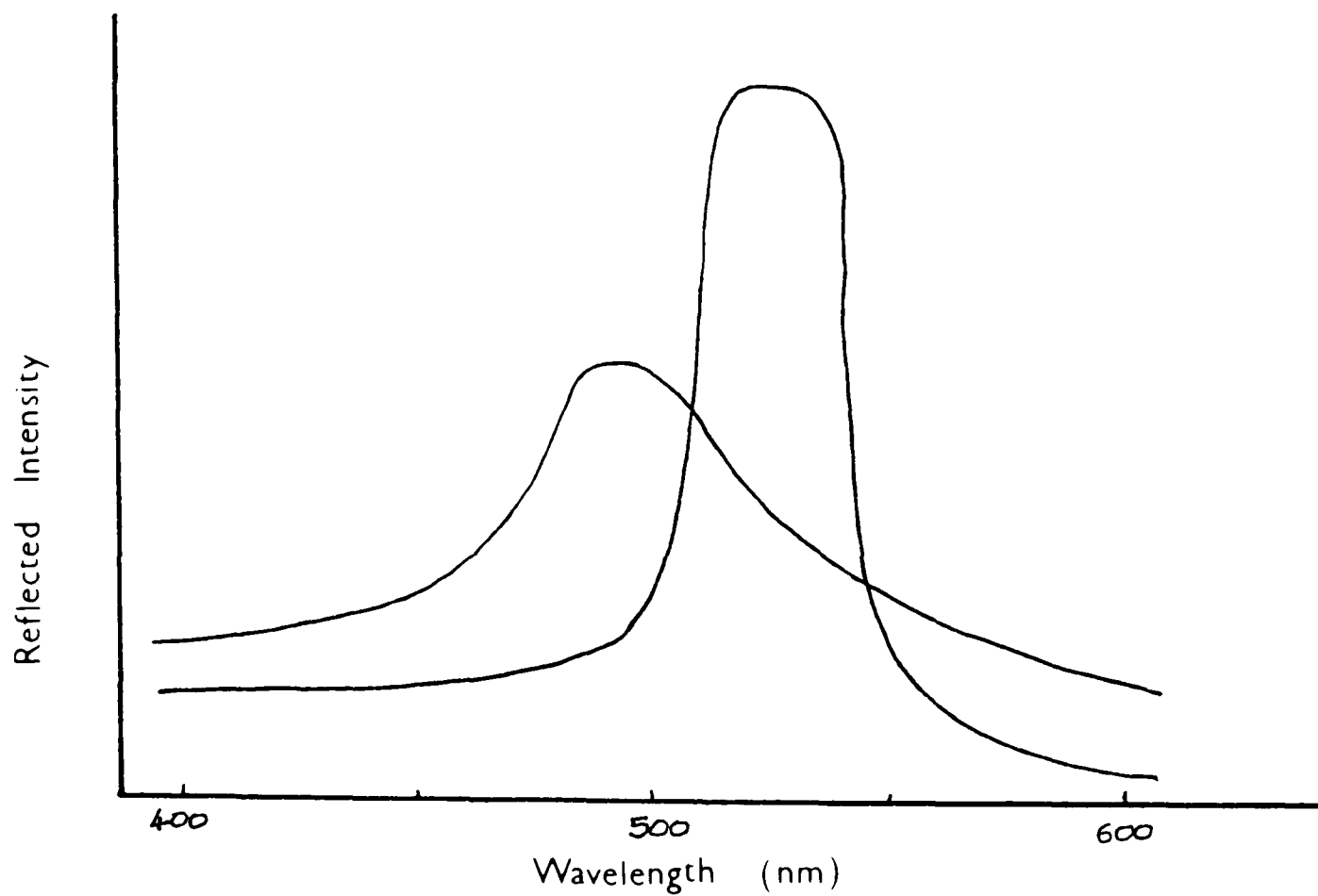


FIGURE 70. Wind tunnel results for the effect of shear stress on the peak reflected wavelength, at different viewing angles. Mixture E44+40%CB15, (a) looking against the flow direction, and (b) looking along the flow direction.

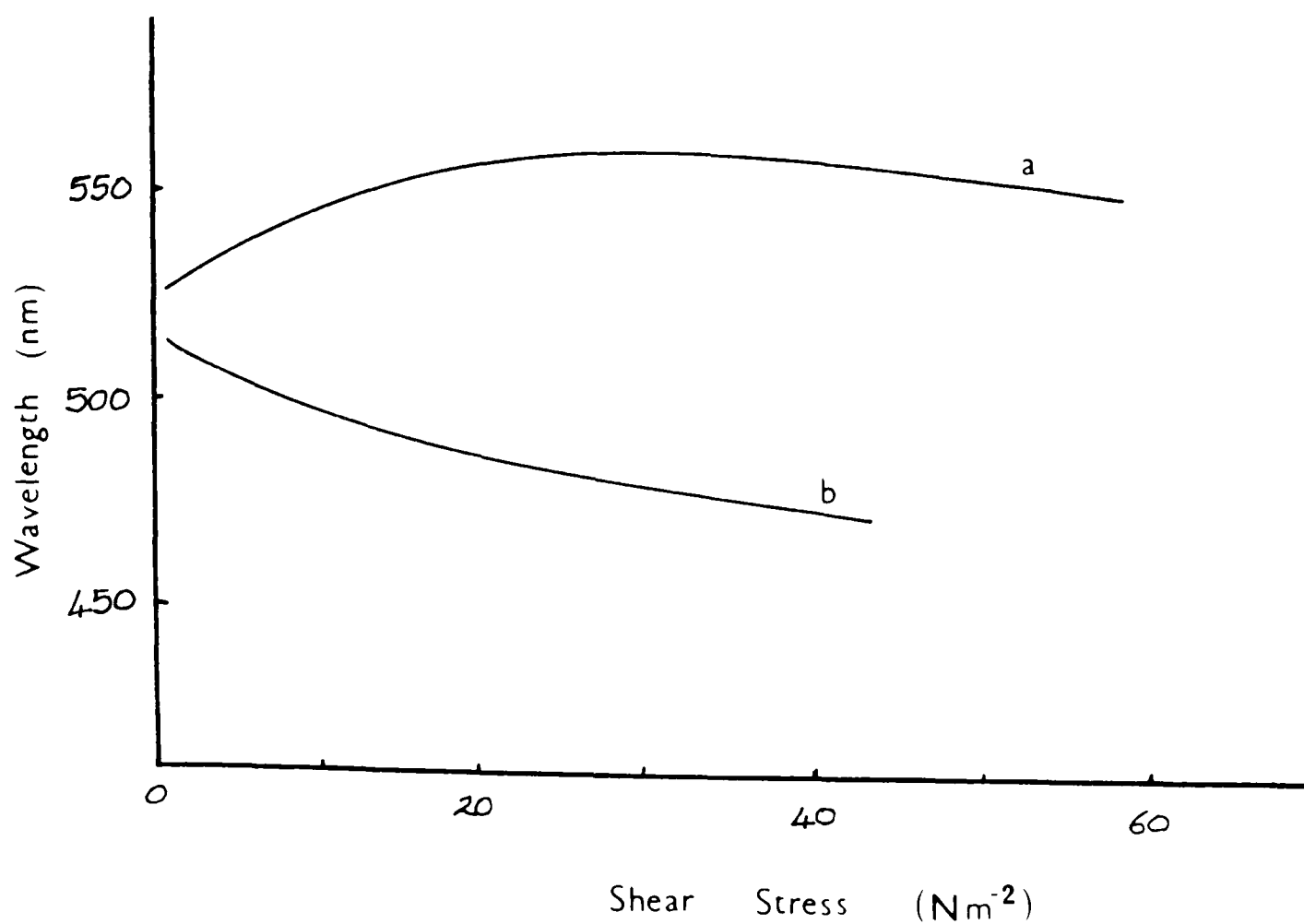
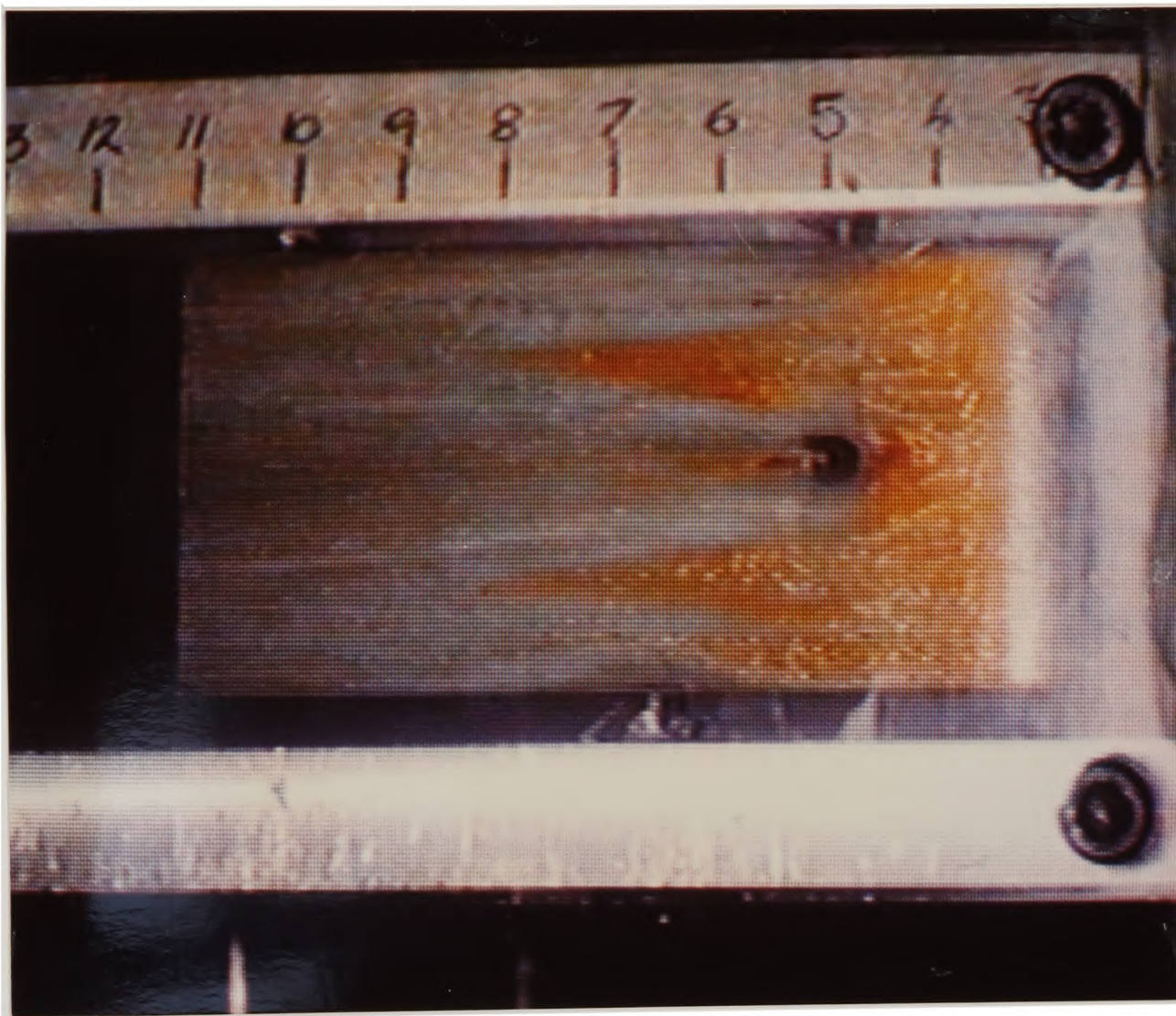
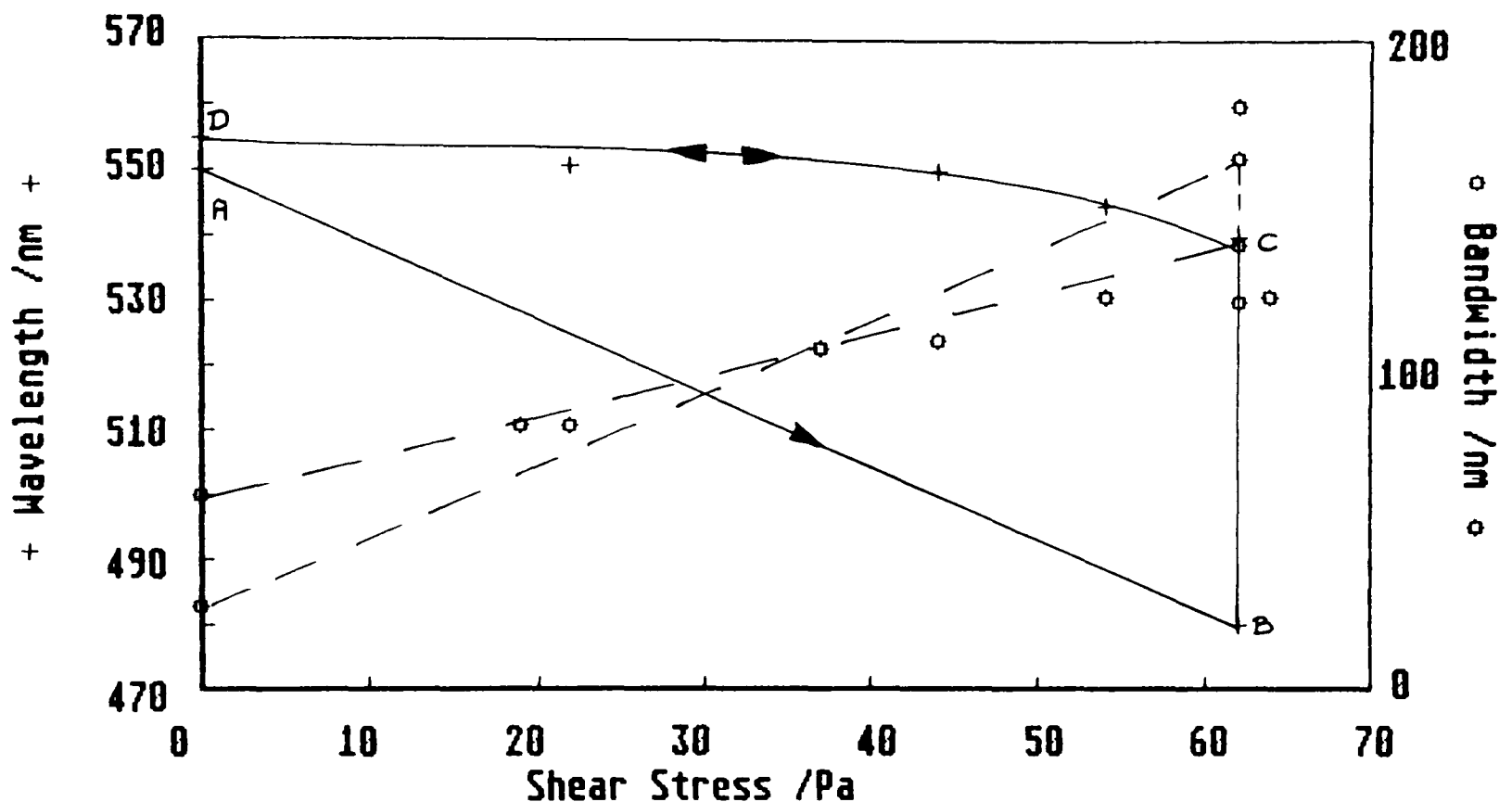


PLATE 12. Cholesteric liquid crystal used to visualise the laminar to turbulent transition location on the aluminium blade in the blow down wind tunnel. A large trip has been located at the centre of the blade which is causing a turbulent wedge. Bright colour is maintained in the material at the front of the blade which is subjected to low shear laminar flow.



resulted in the line C - D being traversed. This time dependance was thought to be linked in this case to the material flowing away from the surface and leaving a thinner layer, though the complication of a time dependance is unattractive for a practical method.

FIGURE 71. Wind tunnel results showing the effect of a sudden increase of shear stress on the peak reflected wavelength, with subsequent relaxation over time.



Although small changes in colour were therefore detectable with coarse, dramatic changes in shear stress, the technique did not allow an accurate assessment of the shear stress/colour relationship, thus limiting its value to transition location where sharp changes in surface shear stress may occur.

A slight improvement on this method may be achieved by using an infra-red to visible colour change. A cholesteric mixture having a pitch length in the infra-red at the operating temperature, e.g. E7+20%CB15, was applied to a black background such that the layer would be transparent to visible light,

and the surface, in the unsheared condition, would appear black. When sheared the layer would distort and produce a reflection in the visible (red). Although this method in principle should provide a clear and reversible indication of high shear regions, giving an on/off effect under ideal conditions, the perceived colour change will be dependent on the lighting conditions as before.

In order to evaluate this method the material (E7+20wt%CB15) was sandwiched between two glass disks. A bright red reflection was produced when one disk was rotated over the other, which completely disappeared when the shear was removed. However the disks had to be viewed normally, as otherwise the colour change produced was between two different hues of red, which was hard to discern. This method was not pursued further.

In order to achieve more visible colour changes that could be accurately calibrated with shear stress two new methods employing liquid crystals were developed and evaluated, and these are discussed in detail in the next chapter.

4:3. ALTERNATIVE METHODS TO MEASURE SURFACE SHEAR STRESS USING LIQUID CRYSTALS.

As a consequence of the limitations of using the shear induced colour change in cholesteric liquid crystals to measure surface shear stress discussed above, two alternative techniques were developed and evaluated. The first technique, which shows great promise, employed the shear induced texture change from the focal-conic texture into the Grandjean texture in a cholesteric liquid crystal. The second method assessed the use of birefringence colour changes in an aligned nematic layer applied to the surface.

4:3.1. Focal Conic to Grandjean Texture Change Effect

This method appeared the more promising and so greater emphasis was placed on its thorough evaluation. It is well known that mechanical shearing may be used to align liquid crystal phases.^{71,77} so it was hoped that a shear threshold at which alignment occurred could be calibrated for a particular material. At this shear level the material would flow align from the colourless focal-conic to the coloured Grandjean texture, producing a visible boundary which would correspond to a particular surface shear stress. The material would, as usual, have to be applied to a blackened surface, but because a single boundary/colour change would be used the complexities relating to viewing and lighting angle effects would be removed.

The material may be applied to the surface in neat form, by painting the material on with a brush, silk screen printing, using a doctor blade or spraying on in a solvent. The mixture used was formulated to have a wide enough cholesteric phase to cover the full range of test temperatures encountered, but with a clearing temperature which was not too high, to enable easy heating of the mixture into the isotropic phase in order to achieve

the initial focal conic state. The initial focal-conic texture could be generated by three methods:

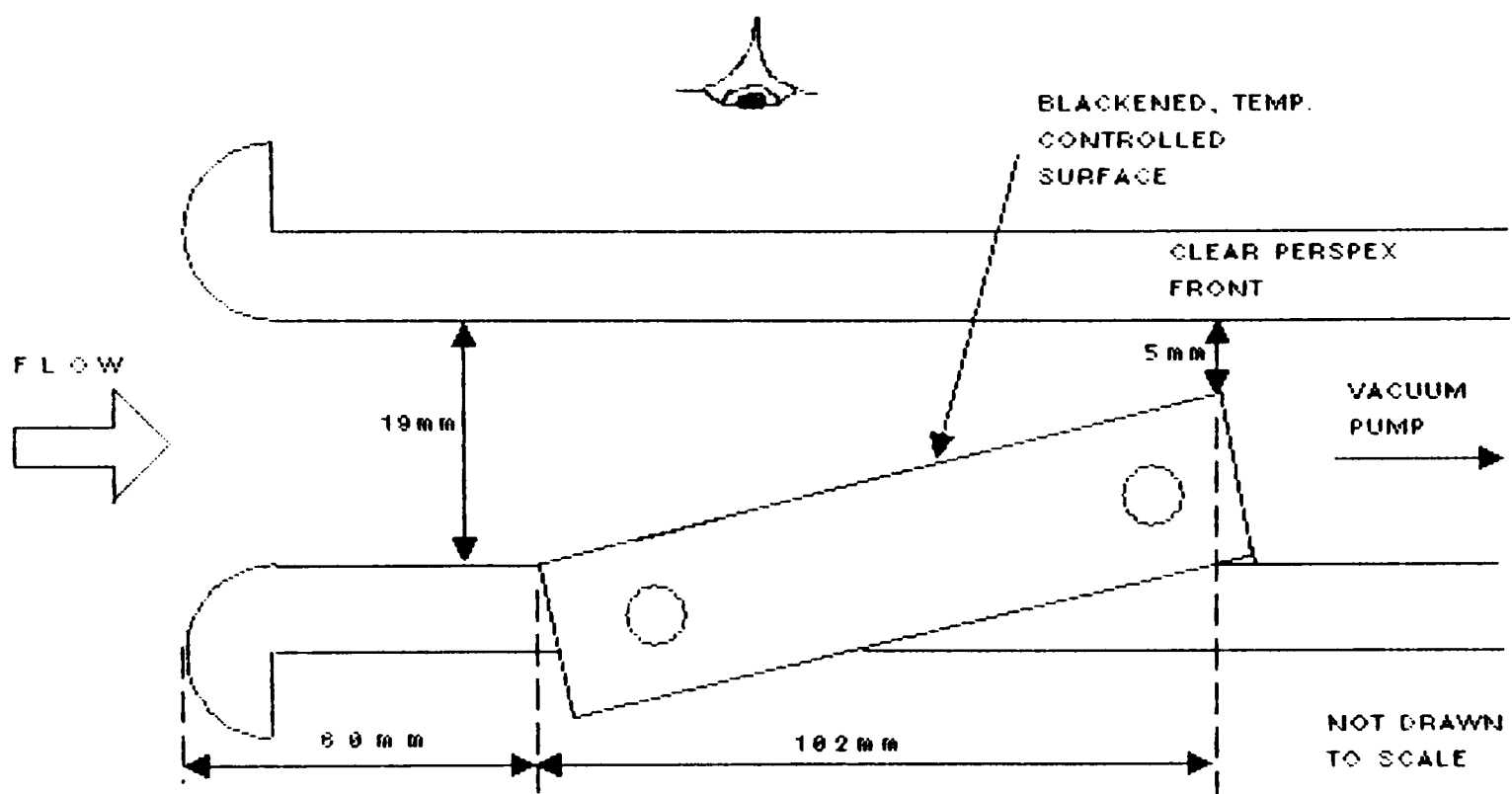
1. Solvent Method. The neat liquid crystal material may be mixed with a suitable solvent to produce an isotropic liquid. This may then be conveniently sprayed onto the model surface to give a thin, even layer. As the solvent evaporates off, the cholesteric phase forms in the thermodynamically preferred focal-conic texture. Suitable solvents would include 1,1,1 trichlorotrifluoroethane, geneclene, or indeed many industrial solvents with good volatility, low flammability, low toxicity which do not react with or contaminate the liquid crystal.

2. Focal Conic Promoting Agent Method. The neat material may also be mixed with a focal conic promoting agent. This is a particulate material, which is chemically inert to the liquid crystal, and forms a colloidal suspension causing a nucleation of focal-conic groups. A rough surface would have this effect for a thin layer, but it is more effective to generate the focal-conic state in the bulk material. In the tests described a small particulate additive of silicon dioxide (Aerosil) was mixed with the liquid crystal to achieve this. The particles were small enough (ave. diameter 12nm) so as not to cause any surface roughness in the resulting layer. Aerosil particulates are available from Degusa^{C5}. When mixed in small proportions with the liquid crystal material they strongly promote the focal conic texture. Other useful effects are also produced, such as increasing the mixtures bulk viscosity and non Newtonian flow properties.

3. Heating Method. The material may be applied to the surface in a thin, uniform layer and then heated into the isotropic phase with a hot air gun. On cooling the material will return into the cholesteric phase in the focal conic, disordered texture.

Tests using layers of cholesteric liquid crystal in the focal-conic state were conducted in the same blow down wind tunnel as described in the last chapter, but incorporating a modified channel wall. This consisted of a temperature controlled blackened copper block, which was inserted at an angle in the channel to produce a converging duct. This produced a large shear stress gradient along the block, (see figure 72).

FIGURE 72. Converging duct used to produce a large shear stress gradient in the blow down wind tunnel.

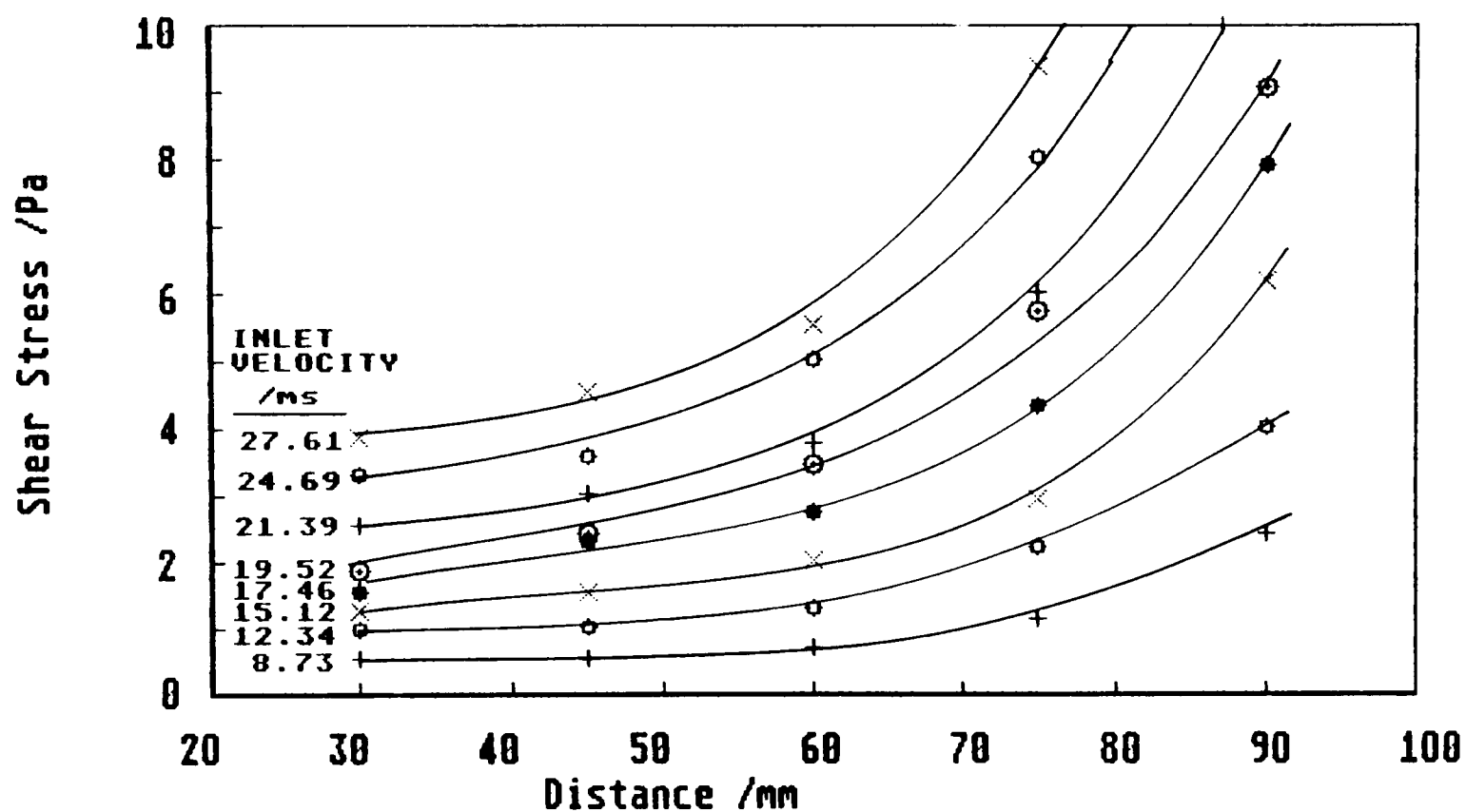


The block temperature was controlled by circulating a thermostated liquid through it, enabling the block temperature to be stabilised to $\pm 0.1^\circ\text{C}$ over the range -20°C up to about 50°C .

An aluminium block of identical dimensions to the copper block was also made, which had static pressure tappings along its length. This block could be inserted in place of the copper block which allowed the shear stress levels along the converging surface to be calibrated by means of Preston tube measurements. Initial tests using the previous channel geometry had indicated that the shear level required to produce the texture change would be low,

hence pressure measurements were taken with an alcohol manometer in order to achieve an accurate calibration. These results are shown in figure 73, for a range of flow rates. Corresponding flow rates were achieved with the copper block in place by matching the pressure values measured at a static pressure tapping on the inlet channel wall.

FIGURE 73. Surface shear stress calibration of the converging duct from Preston tube measurements.



Results of tests using Liquid Crystal applied in a Solvent

A number of different solvents were tried, including geneclene, acetone and paraffin. These differed in their evaporation rates at room temperature. Paraffin evaporated very slowly, and this resulted in large focal conic domains in the liquid crystal layer, which were observed with a polarising optical microscope. The others evaporated relatively quickly and formed small domains. The focal-conic domain size had no detectable effect on the results.

Mixing the liquid crystal with the solvent had the advantage of

allowing the resulting mixture to be sprayed over a test surface to quickly achieve thin, uniform coatings. However, the shear threshold results were not reproducible and in some cases, when a high concentration of solvent was used the colour was very poor. These problems may be due to the amount of retained solvent in the surface layer. It is difficult to remove all traces of solvent from the liquid crystal layer, and the retained solvent has an effect on the viscosity and transition temperatures of the mixture. This makes this method unreliable for quantitative measurements, so it was not used further.

Results of Tests with Aerosil

The addition of aerosil particles to the liquid crystal in small (~1 to 5) weight percentages did not greatly promote the focal conic texture, so tests had to be conducted by heating the material into the isotropic phase. Larger quantities of aerosil additive removed the selective reflection and the material appeared white and cloudy, but a flow aligned order could not then be produced. The particles did greatly increase the mixture viscosity. Hence all results presented below were carried out using heating to achieve the focal conic texture, either with or without the aerosil additive, which was used purely to increase mixture viscosity.

Results of Heating Tests

Test surfaces were coated with one of the following mixtures; E7+35%CB15, E9+35%CB15, S2+33%CB15 and C-N/G114F.

Shear induced flow alignment of the focal conic texture produced a very clear and well defined boundary with the Grandjean texture. The bright selectively reflecting Grandjean texture first appeared at the end of the converging block, i.e. the narrowest point in the channel where the shear stress was highest. At a constant flow rate the boundary between the two textures moved down the channel with time toward lower shear stress regions.

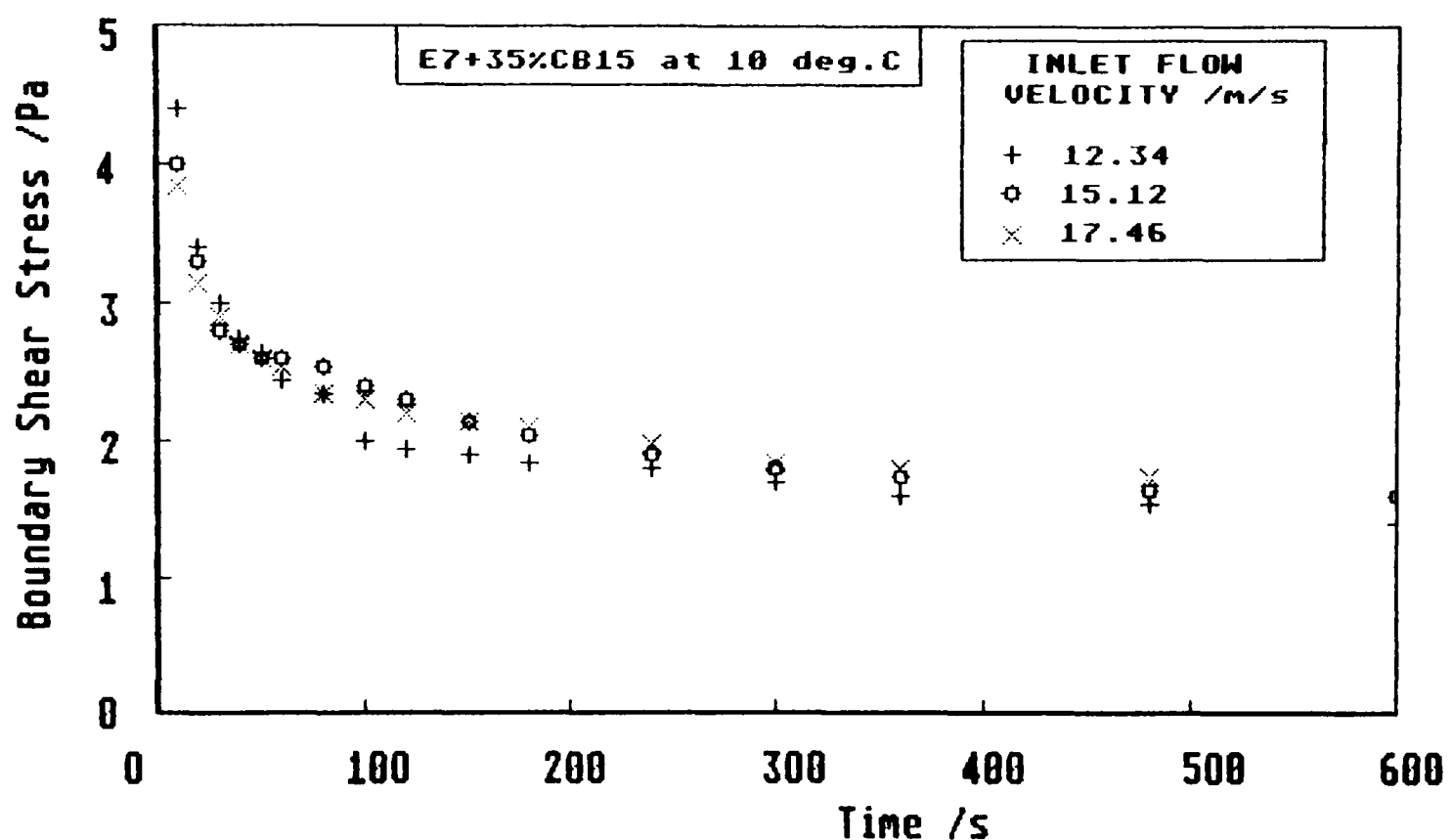
PLATE 13. Sequence of photographs demonstrating the use of the focal-conic to Grandjean texture change method to indicate surface shear stress levels. As the flow rate is increased the alignment boundary moves forward until in the final frame the whole surface is aligned and colour changes begin to occur in the high shear area at the rear of the converging duct. Flow is from right to left.



At higher flow rates the boundary moved forward faster, until at very high rates the shear induced colour change in the Grandjean texture became apparent, see plate 13.

The shear stress level at the boundary was found from the Preston tube calibration (figure 73), by measuring the position of the boundary for the different materials at different temperatures as a function of time. The results for E7+35%CB15 at 10°C are shown in figure 74.

FIGURE 74. Shear stress at the texture change boundary as a function of time, for various inlet flow rates.



The fact that the boundary position is time dependent indicates that there is not a well defined shear threshold at which flow alignment occurs. However, it is interesting to note that the boundary shear stress level was independent of flow rate, at a *particular time*. In other words, after 30 seconds the boundary would have moved further down the duct for high flow rates than for low flow rates, but the boundary position would correspond to the same surface shear stress level in both cases. This indicates that a general

FIGURE 75. Shear stress at the texture change boundary as a function of time, at various temperatures. E7+35%CB15, measured in the converging duct in the wind tunnel.

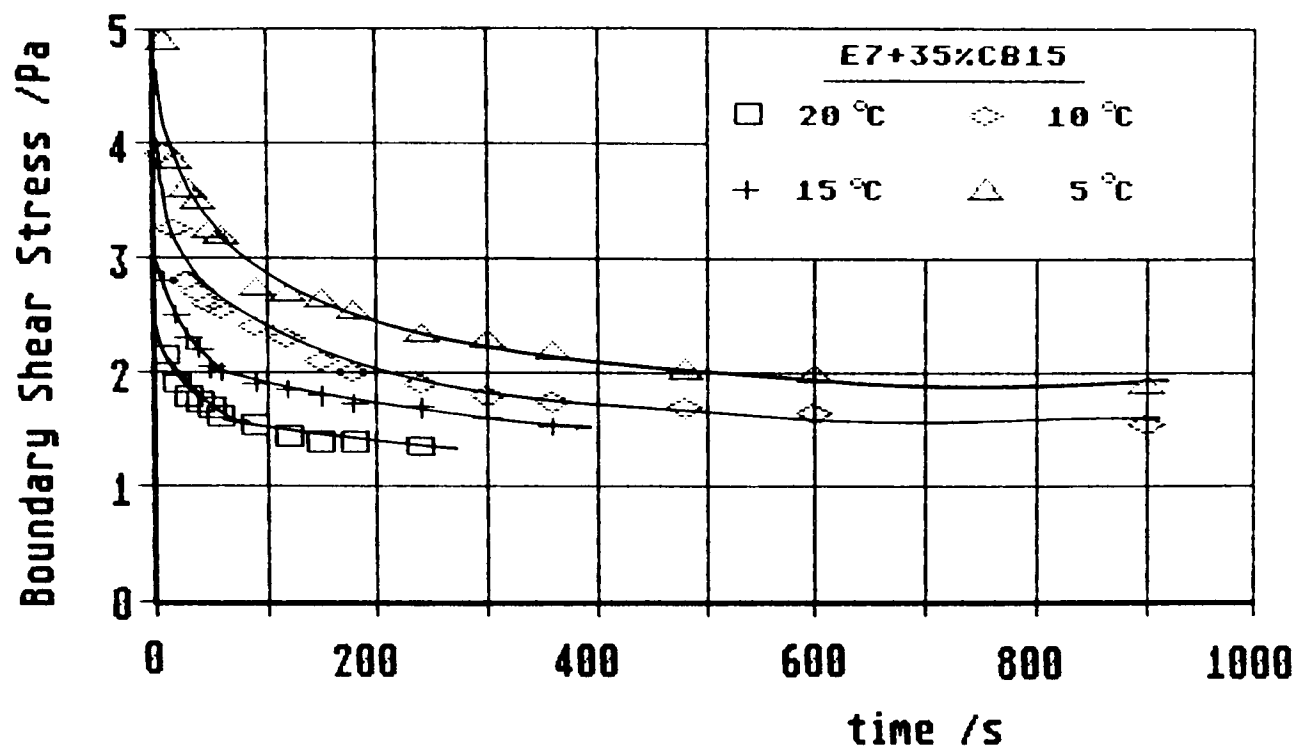
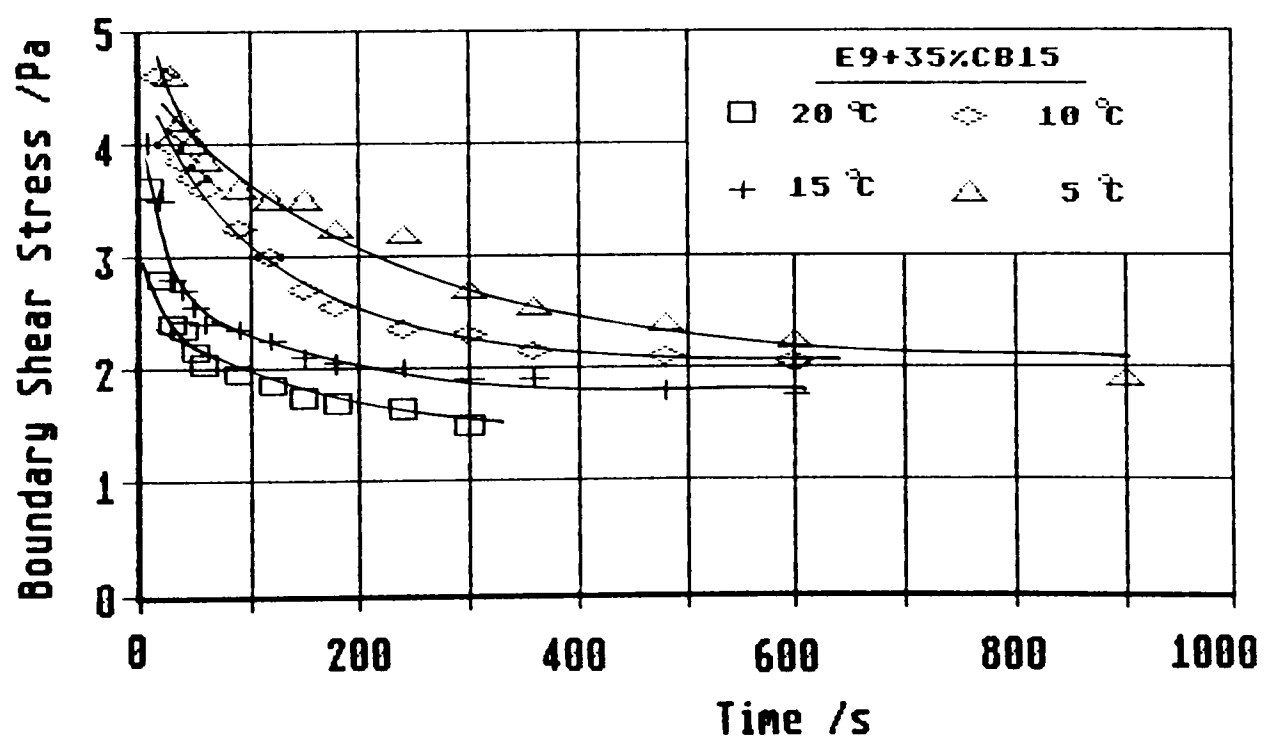


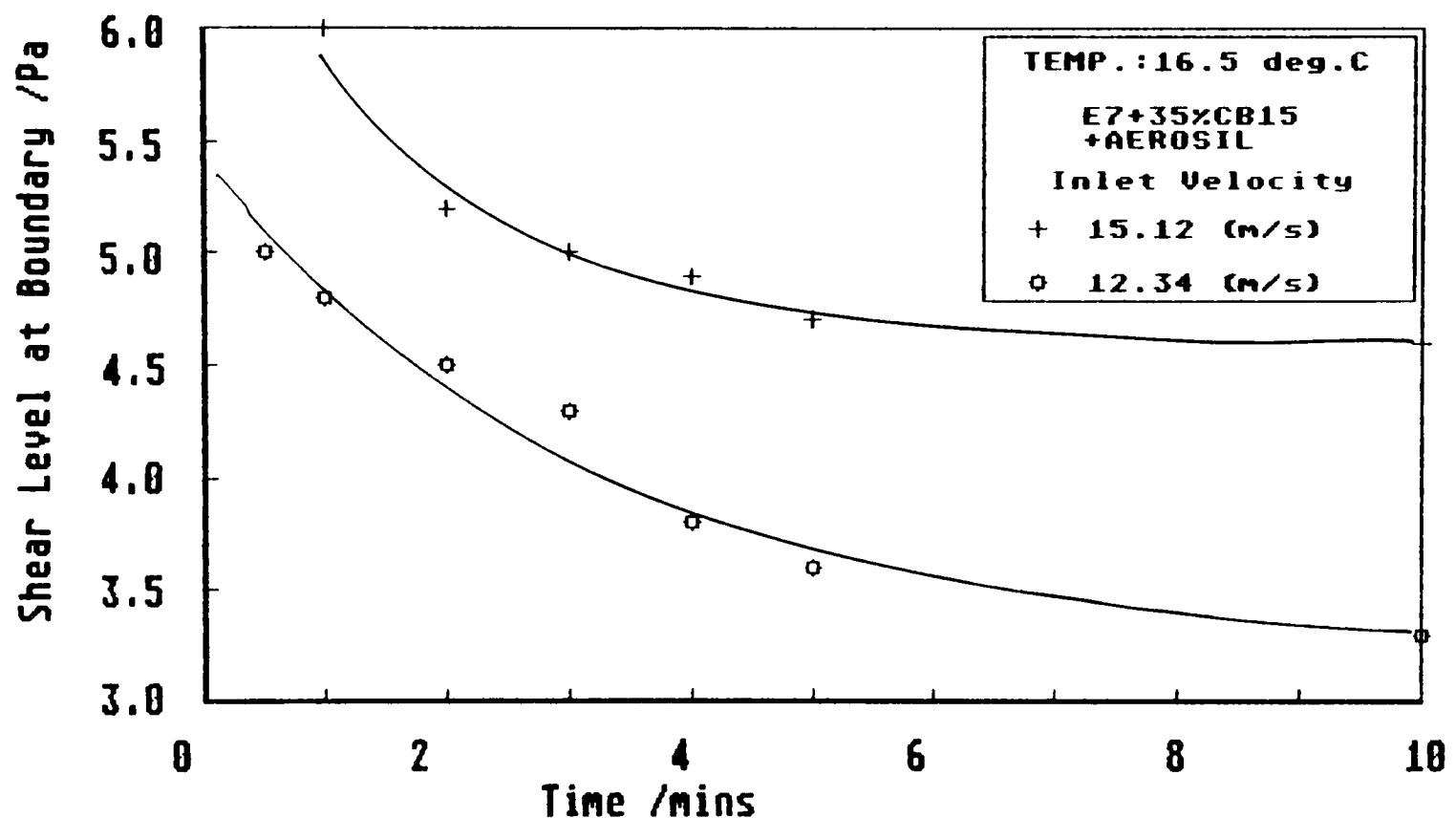
FIGURE 76. Shear stress at the texture change boundary as a function of time, for E9+35%CB15.



calibration could be achieved for one material as a function of time at constant temperature, which could enable a practical measurement method. Also a critical flow driven displacement or displacement gradient to produce alignment is indicated, rather than a shear stress threshold.

The boundary shear stress level over time was also investigated as a function of temperature. At a particular time the shear stress level at the boundary was proportional to the mixture viscosity, as shown in figures 75 and 76 for materials E7+35%CB15 and E9+35%CB15.

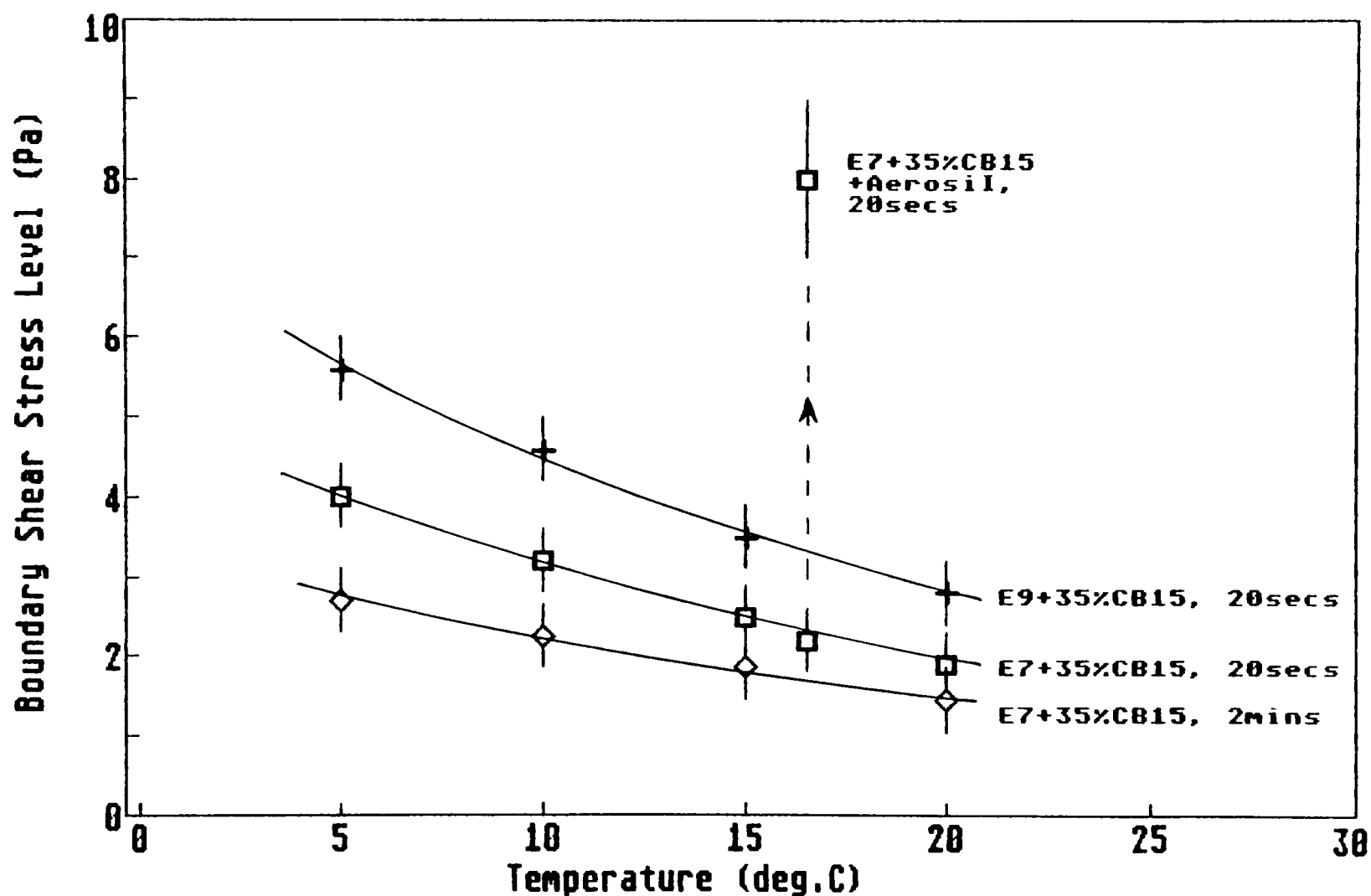
FIGURE 77. Shear stress at the texture change boundary as a function of time for E7+35%CB15+Aerosil, at different inlet flow rates.



Similar tests were carried out on mixtures with 2 wt.% aerosil added, which increased the bulk viscosity of the mixture and the boundary shear stress level. In this case an unexpected variation of the time dependance of the boundary shear stress level with flow rate was observed, see figure 77, though this may be due to thickness variations in the layer between the two tests.

At any particular time (e.g. 20 secs) a plot of the boundary shear stress level against temperature for all materials may be drawn. (figure 78). This clearly shows the effect of material viscosity on the results. For example at 15°C the bulk viscosity of the E9 mixture was 194cp, approximately double that of the E7 mixture, whereas the boundary shear stress after 20 seconds has only increased by a factor of 1.4 between the two mixtures. Adding aerosil produces a much more viscous mixture, with correspondingly higher boundary shear stress levels.

FIGURE 78. Shear stress at the texture change boundary as a function of temperature for various materials.

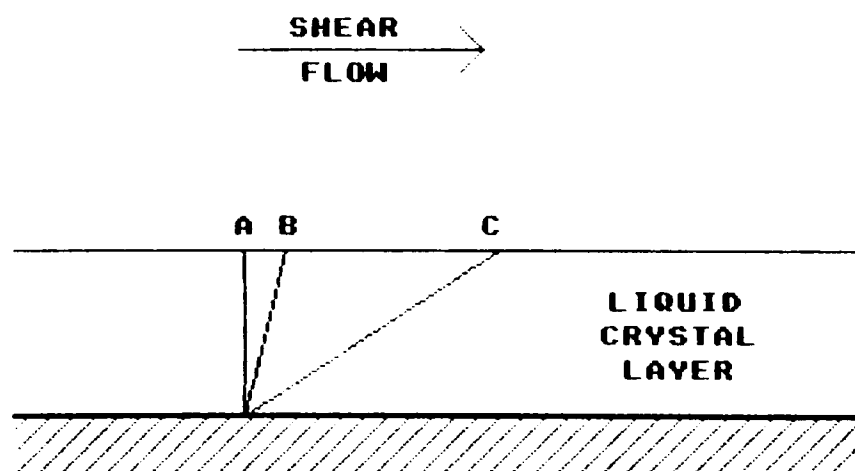


These results indicate that a single material may be used to measure a range of shear stress values over a range stretching from the boundary shear stress at a maximum time up to a limit dictated by the accuracy with which flow onset occurs. For example E7+35%CB15 could

reasonably be used to measure between about 1.2 Pa to 3 Pa at 15°C. Different mixtures (or different temperatures) could be used to access different ranges. In order to calculate the boundary shear stress the time from flow onset and a calibration curve for the material at the operating temperature are required, such as figures 75 or 76. (page 144)

The time dependance of the boundary shear stress level implies that a certain flow driven displacement gradient is required to align the material, and at high shear rates the displacement is achieved quickly, while at low rates it takes longer.

FIGURE 79. Simple model of shear stress applied to a liquid crystal layer. It is assumed that elastic flow occurs from 'a' to 'b', followed by a shear driven displacement from 'b' to 'c' which causes the material to flow align.



Using figure 79 as a model, it may be assumed that a small shear stress, τ_0 , is required to overcome the cohesive elastic binding forces. This level is temperature dependent as shown in figures 75 and 76. Once this level has been reached the layer starts to flow (from b to c). In this region flow alignment occurs until at c a well ordered Grandjean texture is achieved. The point at which the eye perceives a colour onset may occur between b and c.

Let us assume, initially, that there is a critical displacement gradient, d_c/x , required for flow alignment.

From the definition of surface shear stress:

$$\tau = \eta \cdot \frac{dv}{dx}$$

If a threshold exists then we may modify this for the shear on the liquid crystal layer to:

$$\tau = \tau_o + \eta \cdot \frac{dv}{dx}$$

The displacement as a function of time may be calculated from the shear rate, dv/dx , :

$$\text{displacement, } d_c = x \cdot \frac{dv}{dx} \cdot t$$

where x is the layer thickness. Therefore:

$$\tau = \tau_o + \frac{\eta}{t} \left(\frac{d_c}{x} \right) \quad - - - 4.4 - -$$

Hence plotting τ against $1/t$ should produce a straight line which intercepts the y-axis at τ_o , and has a slope of $\eta \cdot d_c/x$.

Figures 80 and 81 present plots of τ against $1/t$ for the E7 and E9 based materials. These are clearly not straight lines, and indicate that either η or the ratio d_c/x is larger at longer times, i.e. when the boundary was in the low shear stress regions. In order to analyse these curves more information on the rheological properties of the materials was required. Therefore a series of experiments was conducted to determine how the material bulk viscosity varies in the focal-conic and Grandjean textures as a function of shear rate and time.

FIGURE 80. Measured shear stress level at the texture change boundary as a function of 1/time for E7+35%CB15.

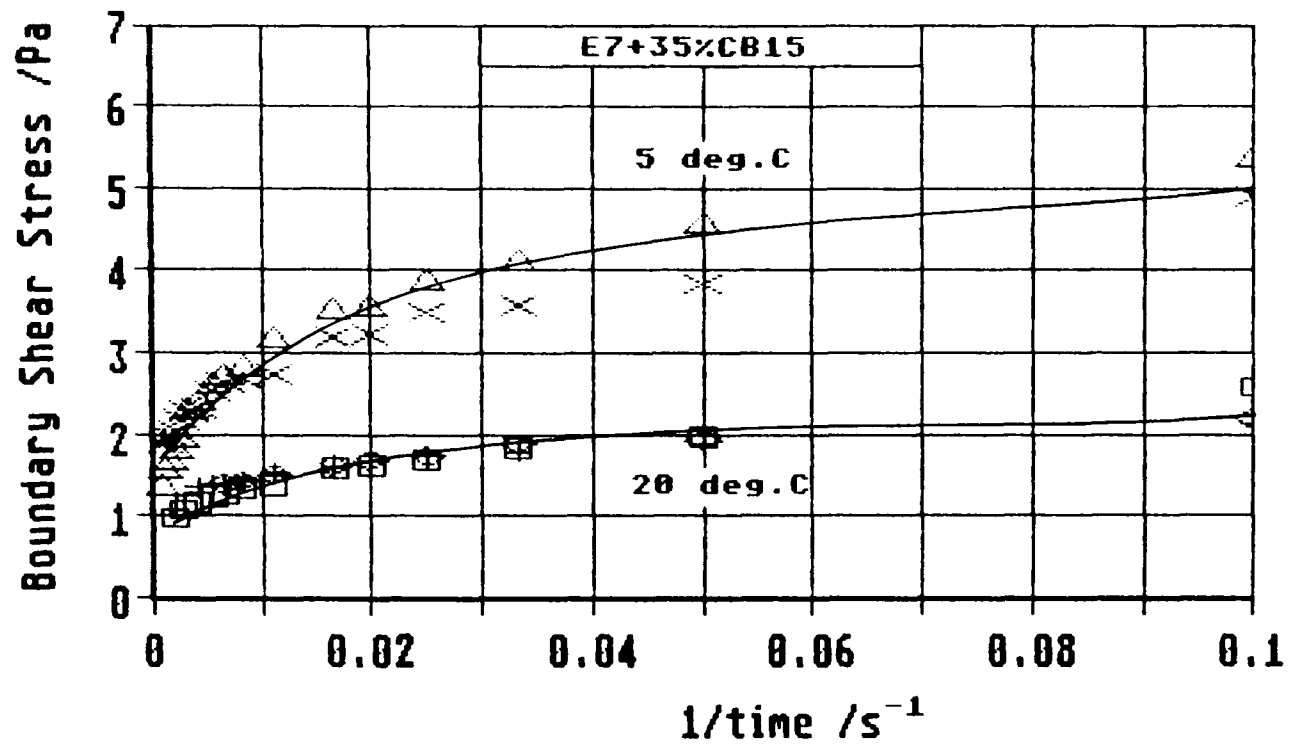
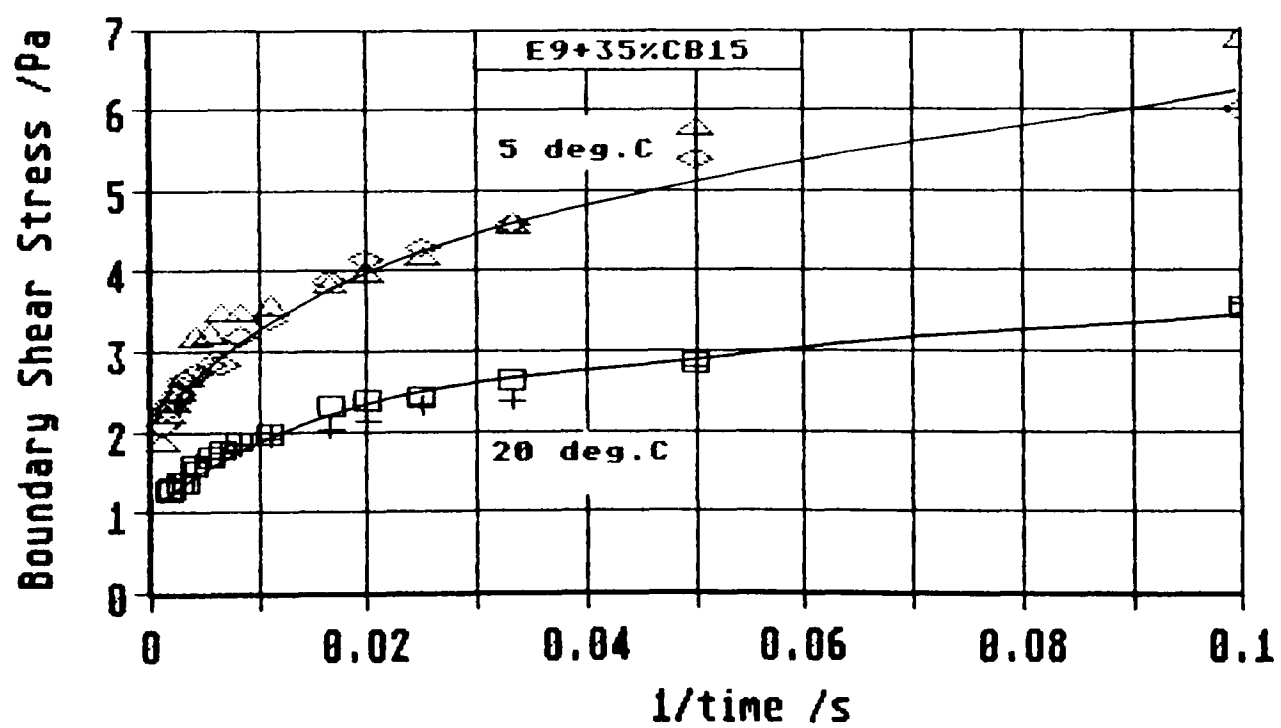


FIGURE 81. Shear stress at the texture change boundary as a function of 1/time for E9+35%CB15.



4:3.1.1. Viscosity Studies

Numerous studies have been carried out on the complex viscosity properties of nematic liquid crystalline materials^{77,78,79}, which have shown that the angle made by the nematic director to the flow direction is dependent on the viscosity coefficients, but independent of shear rate.

The situation for cholesterics is more complex as a consequence of the helical ordering. Theoretical calculations based on Leslie's theory^{14,80,81} indicate that the apparent viscosity is pitch dependent, but this dependence diminishes when the pitch length becomes small, and is insignificant for pitches producing a reflection in the visible.

Other studies^{17,82,83,84} indicate that the viscosity is extremely dependant on the shear rate, increasing by a factor up to 10^6 at very low rates of shear²⁵, and that texture changes can occur when the shear force overcomes molecular cohesive forces⁶⁷. References 85,86 and 87 have studied the changes in viscosity that occur over the isotropic to cholesteric phase transition for both cholesteryl ester and chiral nematic materials. In all cases the changes observed are dependant on the orientation of the material helix to the flow direction. It appears that no studies have been directed specifically toward the viscosity changes occurring at a focal conic to Grandjean texture change.

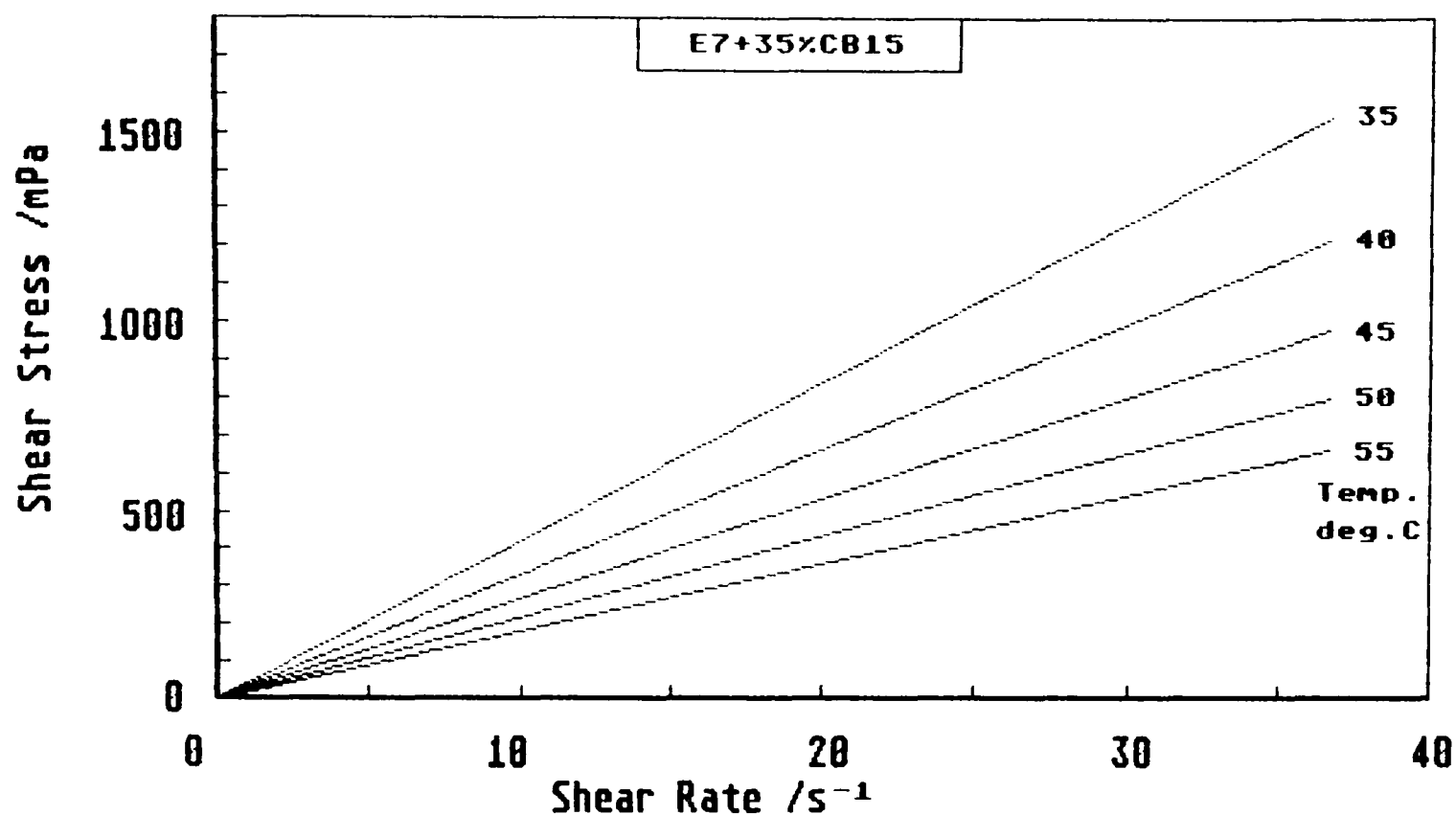
Tests were therefore carried out using a Contraves Low Shear 30 concentric cylinder viscometer as described previously, see figure 43.(page 92) A programmable unit was attached to the instrument to enable plots of shear rate, temperature and time to be carried out and plotted on an x-y recorder. Plots of shear stress against shear rate provided a useful insight into the material flow characteristics, and these are examined below for each phase.

(i) Isotropic Phase

The isotropic phase of a liquid crystalline material behaves as a

Newtonian fluid, hence plots of shear stress against shear rate were straight lines through the origin, with a slope equal the the viscosity, see figure 82.

FIGURE 82. Shear stress against shear rate for E7+35%CB15 measured in the isotropic phase on the Contraves Low Shear 30 viscometer.



The viscosity is given by:

$$\eta = \frac{\tau}{dv/dx}$$

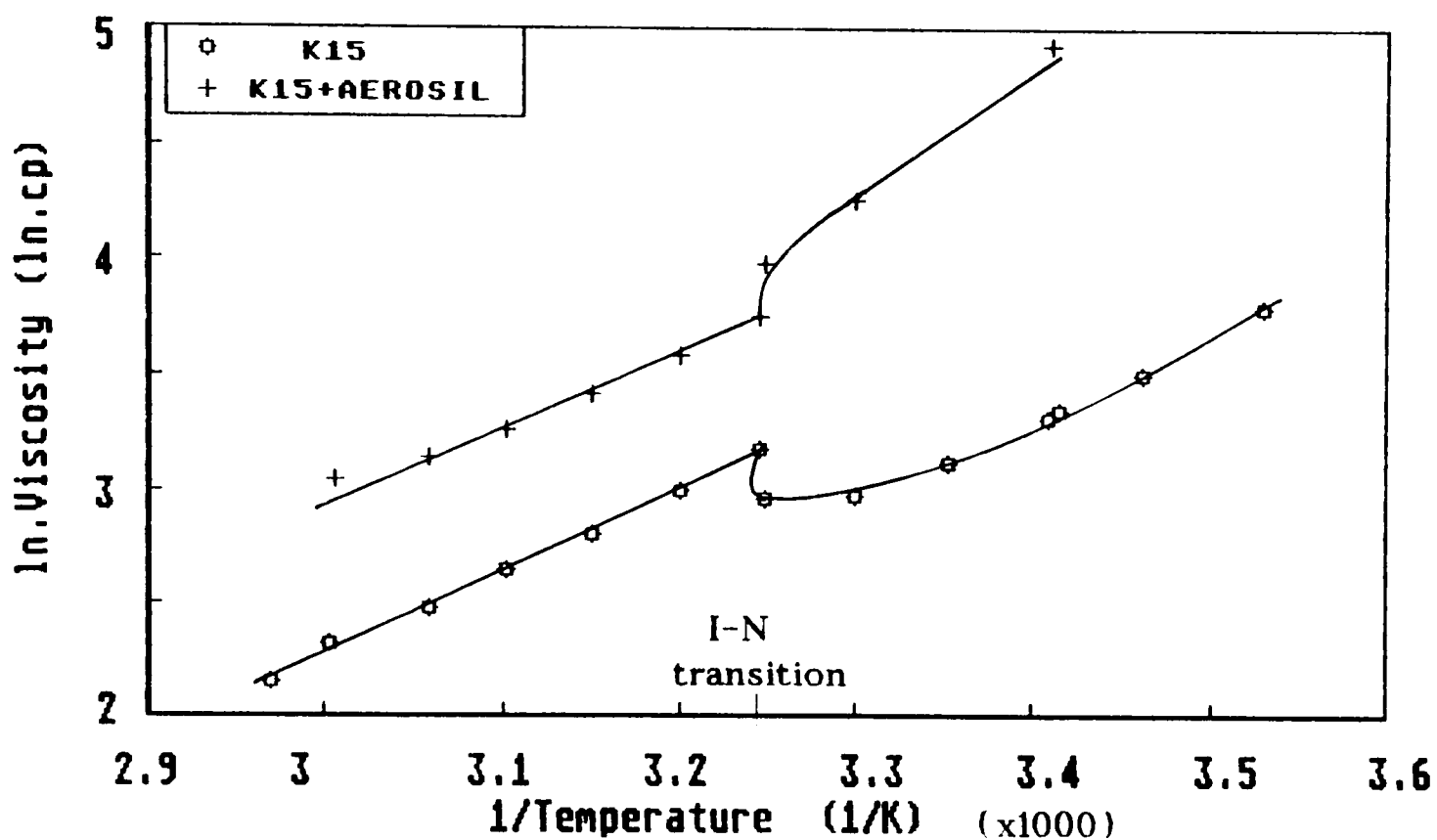
An approximation to the viscosity in the liquid crystal phase may be obtained by extrapolating the isotropic values to lower temperatures. This may be readily achieved using the straight line plots of $\ln.\eta$ verses $1/T$, as reported in chapter 3:6.

(ii) Nematic Phase

These experiments were carried out using 4-n-penty-4'-cyanobiphenyl (K15). In the nematic phase the material flow aligned to give a Newtonian 'aligned' viscosity value. The measured viscosity dropped on entering the flow aligned nematic phase from the isotropic phase, see figure 83. Addition of 2%

by weight of Aerosil to the material greatly increased the bulk viscosity (by approximately a factor of 5) and caused a rise at the nematic to isotropic transition, probably as a result of the increase in the disclination density induced by the particles.

FIGURE 83. $\ln(\text{viscosity})$ against $1/(\text{temperature})$ for the nematic material K15 measured in the isotropic and flow aligned nematic phases. The upper curve shows the effect of adding aerosil to the material.

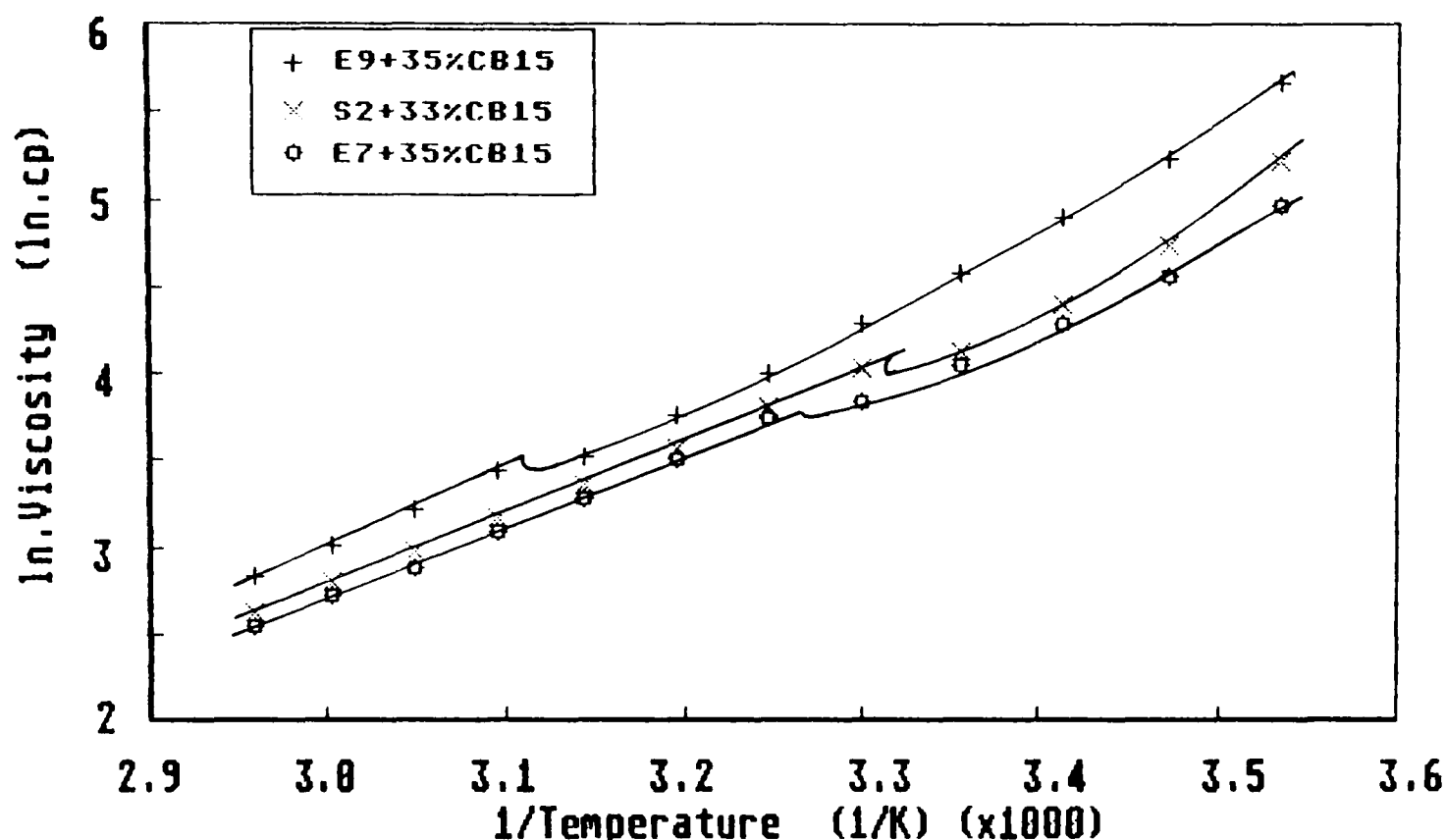


(iii) Cholesteric Phase

As in the case for the nematic material, *flow aligned* viscosity values were obtained for chiral nematic materials which showed Newtonian behaviour. Hence plots of flow aligned viscosity against temperature could be drawn for the materials used, see figure 84. The shear rate used had to be high enough to completely flow align the material. If a high shear rate was applied, plots of shear stress against temperature could be plotted directly from the instrument, which corresponded closely with figure 84. A TM75/74 mixture was examined and this showed a large peak in viscosity at the cholesteric to isotropic transition, which has been attributed to the presence

of a blue phase⁸⁷. see figure 85.

FIGURE 84. Log.(viscosity) against $1/(\text{temperature})$ for the cholesteric materials E7+35%CB15, E9+35%CB15 and S2+33%CB15 measured in the isotropic and flow aligned cholesteric phases.



In order to analyse the viscosity changes occurring at the focal conic to Grandjean texture change, and the dependence on shear rate and time two types of plots were utilised.

(a). Plots of shear stress against shear rate. The materials were sheared in the focal conic phase and the resulting viscosity changes were shear rate and time dependent. A typical variation of shear stress against shear rate is shown in figure 86. The viscosity was initially large, but shear alignment occurred over time causing a reduction in the apparent viscosity. The shear rate varied radially through the layer, making exact analysis difficult, however the initial slope may be used as a rough approximation for the bulk viscosity of the focal conic texture. This gave a value of $\sim 2200\text{cp}$. As the shear rate was

FIGURE 85. Apparent viscosity against temperature plotted directly from the Contraves Low Shear 30 viscometer for two materials.

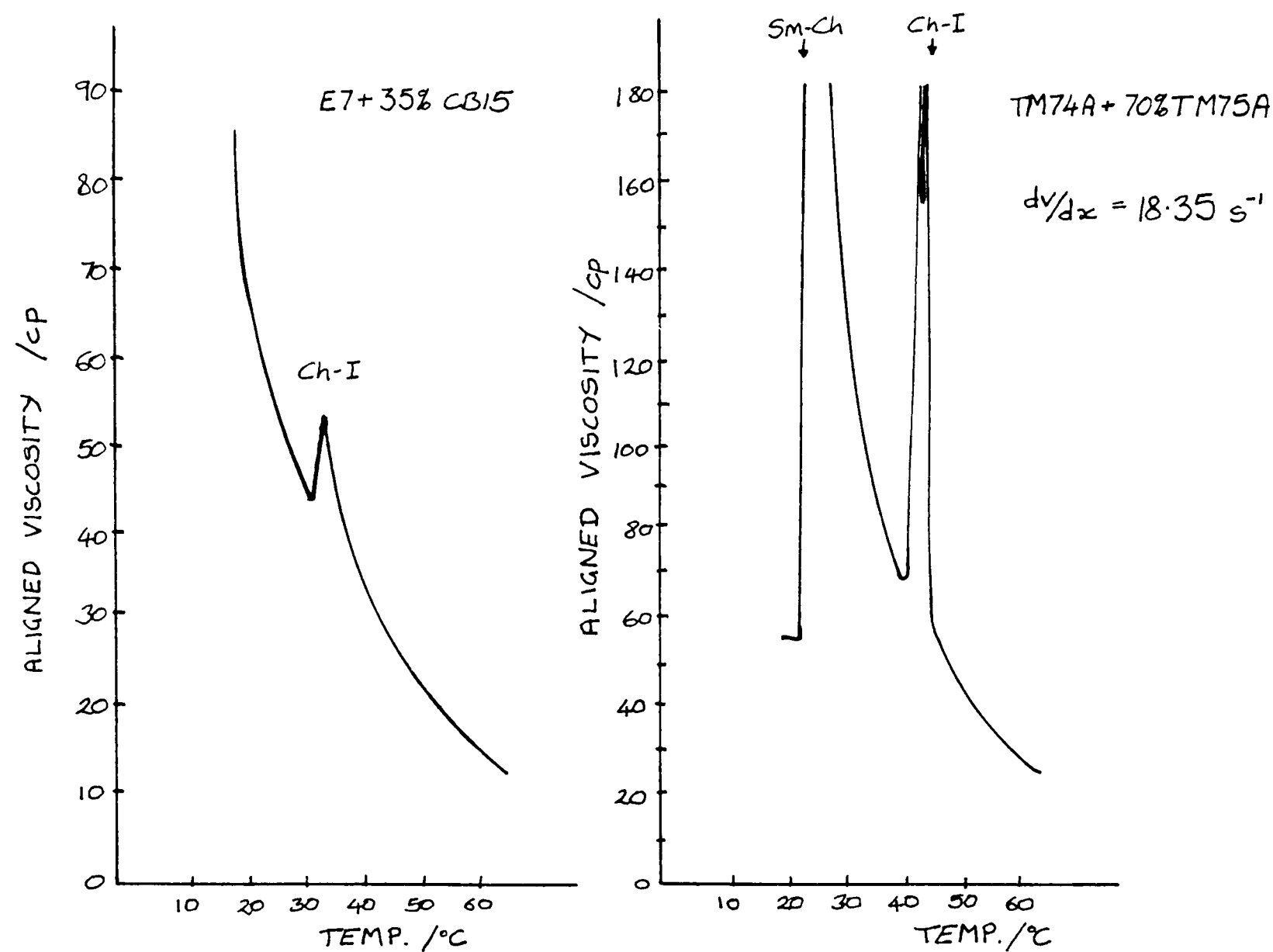
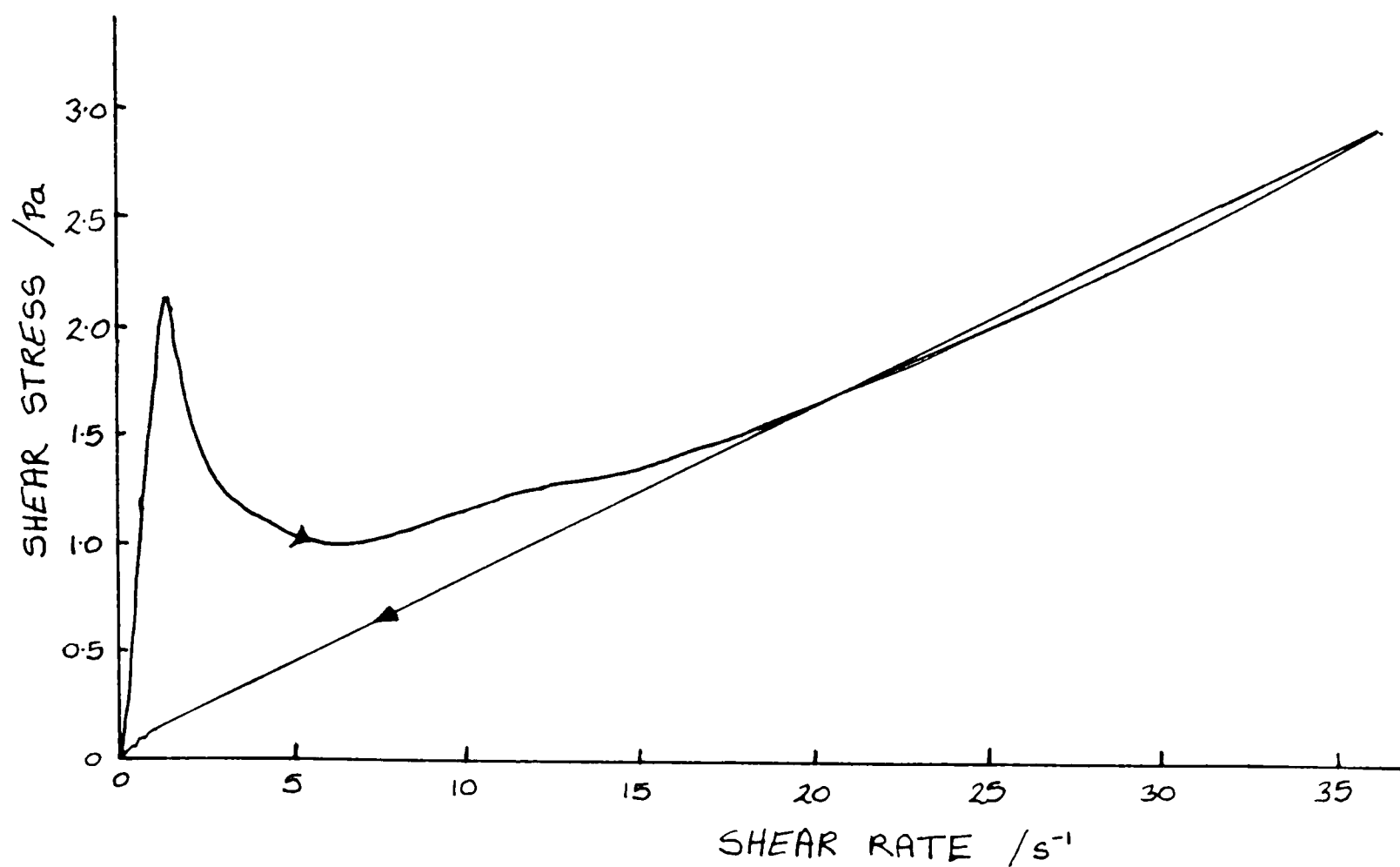


FIGURE 86. Shear stress against shear rate for E7+35%CB15 measured on the Contraves Low Shear 30 viscometer in the cholesteric phase.



increased the material flow aligned radially through the sample, until a single, steady slope is reached indicating that all the material was well aligned. Subsequent raising or lowering of the shear rate followed this Newtonian slope without the peak at previous low shear rates. The small amount of hysteresis evident in the flow aligned curves may be due to flow induced disclinations which increased the apparent viscosity. These relaxed away when shear was removed.

The results given in figure 86 may be converted to plots of bulk viscosity against shear rate and against an average displacement in the sample. These plots are shown in figures 87 and 88. Full alignment is not achieved until a displacement of about 400mm is reached, (although it seems reasonable to assume that the texture became ordered enough to produce selective reflection before this displacement was achieved.) This would give a ratio of d/x of 1600. The first peak in figure 86 occurred after a displacement of the cup of about 1mm.

(b). The second method utilised was to plot shear stress against time for constant shear rates. These plots, see for example figure 89(b) also show a clear peak in the shear stress after a certain time. The shear stress axis in this case is merely a measure of the displacement of the bob. The initial rise should not be interpreted as a rise in viscosity of the material, rather as the period in which the bob is reaching an equilibrium displacement. This is complicated by the fact that the viscosity drops with shearing, which sends the curve downward again. It is only this region which can be used to assess the material viscosity. Figure 89(a) shows that the final aligned viscosity is independent of shear rate at long times (as $\text{viscosity} = \text{shear stress} / \text{shear rate}$). This plot also allows an assessment of the displacement required to produce full alignment, which gives a value of roughly 450mm in each case. This gives a ratio of d/x (displacement gradient) of 1800. Again, at longer times it can be seen that the viscosity rises a little before leveling off. This is thought to

FIGURE 87. Apparent viscosity against shear rate estimated from the results of figure 86, for E7+35%CB15 at 20°C.

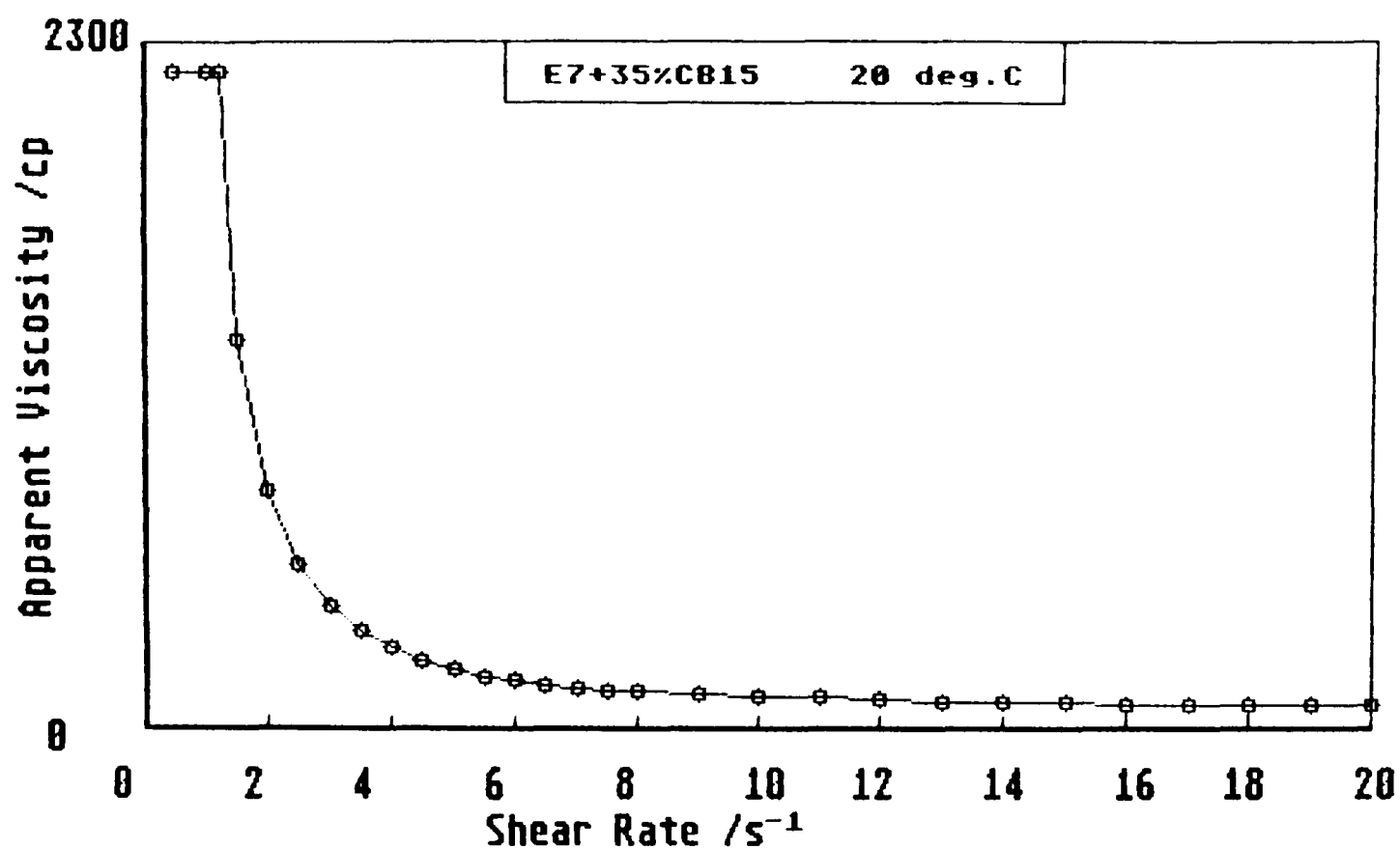


FIGURE 88. Apparent viscosity against displacement calculated from figure 86 for E7+35%CB15 at 20°C.

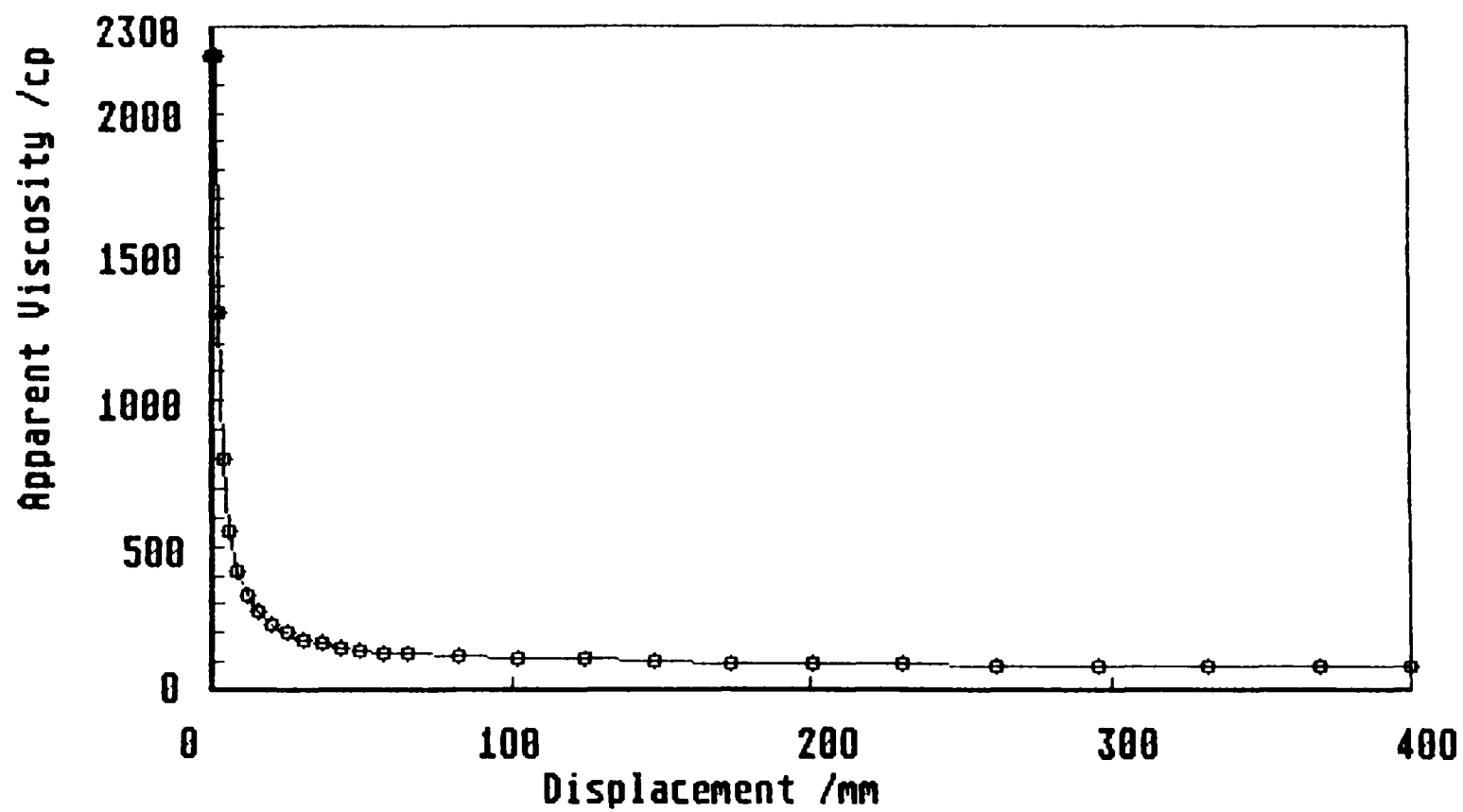


FIGURE 89.(a). Shear stress as a function of time measured at constant shear rates on the Contraves Low Shear 30 viscometer. E7+35%CB15 at 20°C.

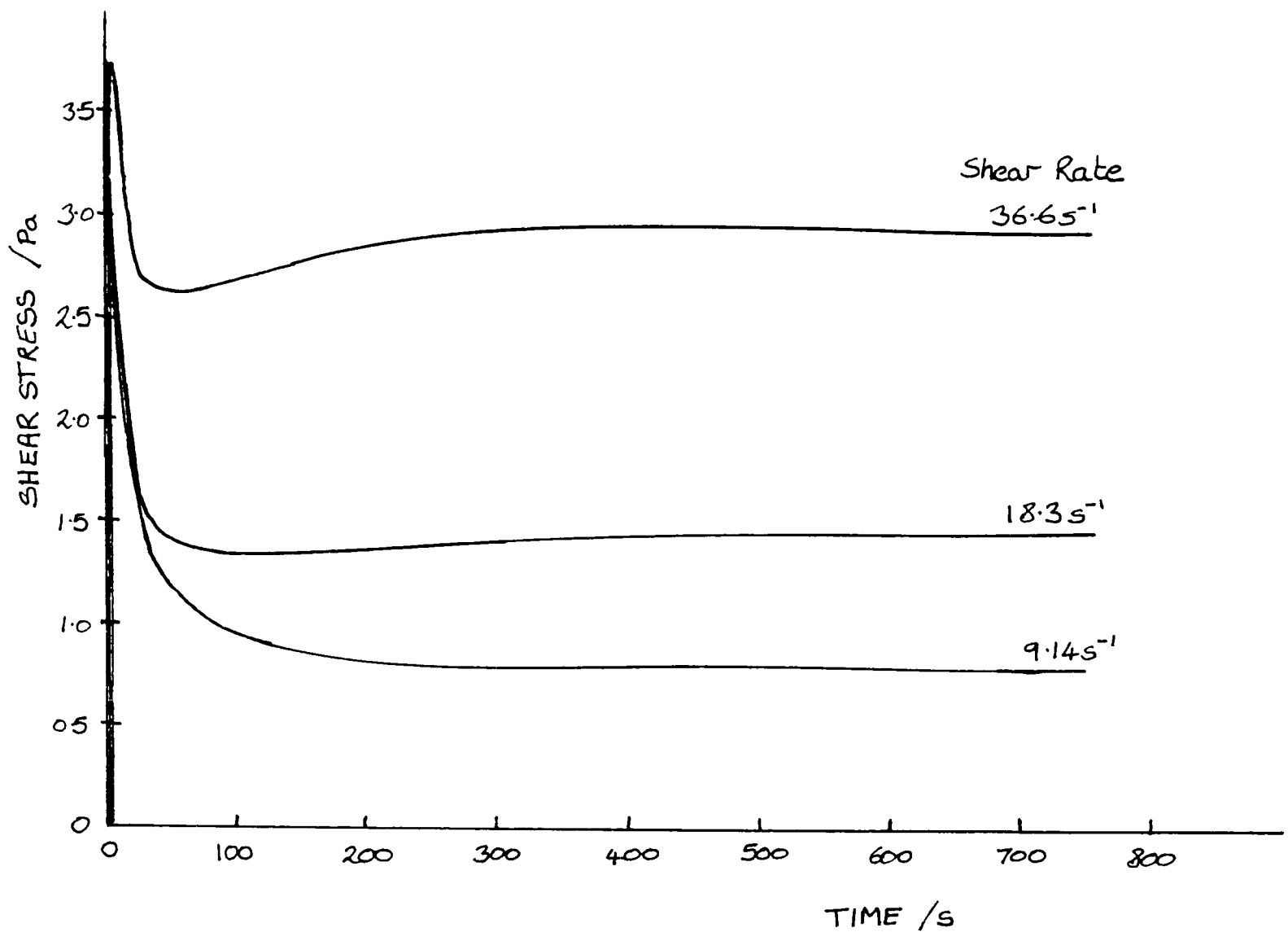
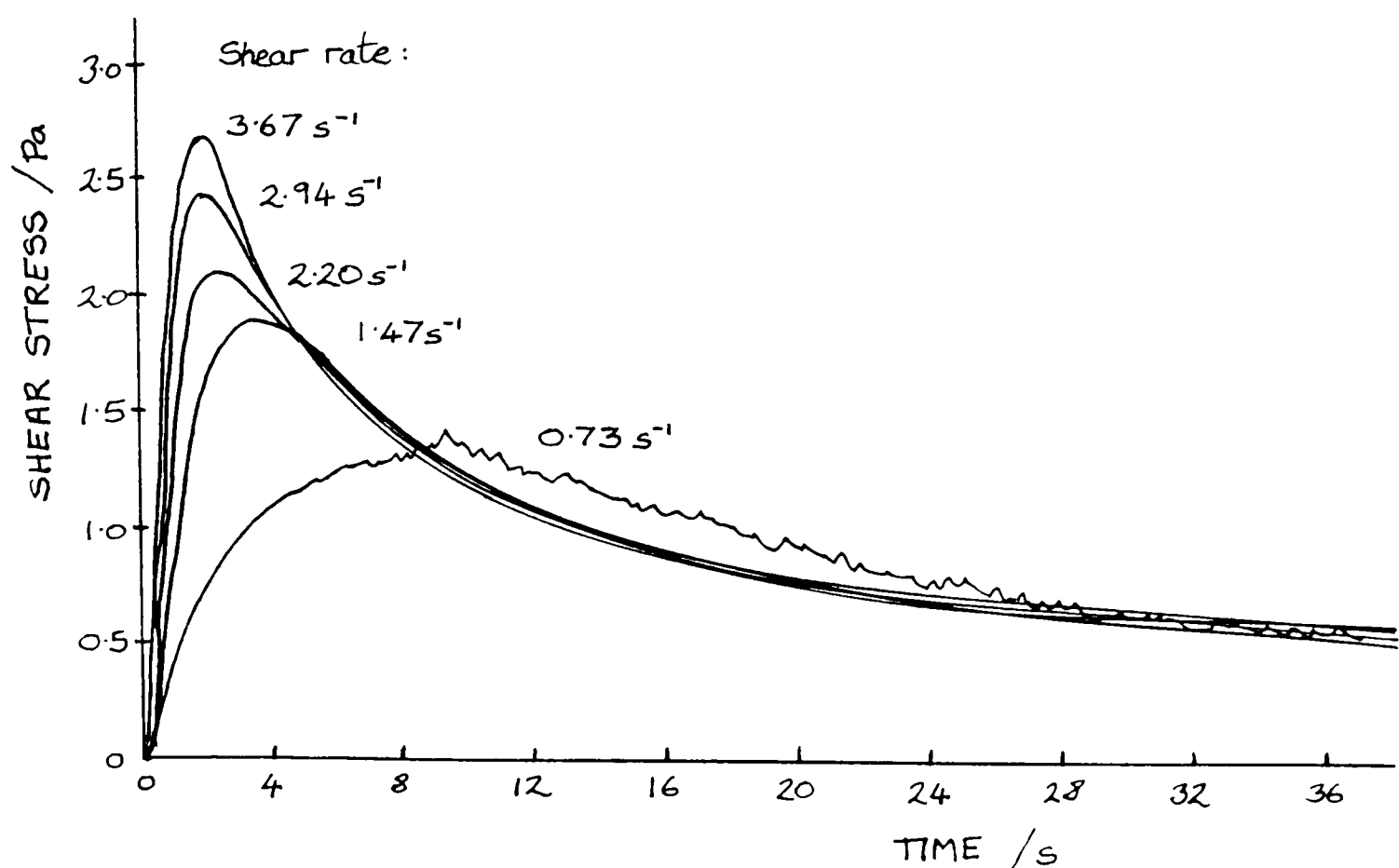


FIGURE 89.(b). Shear stress as a function of time measured at constant shear rates on the Contraves Low Shear 30 viscometer, at low shear rates. E7+35%CB15 at 20°C.



be due to the onset of shear induced disclinations, referred to above.

(iv) Cholesterics with Aerosil

The addition of small percentages of Aerosil to the liquid crystal has been observed to increase the material bulk viscosity, as well as to promote pseudo-plastic rheological properties. The non-Newtonian behaviour was evident both in the isotropic and cholesteric phases, as shown in figure 90 for E9+35%CB15 at various temperatures. At large shear rates a viscosity may be estimated from the straight line part of the curves. The viscosity at zero shear is still finite for pseudo-plastics, though it is expected that if larger concentrations of aerosil were added, plastic behaviour would be observed^{C5} and a yield stress would be observed. The original plots of shear stress against shear rate again showed very high initial viscosity, which then reduced as flow alignment occurred, but the flow aligned state is non-Newtonian in this case. The effect of different concentrations of aerosil on E7+35%CB15 is shown in figure 91. A maximum loading of 2% by weight of aerosil was determined qualitatively to enable Grandjean texture formation to be maintained after shearing.

Note: In the plots presented of shear stress against shear rate, the shear rate was ramped up and down at a steady rate, ($7.32 \text{ s}^{-1}.\text{min}^{-1}$). Changing this rate could have an effect on the shape of the non-aligned regions due to the time dependence of the viscosity at any shear rate. Plots at various rates of change of shear stress were found to give the same initial and final slopes, which were thought to correspond to the focal-conic and flow aligned Grandjean texture viscosities, respectively.

The fact that a shear gradient is always present in a concentric cylinder viscometer led to a final set of experiments on a Brookfield cone and plate viscometer. This machine was not as sophisticated as the Contraves as

FIGURE 90. Shear stress against shear rate measured on the Contraves Low Shear viscometer for E9+35%CB15+2.5%Aerosil, at various temperatures in the isotropic and cholesteric phases.

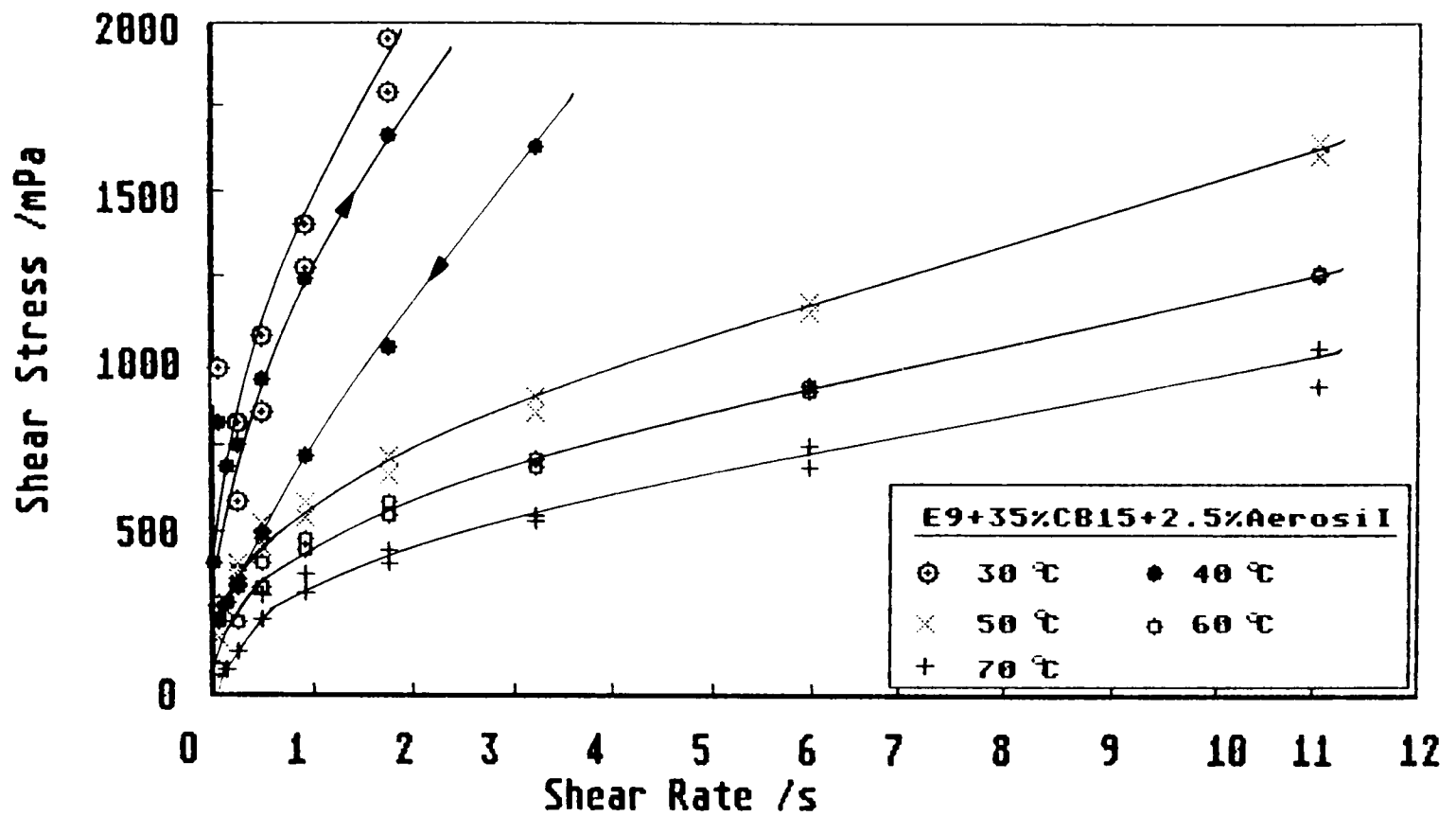
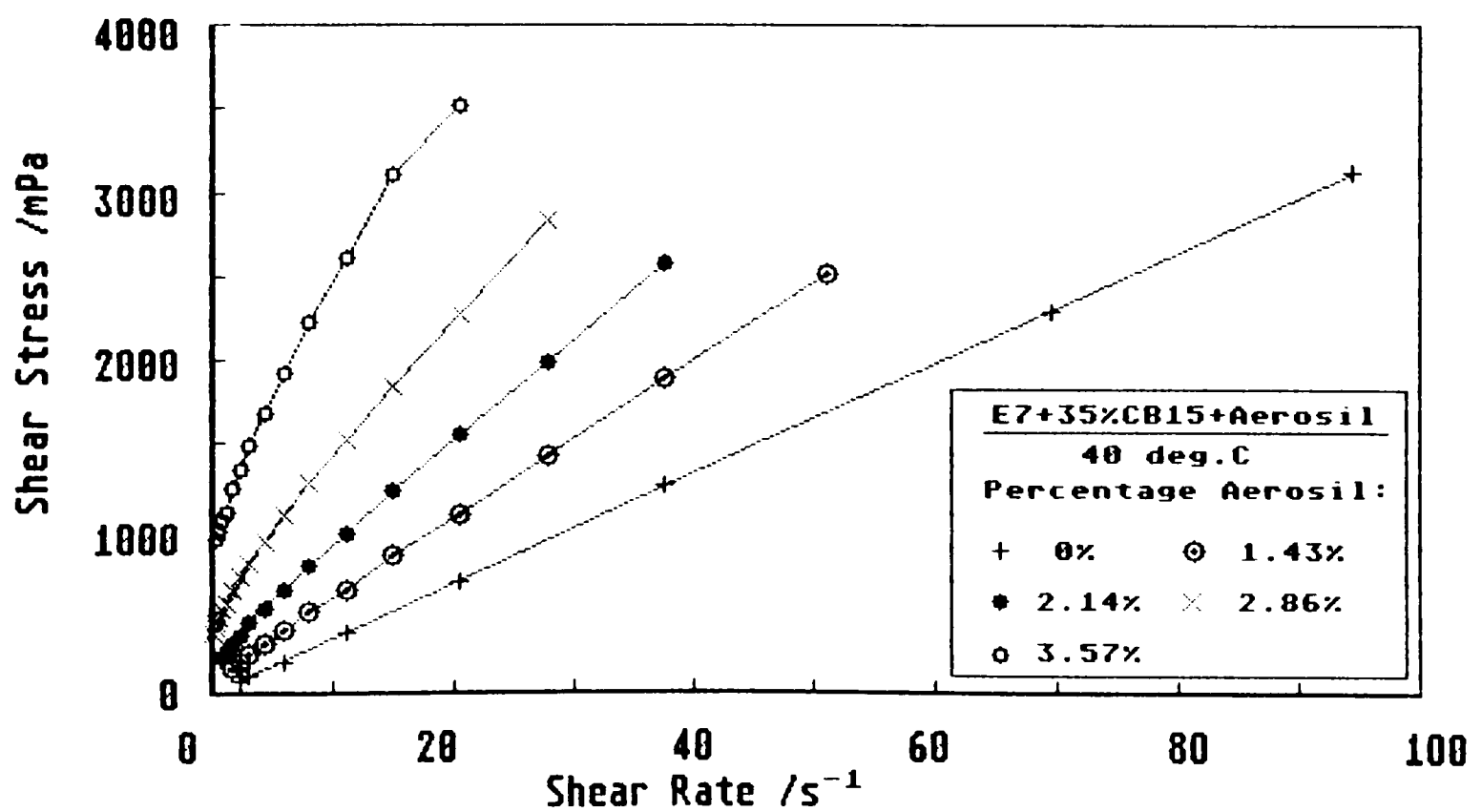
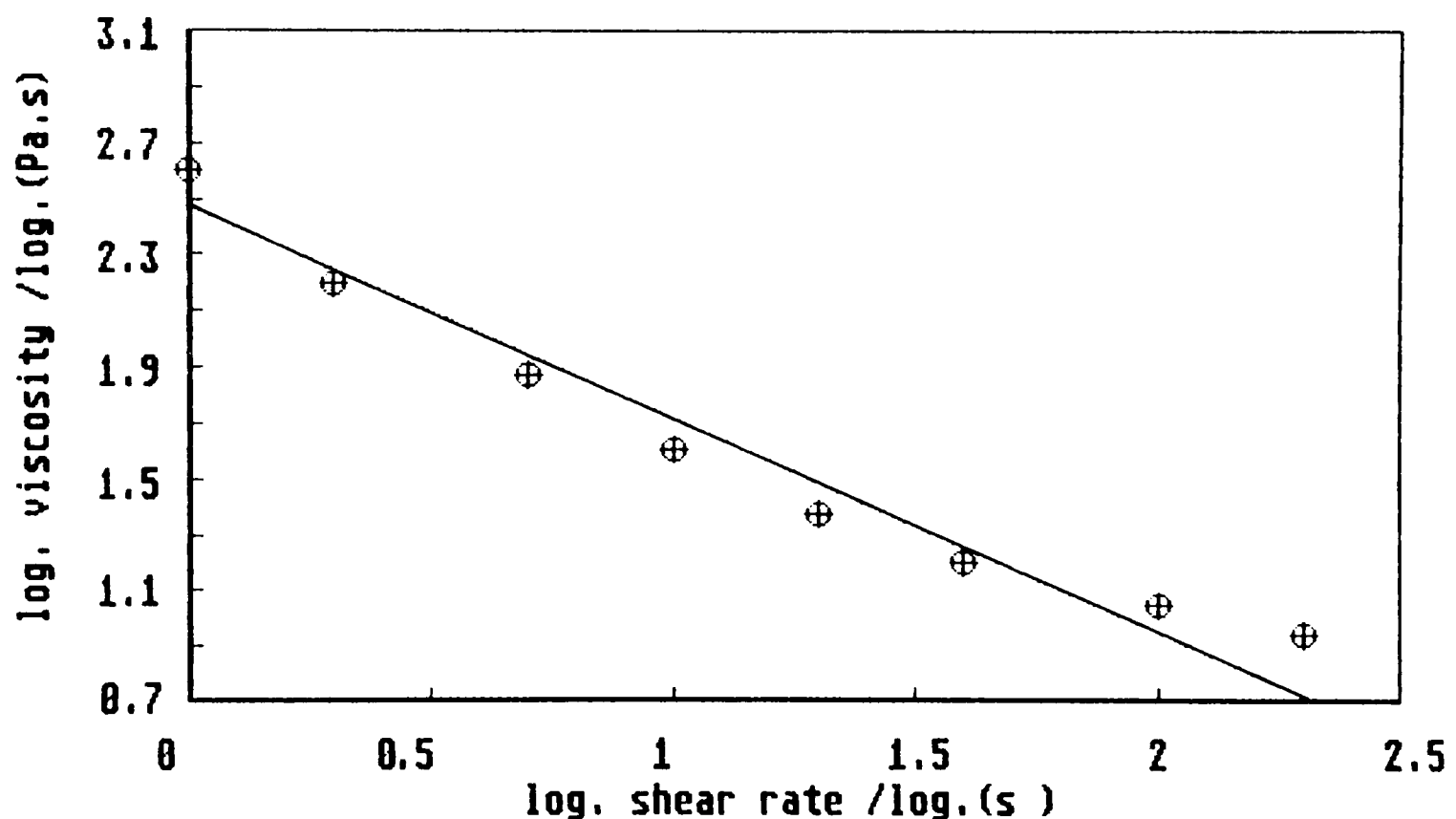


FIGURE 91. Effect of changing the weight percentage of aerosil added to E7+35%CB15 on the rheological properties at 40°C, measured in the Contraves Low Shear viscometer.



measurements had to be taken manually and scans of shear rate were impossible. It did, however, offer the great advantage of giving a constant shear rate over the whole sample. E7+35%CB15 was used, and measurements taken at 20°C over a range of shear rates. The results indicated higher viscosity values than the concentric cylinder viscometer at any shear rate, indicating that in the latter case measurements could only be trusted once full alignment had been produced, as otherwise the shear rate gradient gave misleading results. The Brookfield results are plotted on a log.-log. scale in figure 92.

FIGURE 92. log.(viscosity) verses log.(shear rate) for E7+35%CB15 at 20°C measured in the Brookfield cone and plate viscometer.



The fact that this plot is approximately linear indicates a power law dependence. A straight line can be fitted to fulfill the equation:

$$\eta = \frac{A}{\left(\frac{dv}{dx}\right)^n} \quad \begin{array}{l} dv/dx = \text{shear rate} \\ \tau = \text{shear stress} \end{array}$$

$$\text{but } \eta = \frac{\tau}{(dv/dx)} \quad \text{hence } \frac{(dv/dx)^n}{dv/dx} = \frac{A}{\tau}, \quad \text{therefore } dv/dx = \left(\frac{A}{\tau} \right)^{1/(n-1)}$$

the distance moved by the layer, $d = x \cdot \int \frac{dv}{dx} \cdot dt$, (x is the layer thickness)

$$\text{therefore } d = x \cdot \left(\frac{A}{\tau} \right)^{1/(n-1)} \int dt = x \cdot \left(\frac{A}{\tau} \right)^{1/(n-1)} \cdot t \quad - - (4.5) - -$$

Assuming that a visible colour appears after a constant displacement gradient, d/x , then a relationship between shear stress and time exists for the colour change boundary which can be checked against the wind tunnel results, e.g. figure 80.

A rough fit to figure 92 gives values for A and n of 2.97 and 0.76, respectively. A good value for the ratio d/x has not yet been found, as it cannot be assumed that colour only appears once good alignment has been achieved. In order to determine this ratio, which is the displacement gradient required to produce a discernable colour, two experiments were conducted.

4:3.1.2. Mechanical Displacement Experiments

The purpose of these tests was to determine independantly the displacement required to flow align the focal conic texture to produce colour.

(a). Radial Displacement.

Two flat glass plates, 150mm in length, were separated by a liquid crystal layer spaced with pieces of mylar. The upper plate was sheared radially over the lower plate about an axis 10mm from the end of the plates, see figure 93. The displacement was produced by a micrometer 160mm from the axis of rotation. Very precise displacements could be achieved as a function of radius.

The material between the plates was initially heated into the isotropic phase and allowed to cool into the focal conic texture. On application of a

FIGURE 93. Radially sheared plates used to determine the displacement required for flow alignment.

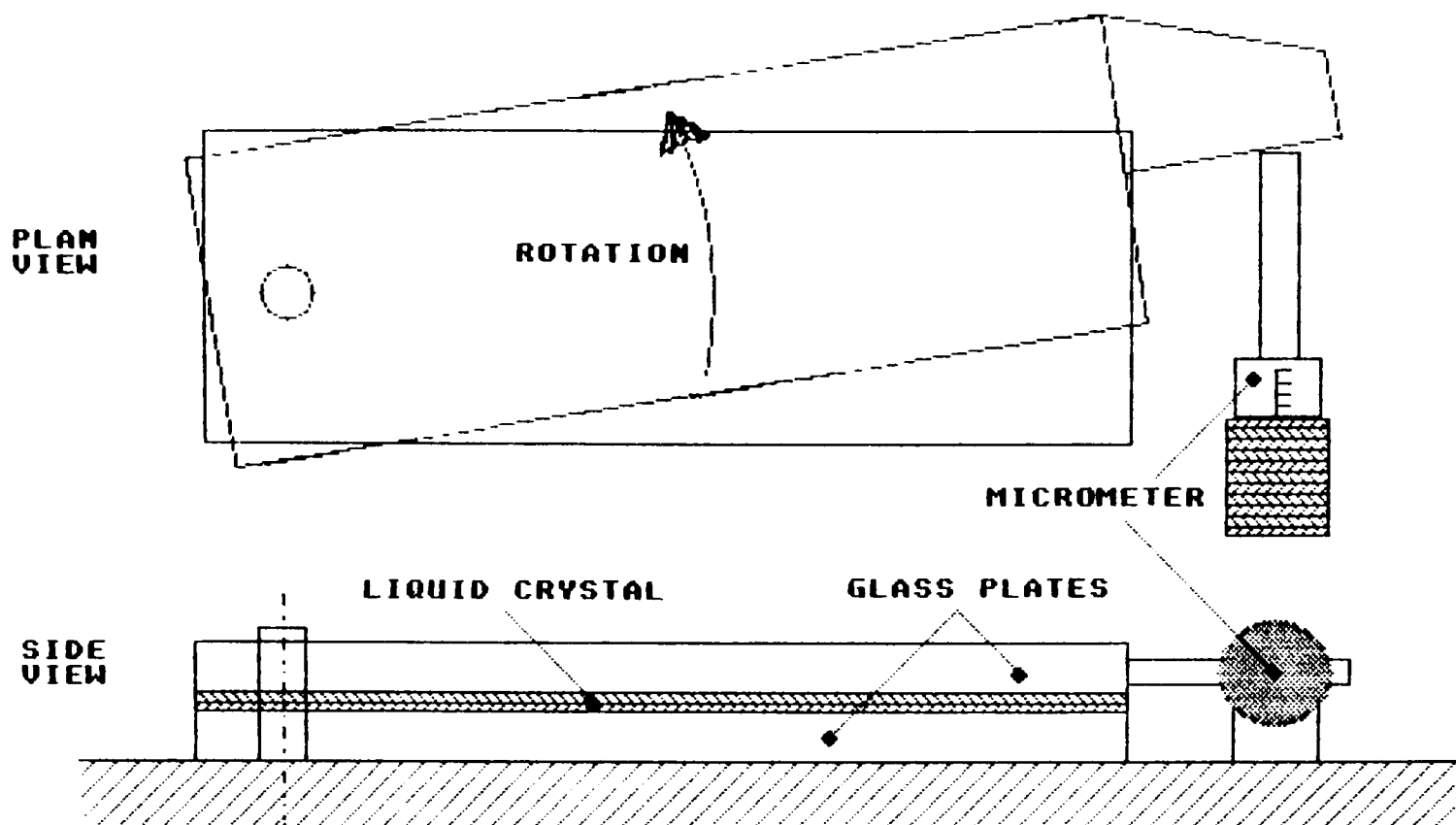
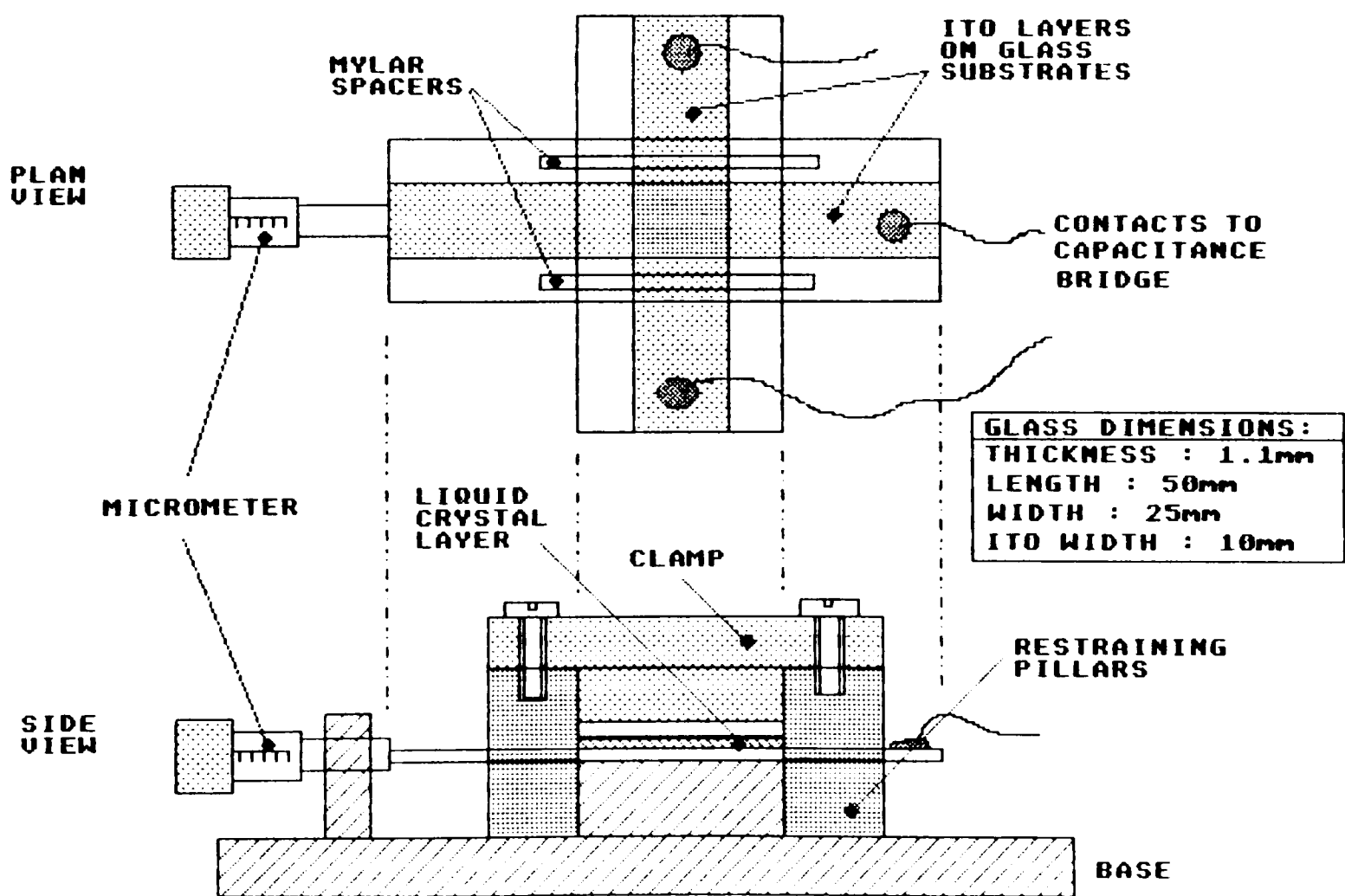


FIGURE 94. Apparatus used to measure the capacitance of a liquid crystal cell as one plate is displaced linearly over the other. The material may also be observed optically at the same time.



displacement the changes in the material were observed optically.

As expected alignment was produced at large radii (i.e. large displacement) and the alignment boundary moved inwards toward the axis of rotation as the displacement was increased. The alignment boundary was extremely diffuse, but an estimate of the required displacement for alignment was taken. This gave a value of $30\mu\text{m}(\pm 10)$ for a $10\mu\text{m}$ thick layer. i.e. a ratio of displacement to thickness (d/x) of about 3 ± 1 .

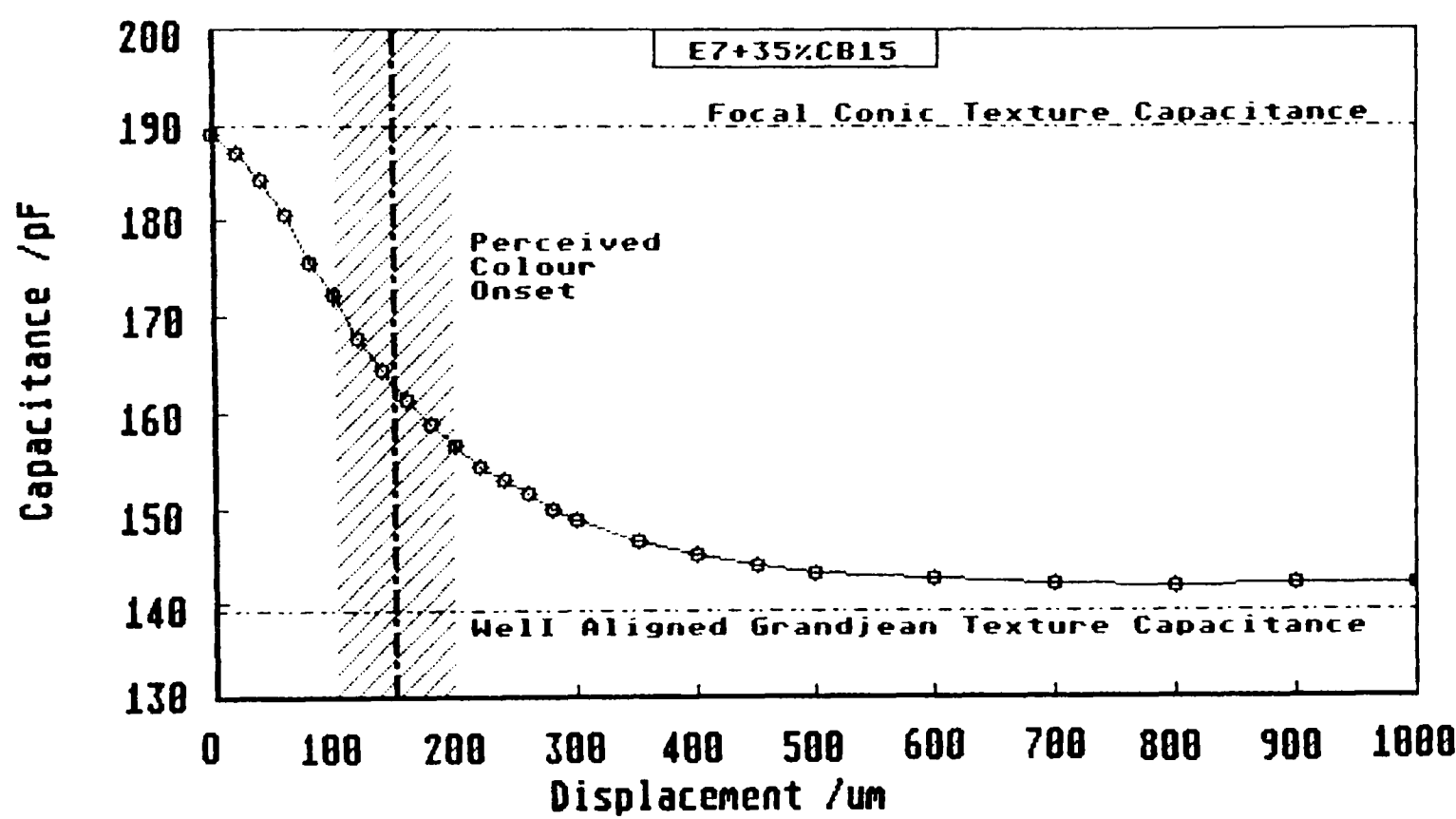
Owing to the diffuse nature of the boundary and the difficulty in specifying exactly where the required displacement occurred a second test was conducted.

(b). Linear Displacement.

Two flat glass slides were again separated by mylar spacers with liquid crystal material in the gap. The upper slide could be displaced linearly over the lower one using a micrometer. Both slides had a 10mm wide ITO layer along their inside surfaces, as shown in figure 94. Indium solder connections were made to the ITO, enabling the capacitance of the resulting cell to be measured. The cell area and thickness were constant and independent of the displacement of the upper slide. The upper slide was constrained to move only in one direction using perspex pillars on either side of the slide. A perspex block was fastened to the pillars, clamping the cell together, and maintaining a constant thickness. This block was made of clear perspex, so the material could be observed visually during the displacement, and these observations could be correlated to the capacitance measurements. The cell was initially heated into the isotropic phase and allowed to cool to the focal conic texture. The cell capacitance was measured as a function of displacement on an LCR bridge, operating at 1 kHz.

The results for one cell thickness are shown in figure 95. Tests were done on cell thicknesses ranging from $15\mu\text{m}$ to $60\mu\text{m}$. The alignment clearly improved continuously with displacement until the well aligned Grandjean

FIGURE 95. Results of the measurement of cell capacitance as a function of displacement, clearly indicating the progression of flow induced alignment. Cell thickness, 55um, Material. E7+35%CB15.



texture was reached. This material has a positive dielectric constant, so that $\epsilon_{\perp} < \epsilon_{\parallel}$, hence the capacitance dropped as the material aligned to orient the director perpendicular to the field direction.

It was observed by eye that the selective reflection occurred after a displacement of about 100 to 200 μm . Again the alignment did not occur suddenly at a particular displacement, but gradually. Results are tabulated below:

<u>Cell Thickness</u>	<u>Colour Onset</u>	<u>d_c/x</u>
55	130	2.4
50	160	3.3
38	100	2.6
20	80	4.0
μm	μm	

The value of the ratio d_c/x is consistent with the radial displacement results.

Analysis and Discussion

It appears that a small displacement gradient is required to flow align the material before any selective reflection colour becomes visible, but that a much larger displacement is required before the material becomes well aligned. Hence to enable a useable and quantitative system for surface shear stress measurement using liquid crystals each material must be 'calibrated' at the expected operating temperature. From this calibration curve, e.g. figures 75 and 76, the surface shear stress at any time may be estimated.

It has already been shown that these curves cannot be fitted with a

simple analysis relating shear stress at the texture change boundary to the elapsed time, (equation 4.4). A reasonable value of d/x is now available which may be used with equation (4.5), to take into account the variation of bulk viscosity with shear rate. This equation can be rearranged as:

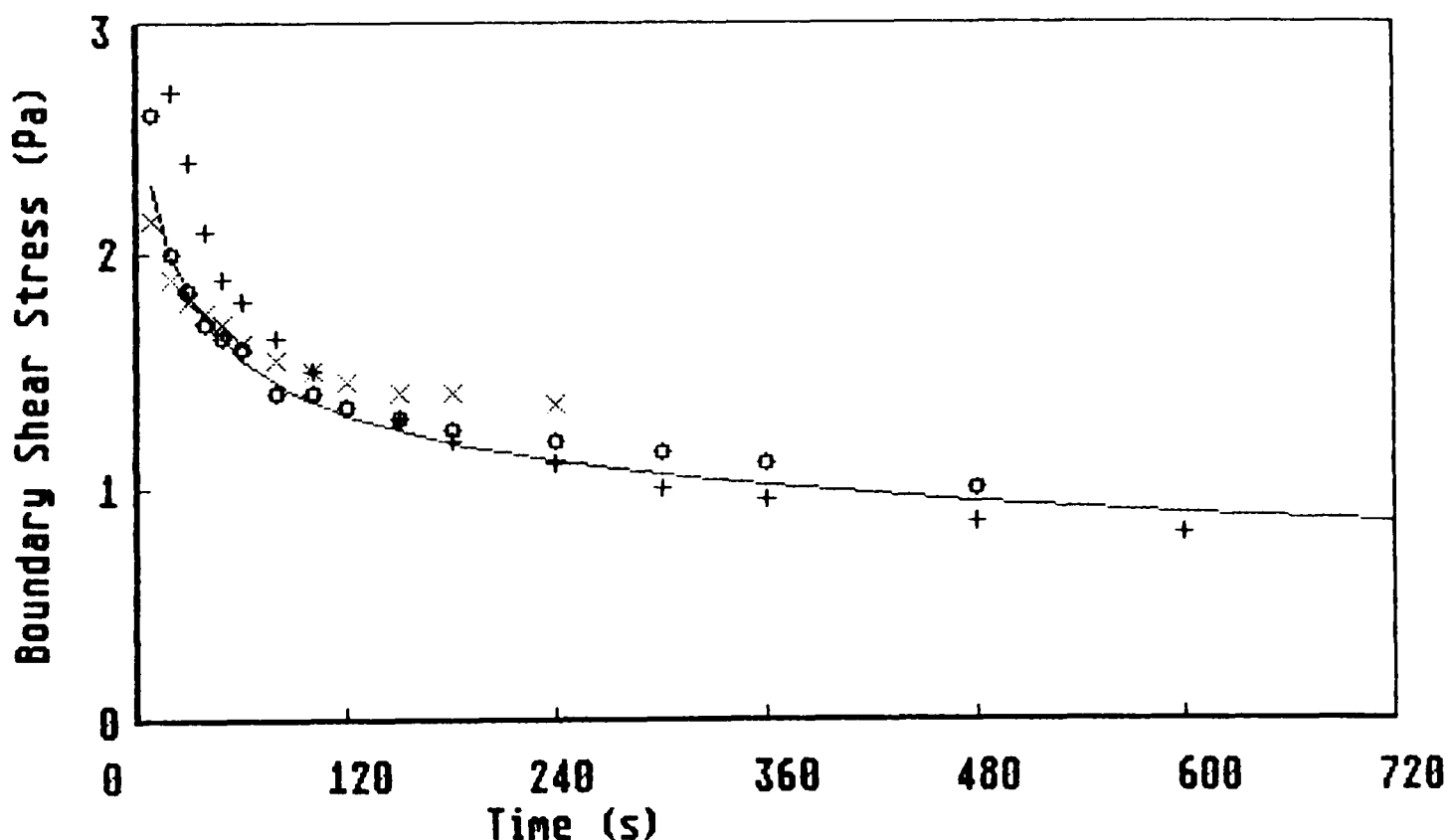
$$\tau = \left(\frac{A^{1/(n-1)} d \cdot t}{x} \right)^{n-1} \quad - - (4.6) - -$$

It is now possible to test if the fit to the Brookfield viscometer results corresponds to the wind tunnel results. Using the following values, obtained from figures 92 and 95:

$$A = 2.97, \quad n = 0.76, \quad d/x = 4$$

The fit to the wind tunnel results (figure 75) for E7+35%CB15 at 20°C is seen to be extremely good, (figure 96). This is a much better fit than assuming no variation of viscosity with shear rate, as with equation 4.4, and enables a quantitative approximation for the texture change boundary shear stress to be calculated at any time from a knowledge of the material viscosity properties.

FIGURE 96. Fit (full line) to the experimental wind tunnel points (for E7+35%CB15 at 20°C) using equation 4.6.



Although there is no doubt that further studies will be required to refine the technique, it forms the basis for a versatile system to measure surface shear stress which may find application in many areas where aerodynamic performance is of importance, or where shear stress/flow visualisation is required, for example aerodynamic testing of aircraft, cars, ships hulls, and so on.

4:3.1.3. Wind tunnel 'horse shoe vortex' studies

A series of tests were carried out to examine the surface shear stress field around a cylinder transverse to channel flow and to correlate these to the heat transfer patterns under the same conditions. These tests were relevant to blade cooling, which has already been studied extensively^{38,43}. Initial tests were conducted in a small blow down wind tunnel, and the results were sufficiently encouraging to progress to a larger wind tunnel, due to the well defined nature of the flow down the tunnel. For this test a large flat perspex plate was used (approx. 25cm by 40cm) with a 4cm diameter perspex cylinder on the plate.

The shear stress tests used E7+35%CB15 painted over the surface and heated into the isotropic phase with a hot air gun. With the plate vertical and using a flow speed of about 40ms^{-1} the method produced very clear and detailed flow patterns over the surface. The result was a mixture initially of shear indication and subsequently flow visualisation, as the liquid crystal material formed distinct rivulets which moved along the plate. The accuracy of the quantitative measurements was limited as the flow rate in the tunnel could only be increased relatively slowly to the operating conditions. Nevertheless, regions of high and low shear were clearly visible, and in particular, three areas of low shear were observed, in which the liquid crystal did not flow align at all, as shown in plates 14 and 15. There appeared to be a low shear region at the front of the pedestal, (a), which is in fact an area of

PLATE 14. Use of the shear induced texture change method to indicate the surface shear stress levels associated with the horse shoe vortex around a pedestal. The method clearly indicates very low shear areas both in front of and behind the pedestal, as well as providing excellent flow visualisation once the material has flow aligned. Material : E7+35%CB15, Flow from left to right at 40ms^{-1} . The plate was vertical during the test which accounts for the slight asymmetry in the pattern.

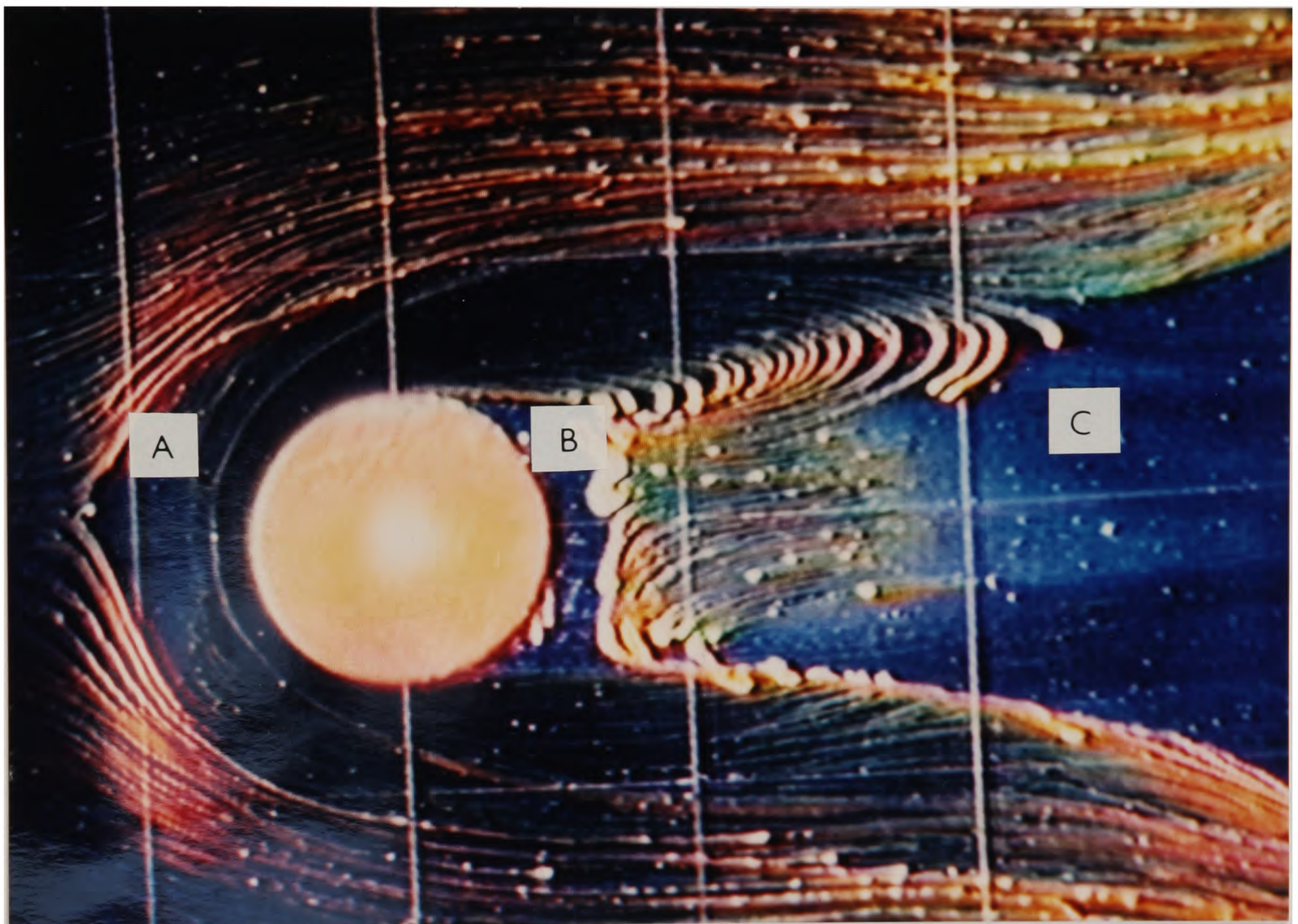
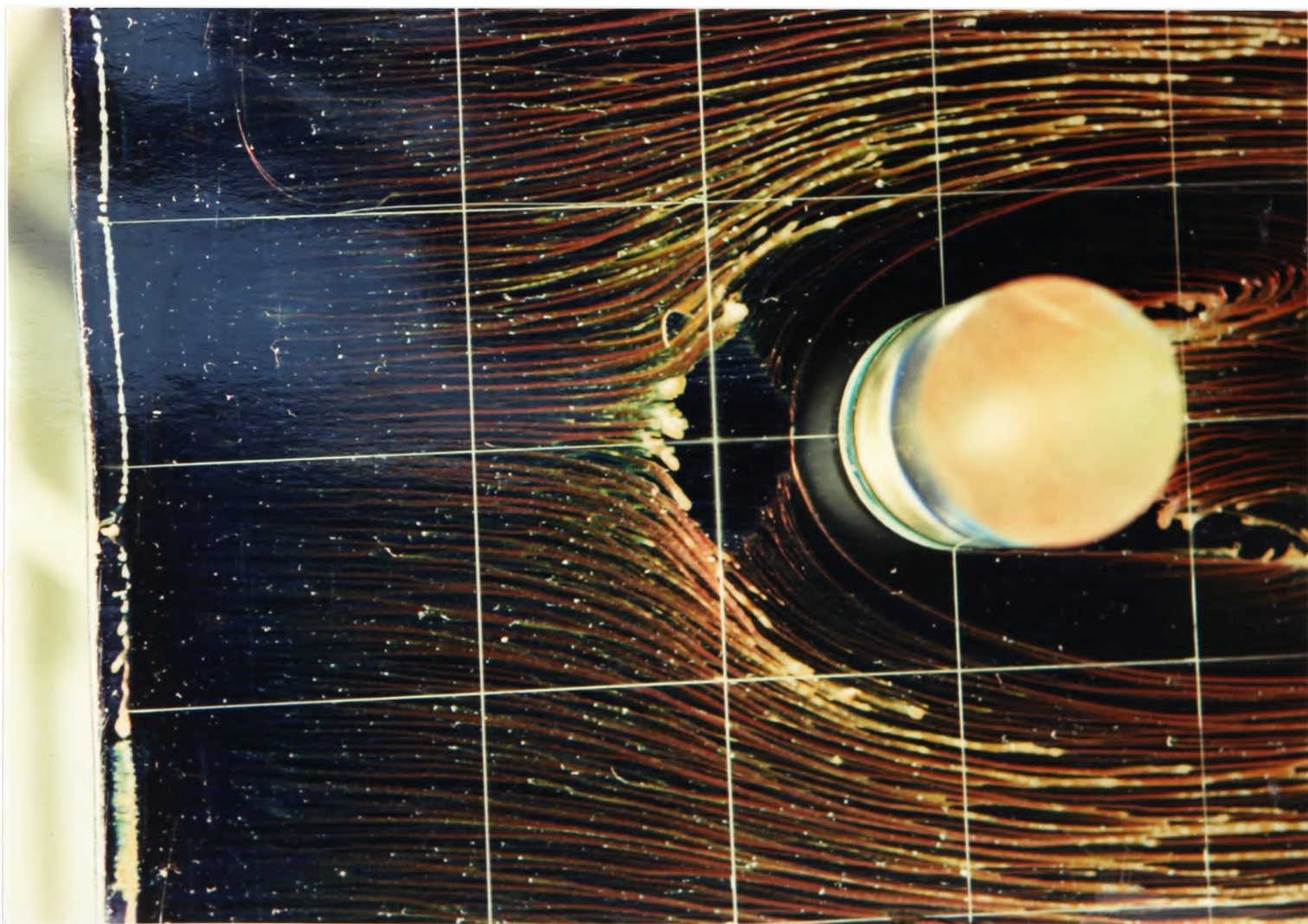
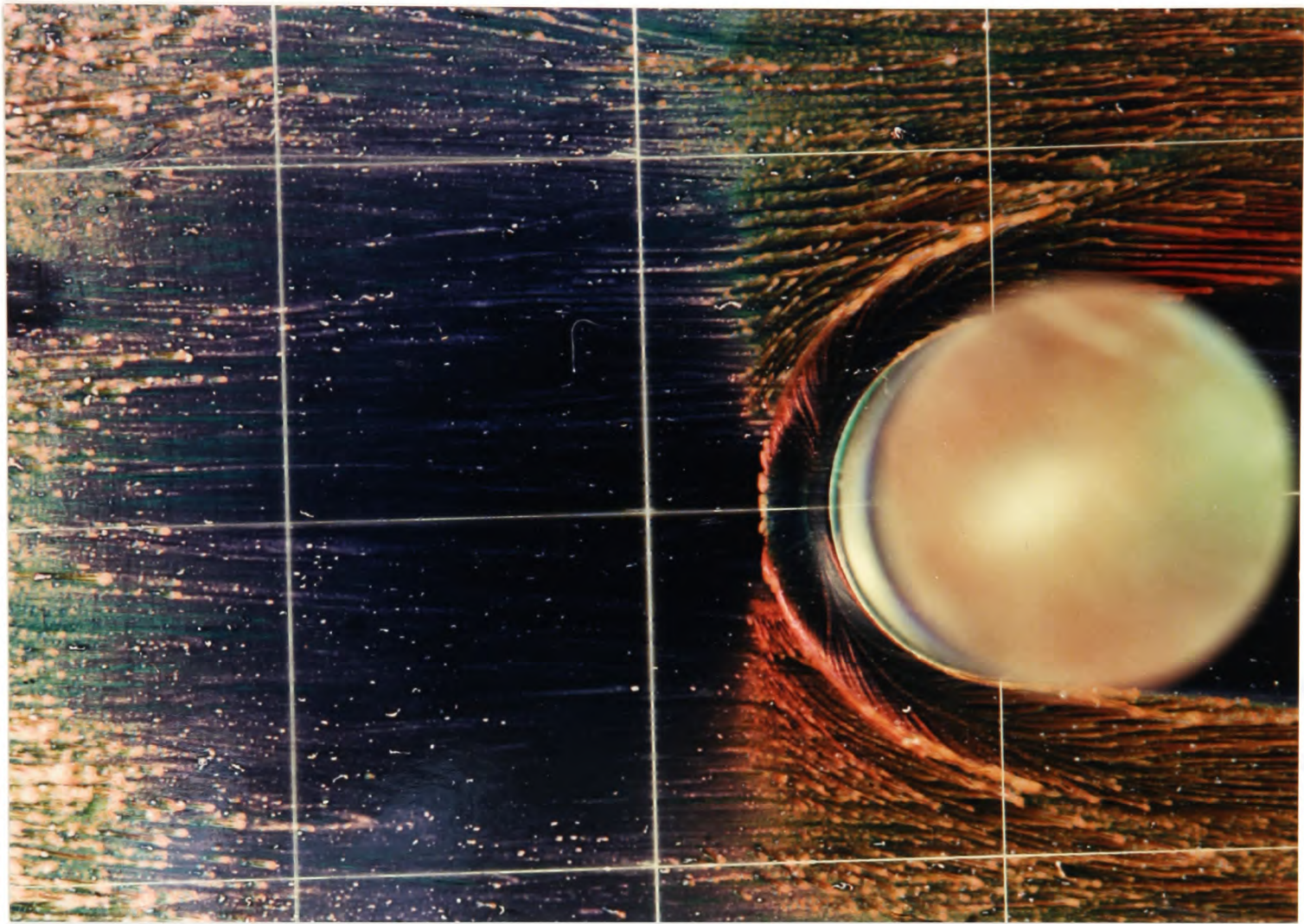


PLATE 15. Further pictures of the use of E7+35%CB15 to visualise surface shear stress levels around a pedestal. In these photographs the flow was applied for a set time. The flow was then stopped and the model photographed.



high heat transfer. The other low shear regions are immediately behind the pedestal, (b), and further downstream of the pedestal, (c).

Plate 16 provides a plot of results taken from the video, which indicates the shear rates over the model. The accuracy of the times given is limited, but an estimate of the shear rate at each time may be taken from figure 75 and 80 for E7+35%CB15 at 20°C. Hence the following values provide reasonable estimates:

<u>Time Line</u>	<u>Shear Stress/Pa</u>	<u>Time Line</u>	<u>Shear Stress/Pa</u>
30s	1.9	4mins	1.35
1min30s	1.6	5mins	1.3
3mins	1.45	8mins	1.1
		10mins	1.0

These results can be compared to theoretical estimates of the shear stress on the plate obtained from equations 4.2 and 4.3 for laminar and turbulent flow, reproduced below:

$$\text{laminar:} \quad \tau_o = (70415 \times v \times d)^{-1/2} \times 0.4425 \times v^2$$

$$\text{turbulent:} \quad \tau_o = (70415 \times v \times d)^{-1/5} \times 0.03806 \times v^2$$

Using a velocity of 40m/s and a distance, d, of 10cm results in the following values:

laminar flow shear stress : 1.33 Pa

turbulent flow shear stress : 4.95 Pa

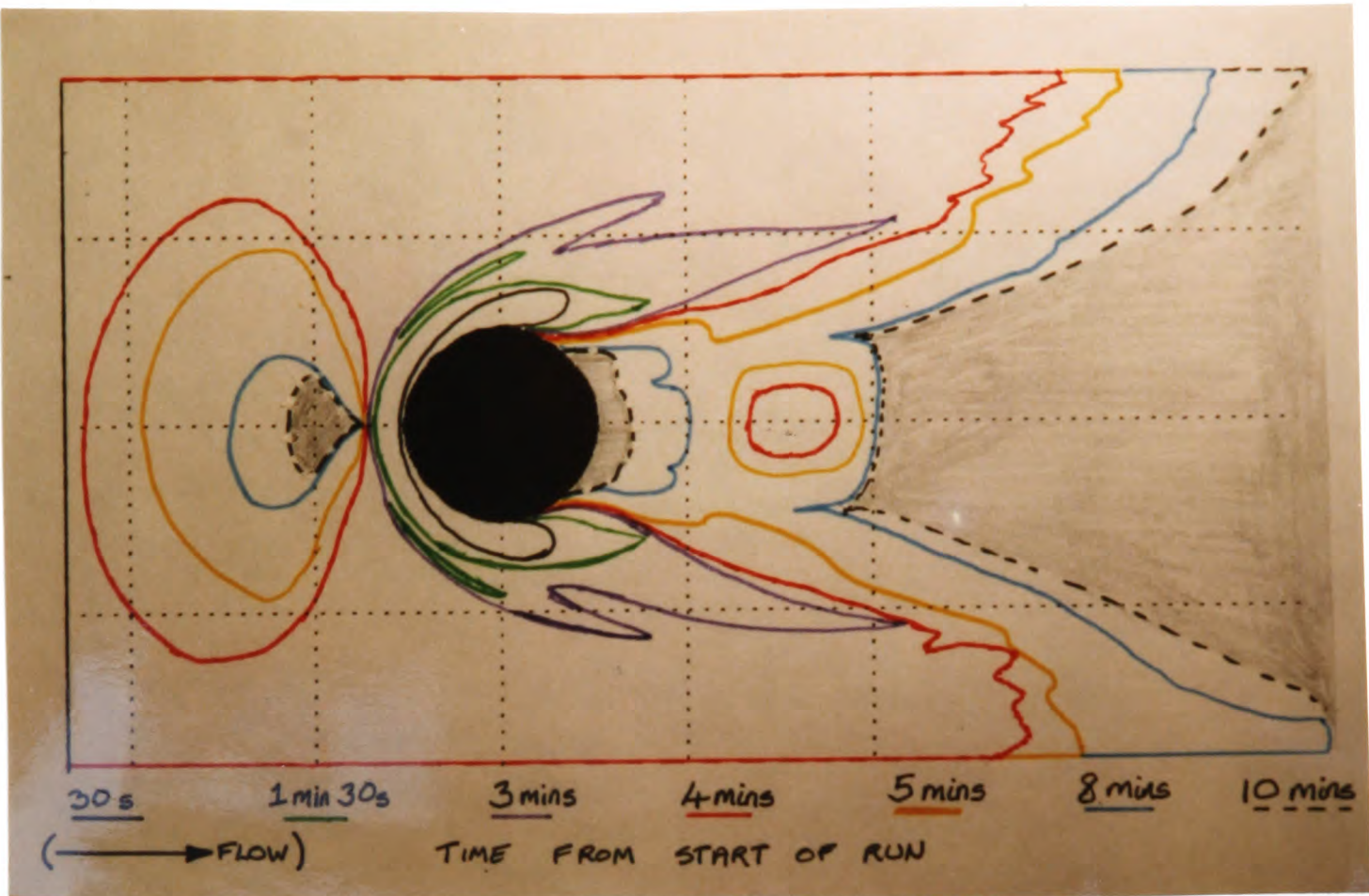
The actual flow in the wind tunnel is probably turbulent to some degree, but nevertheless, considering the inaccuracies mentioned above, these figures are in good agreement with the experimental method.

The heat transfer mapping was done by using a mixture of three encapsulated liquid crystals to provide a three event mixture. The perspex plate was pre-heated well above the highest of these and then allowed to cool under the test conditions. This allowed two clear isotherms to be seen as the

PLATE 16.(a). Indication of the shear stress levels which develop around a pedestal. These lines indicate the position of the texture change boundary after certain times during the flow. Hence the time may be calibrated to a shear stress, as attempted in the text. E7+35%CB15 at room temperature. The flow conditions are identical to those described for plate 14.

(b). Final flow visualisation around a pedestal using E7+35%CB15 after removal of flow.

a.



b.

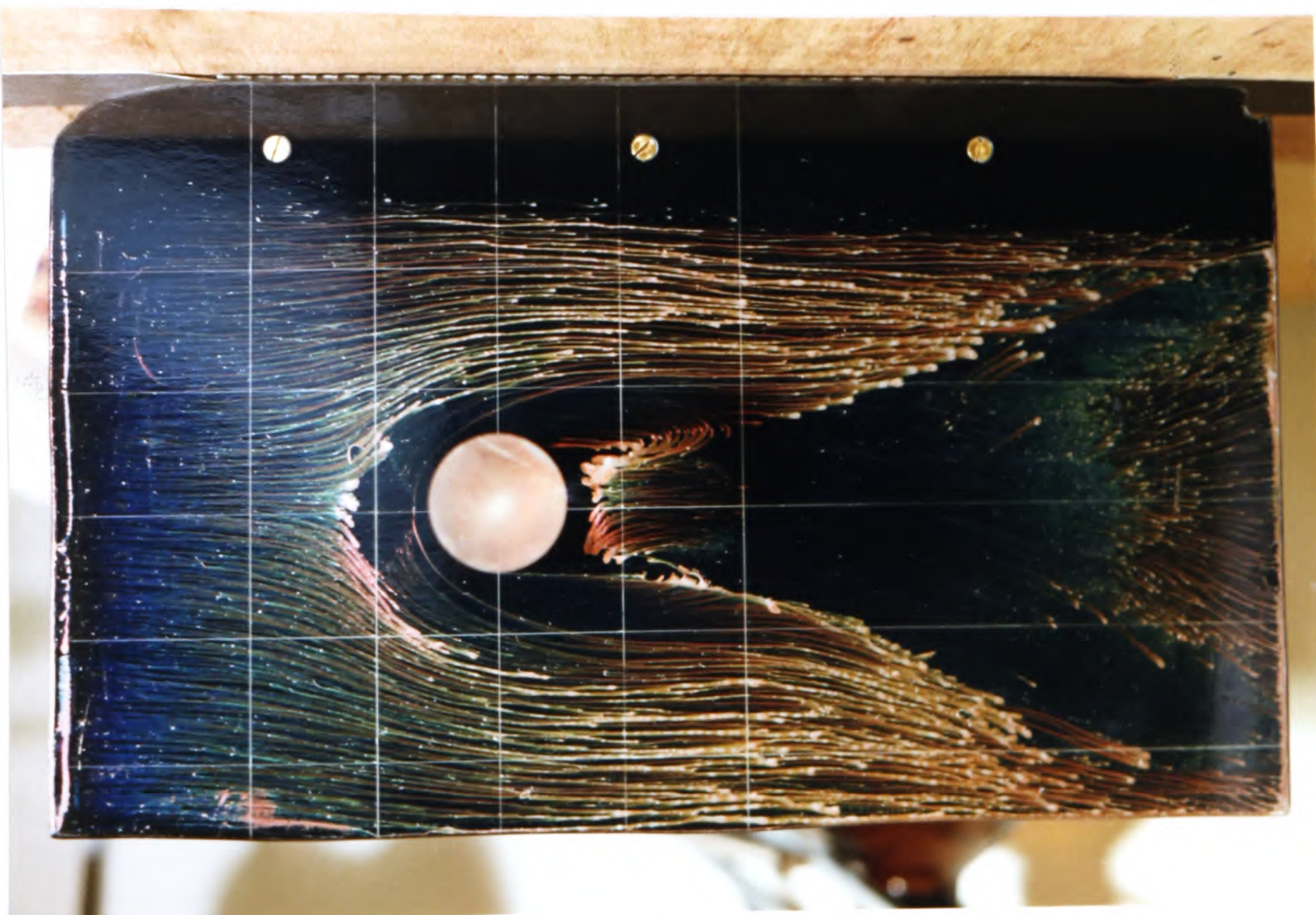
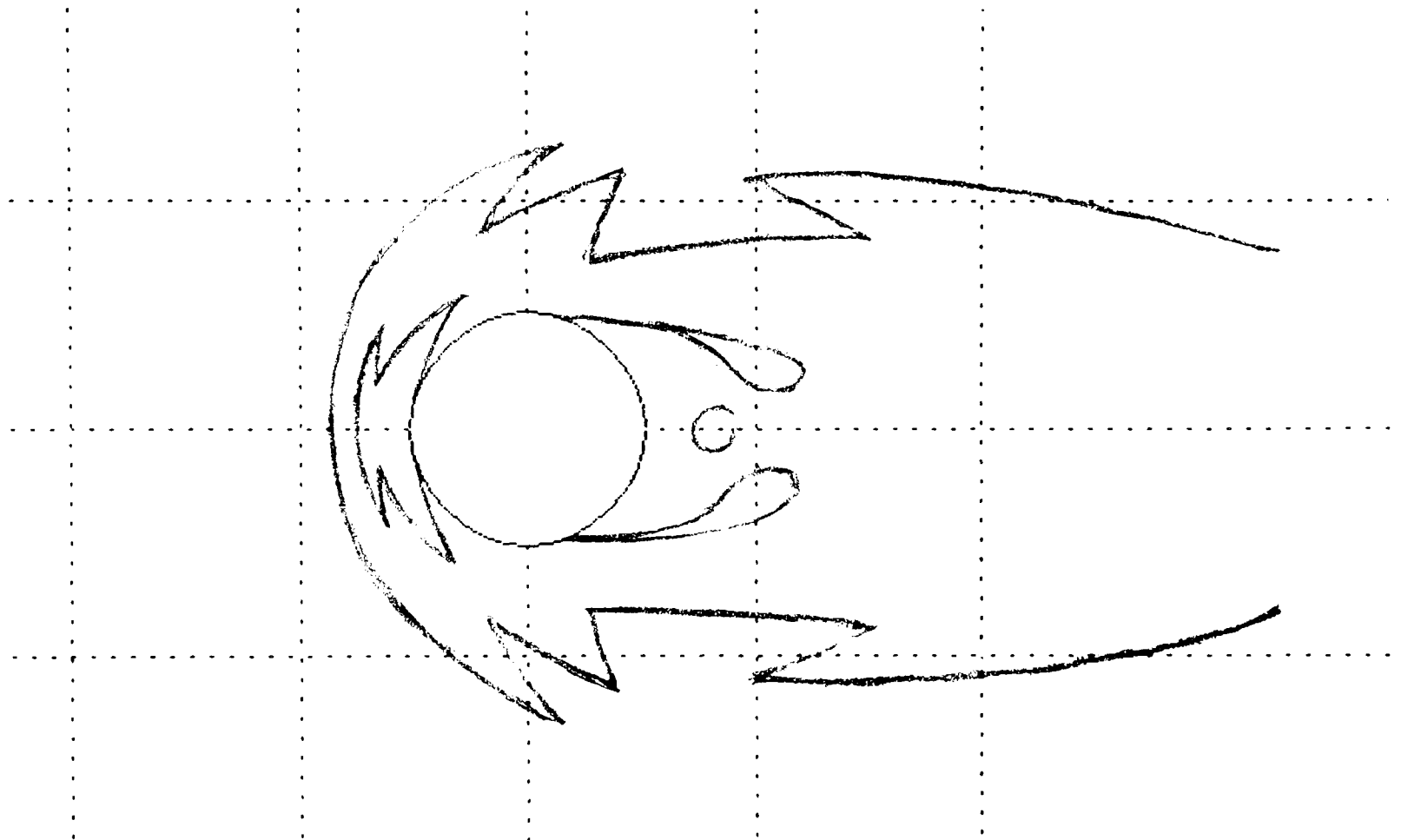


FIGURE 97. Pattern of isotherms around a pedestal under the same flow conditions as plate 16.



model cooled (the highest temperature isotherm passed too quickly to be used). These are sketched in figure 97, and may be compared with plate 16. Video recordings of these tests revealed an unexpectedly high volume of complex behaviour, and a detailed analysis of this is the subject of ongoing research beyond the scope of the present work.

4:3.2. Nematic Effect

A short series of tests were conducted to assess the possible use of an aligned nematic surface layer for surface shear stress measurement. An initially well aligned nematic layer would become disordered under the action of a shear and this would produce a change in the birefringence colours if the layer were viewed between crossed polarisers. The method requires the production of a well aligned, even surface layer of neat nematic material. Celgard cannot be used in this case, as it protects the absorbed material from shear stress, so the nematic material was applied in neat form to the surface.

The material was aligned homeotropically (i.e. the director perpendicular to the surface) on a perspex surface which had been treated with lecithin. This is a clear chemical which promotes homeotropic alignment of liquid crystal films. See figure 98. Ideally the layer would be colourless if viewed in transmission between crossed polarisers with no shear force present. If shear were applied the director should tilt to produce birefringence colours. Increasing the shear stress should increase the director tilt. However, in practice a number of problems intervened.

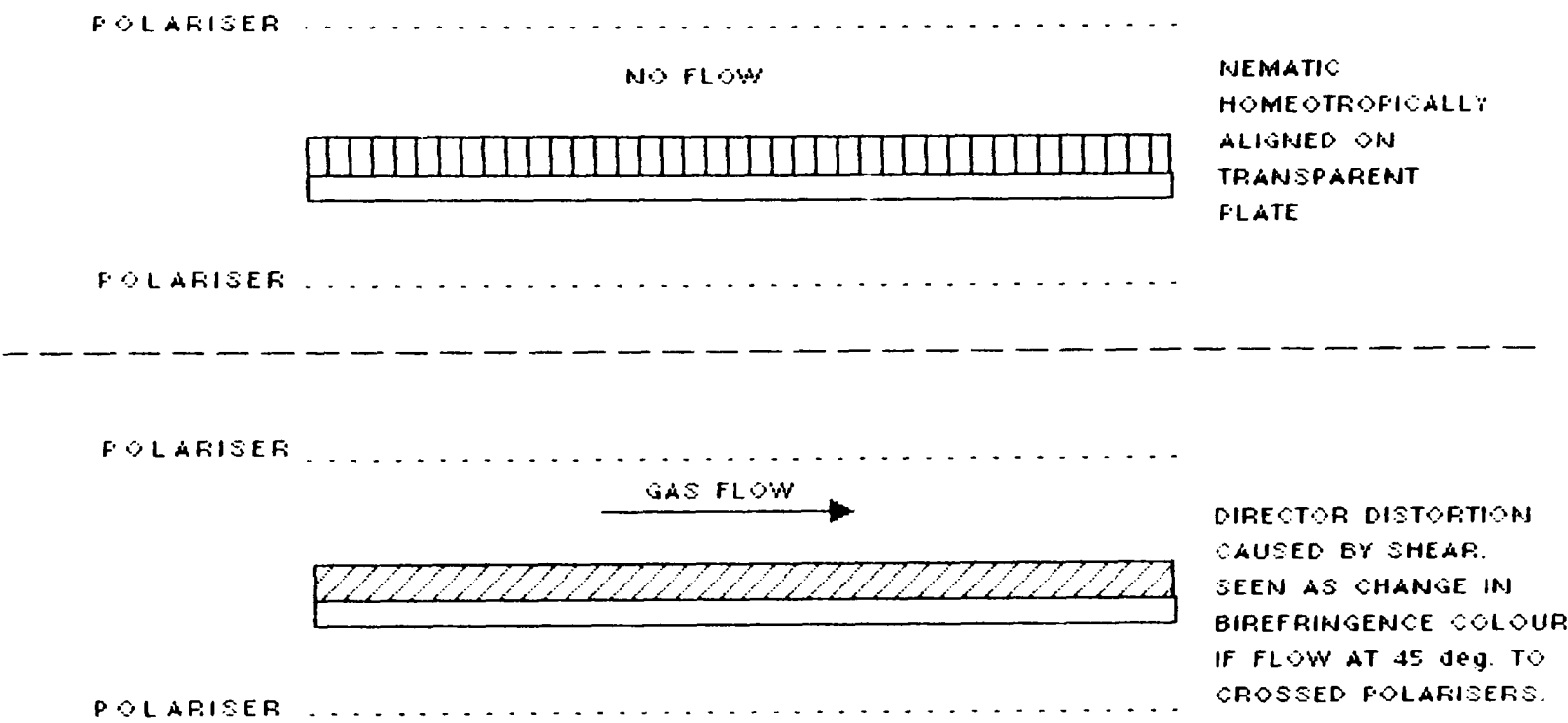
Firstly, it was almost impossible to achieve a layer of sufficient thickness uniformity with good initial alignment, to produce even birefringence colours, so the layer was multi-coloured with no shear present.

Secondly, the layer thickness changed as the shear was applied, due to the nematic material flowing over the surface.

Nevertheless, flow patterns were apparent, so it was decided to

pursue the method further. A mixture of pleochroic dyes was added to the nematic which absorb light over the visible range of the spectrum, enabling operation with only one polariser. The transition moment of the dyes align cooperatively with the nematic director and the layer behaves as a low efficiency polariser.

FIGURE 98. Use of homeotropically aligned nematic layer to visualise shear stress levels from changing birefringence colours.



If the layer is homeotropically aligned, rather than examining colour changes as the director tilts, there should be a variation in the transmitted intensity with shear, as the absorption of the dye increases as it is tilted over. This variation was observed in practice, but good calibration could not be achieved as the material still tended to flow from the surface. Unfortunately a shortage of time precluded further work on these effects, but it has provided useful groundwork for an ongoing research programme.

4:3.3. Summary and Conclusions

The method of most interest at present is therefore the texture

change effect applied to the measurement of surface shear stress. The use of liquid crystals in shear stress studies represents a significant technical advance and much new information can be obtained using the technique, which is relatively straightforward to use, compared to many other, more involved shear stress measurement methods. Rolls Royce and Messerschmidt have shown an interest in this work and in flight tests are scheduled to be conducted at RAE Farnborough to assess it. Separation detection at the front of a plate has recently been successfully carried out at Oxford.

This method has been filed for a patent by the MOD. Patent application number 8804176, inventors P. Bonnett, T.V. Jones and D.G. McDonnell.

5. PRESSURE

5.1. PRESSURE

This chapter will investigate the sensitivity of liquid crystals to pressure. This has a possible application in the measurement of pressure, which is another important parameter in aerodynamic design.

Pressure is currently measured accurately only at specific points and as with the other parameters discussed above it would be preferable to obtain entire surface maps by a non intrusive means. Liquid crystals are known to be sensitive to pressure^{17,18} so the present work was therefore instigated to investigate and develop their use for pressure mapping.

The pressure in a stationary fluid is a scalar quantity dependent only on the density of the fluid, gravity and the depth. If the fluid is flowing, however, the total pressure is a vector quantity, since a pressure gradient must exist in the direction of flow. Moreover, a certain pressure change is also needed to overcome viscous resistance to the flow. The geometry of the channel therefore becomes important as obstacles may produce complex pressure variations. Thus, for example aerofoil shapes are designed to produce a low pressure above and a high pressure below the aerofoil.

Pressure measurement is important in assessing the performance of such aerodynamic shapes and is also commonly used to measure flow velocity, as mentioned in the last chapter. In laminar flow conditions the measured pressure should give a steady value, but in turbulent conditions a fluctuating component is added to this. It is often only necessary to measure the time average of this variation, but devices with a high frequency response may be used to measure this component and so determine the degree of turbulence.

5.1.1. Current Methods for Pressure Measurement

The pressure measurement device should ideally not disturb the flow at all and the simplest way of doing this is to use a device flush with the test surface. A small tube, with the free end connected to a liquid manometer, and its axis normal to the surface may be used. This is called a static pressure tube as it only measures the force perpendicular to the surface. Other devices with faster responses may also be used on the ends of static tubes. For instance, piezoelectric gauges, where the deformation of the crystal by the pressure produces an electrical output, or capacitance gauges, where a diaphragm is subjected to the pressure which varies the gap in a capacitor. Alternatively, a strain gauge may be attached to a flexible diaphragm and the change in gauge resistance calibrated against pressure.

Acoustic microphones may also be mounted flush with the surface. These have a sufficiently fast response to enable the measurement of turbulent pressure fluctuations.

Stagnation pressure may be measured with a Pitot tube in the flow, (see figure 52). The stagnation pressure hole should point directly into the flow. This measures the total pressure in the fluid, and is generally used in the flow rather than at the surface.

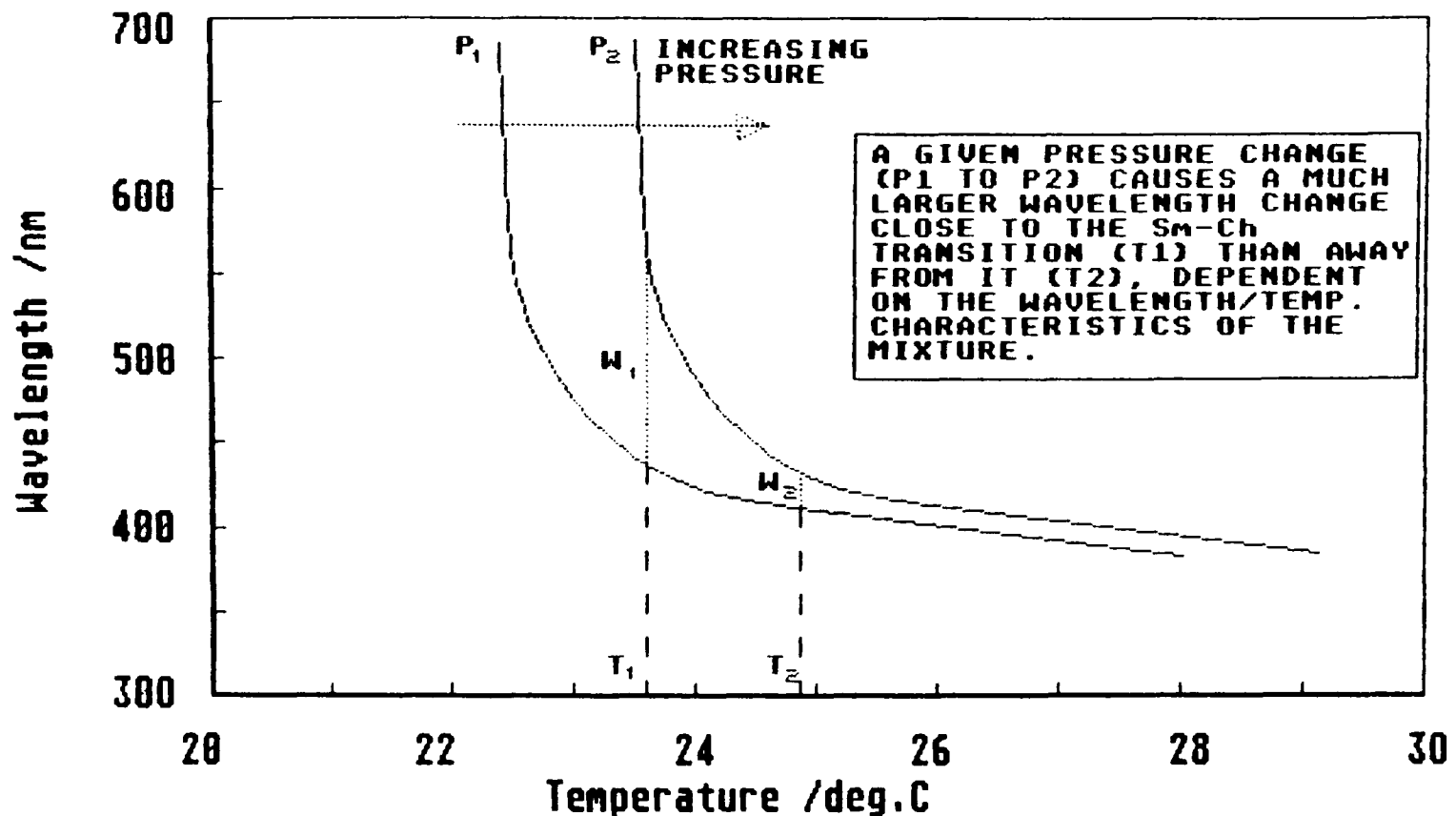
5:2. THE USE OF LIQUID CRYSTALS FOR PRESSURE MEASUREMENT

As described in the introduction, liquid crystals are affected by pressure and the effects may be observed as colour changes resulting from transition temperature changes in the material.

The main problems to be overcome in producing a practical surface pressure mapping system are to protect the liquid crystal from other influences, while at the same time making the material as sensitive to pressure changes as possible.

Previous workers^{17,18,19} have indicated that liquid crystals are not especially sensitive to pressure changes. Colour shifts to the red have been measured on application of pressure¹⁷ to some cholesteryl ester systems. This was explained as being a result of the viscosity increasing with pressure, causing damping of the molecular vibrations and thereby a lengthening of the pitch. The author is of the opinion, however, that for chiral nematics the dominant effect is the change in the transition temperatures as a result of the pressure change. Transition temperatures rise as the pressure increases since the energy necessary to undergo a phase transition increases, and this would cause a shift to the red of a material displaying normal colour/temperature characteristics. Typical changes are only of the order of 30° per kbar. This is not sensitive enough for accurate pressure measurements, but the colour change with pressure will become increasingly more pronounced as the smectic to cholesteric transition temperature is approached due to the increased temperature dependence of the colour in this region. Hence if colour changes are measured to determine pressure the material temperature must be very accurately controlled. See figure 99.

FIGURE 99. Schematic presentation of how selective reflection from a chiral nematic might shift with pressure.



In order to protect the liquid crystal from the effects of shear and contamination the initial studies were aimed at using the cholesteric liquid crystal material encapsulated in a soft binder which would deform and transmit the external pressure to the liquid crystal.

It was intended to study chiral nematic based materials, as studies using cholesteryl esters had shown them to be fairly pressure insensitive, whereas it was hoped that synthetic materials could be tuned to maximum sensitivity. A pressure cell was constructed for the tests and is described below.

5:2.1. Pressure Cell Design

A pressure cell was required which would enable pressures up to 2000psi to be applied to a liquid crystal sample. The cell also must enable transition temperature measurements to be made at different pressures, and

also if possible selective reflection measurements to be made, hence the actual cell must be transparent.

A cell was designed and built to meet these requirements, and is shown schematically in figure 100. The liquid crystal is contained in part A, which is a perspex block with a small test area 3mm in diameter and 1mm deep, designed to withstand 2000psi within the test area. A small perspex lid was bonded with superglue over the test area to seal it. This joint was the weakest part of the system, but was capable of withstanding 2000psi (see stress analysis in next section). The cell was only 3mm thick to enable it to fit inside a Mettler FPS2 furnace and controller for use with a polarising microscope. The perspex faces of the cell next to the test area had to be polished smooth so as not to cause any disturbance to the light passing through the cell, when viewed through the microscope.

In tests the 1mm test area thickness proved too great to enable phase identification from microscopic observations of textures. Therefore new indented lids were constructed to reduce the sample thickness to 0.5mm.

The sample holder, part A, was connected to the brass pressure block, part B, by two brass supports, 3mm thick. These supports held the cell in place in the temperature controller. A 1mm diameter capillary tube ran from the liquid crystal reservoir in part B into the test area in part A. Pressure was applied to Part B through a pressure line connected to its lid, part C. This line only needed to reach 100psi at most. This pressure acted on a piston, part D, inside the pressure block. This piston amplified the pressure by twenty times due to a factor of twenty reduction in area in the piston at the liquid crystal end.

Technical drawings of all the parts mentioned are shown in figure 98(a) to (d), though the operation of the device is probably much better conveyed by the sketch in figure 101.

FIGURE 100.(a). Diagram of the pressure cell design. Part A is the clear perspex cell in which the material is pressurised and viewed through the microscope.

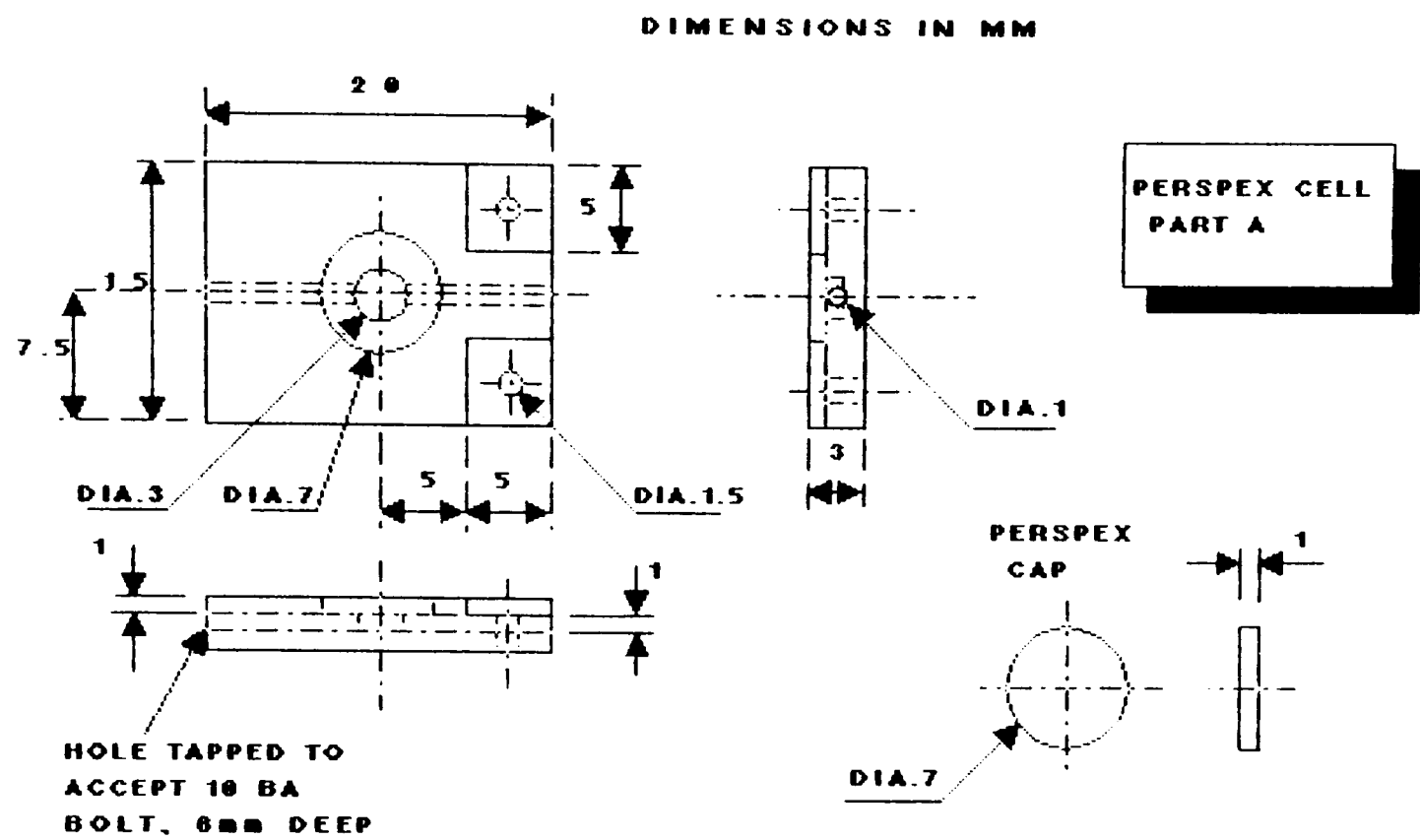


FIGURE 100.(b). Pressure cell design, part B, the brass block used to transmit and amplify the applied pressure.

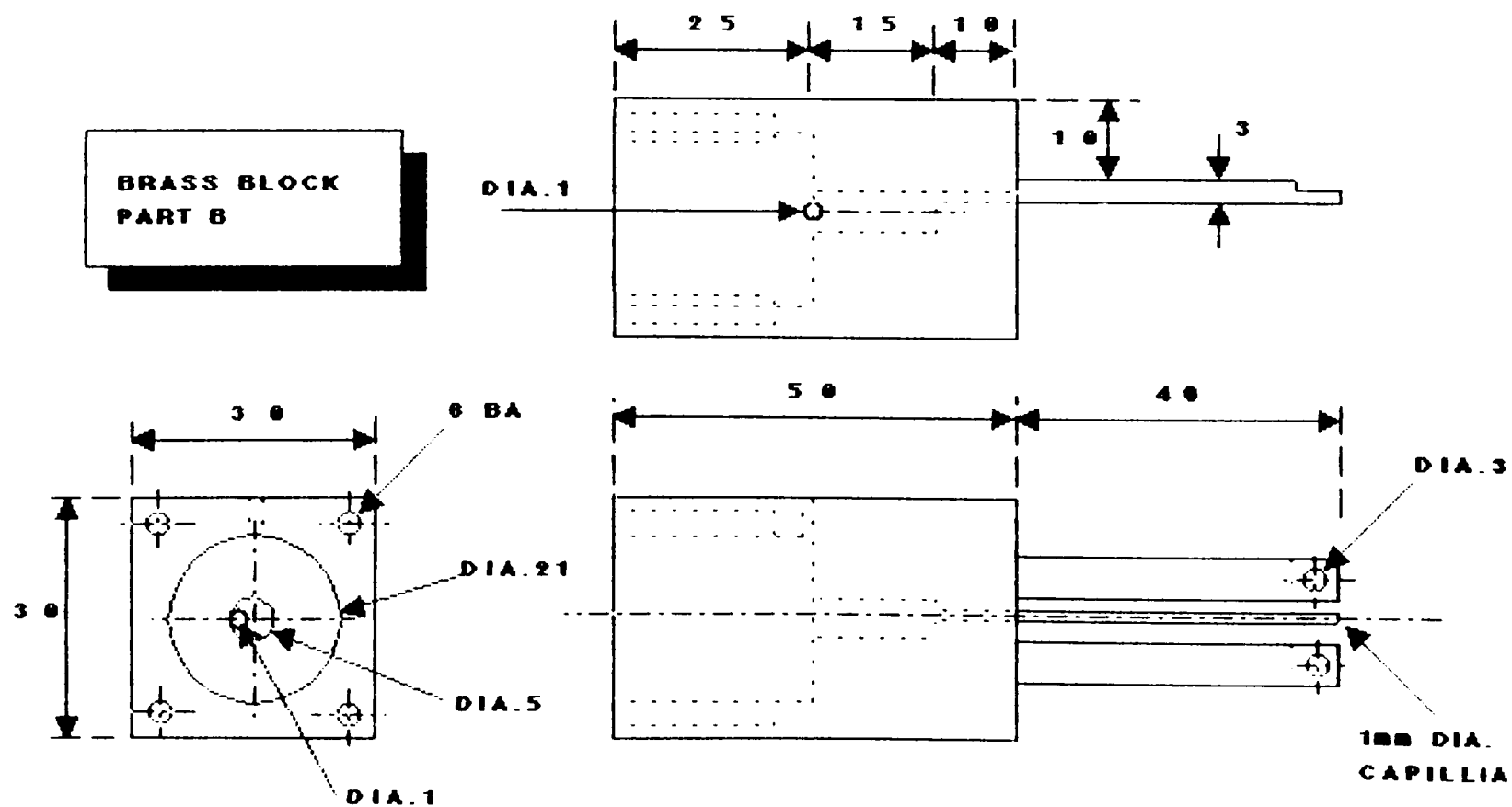


FIGURE 100.(c). Pressure cell design. Brass cap fitted to the top of part B, to which the external pressure line is connected.

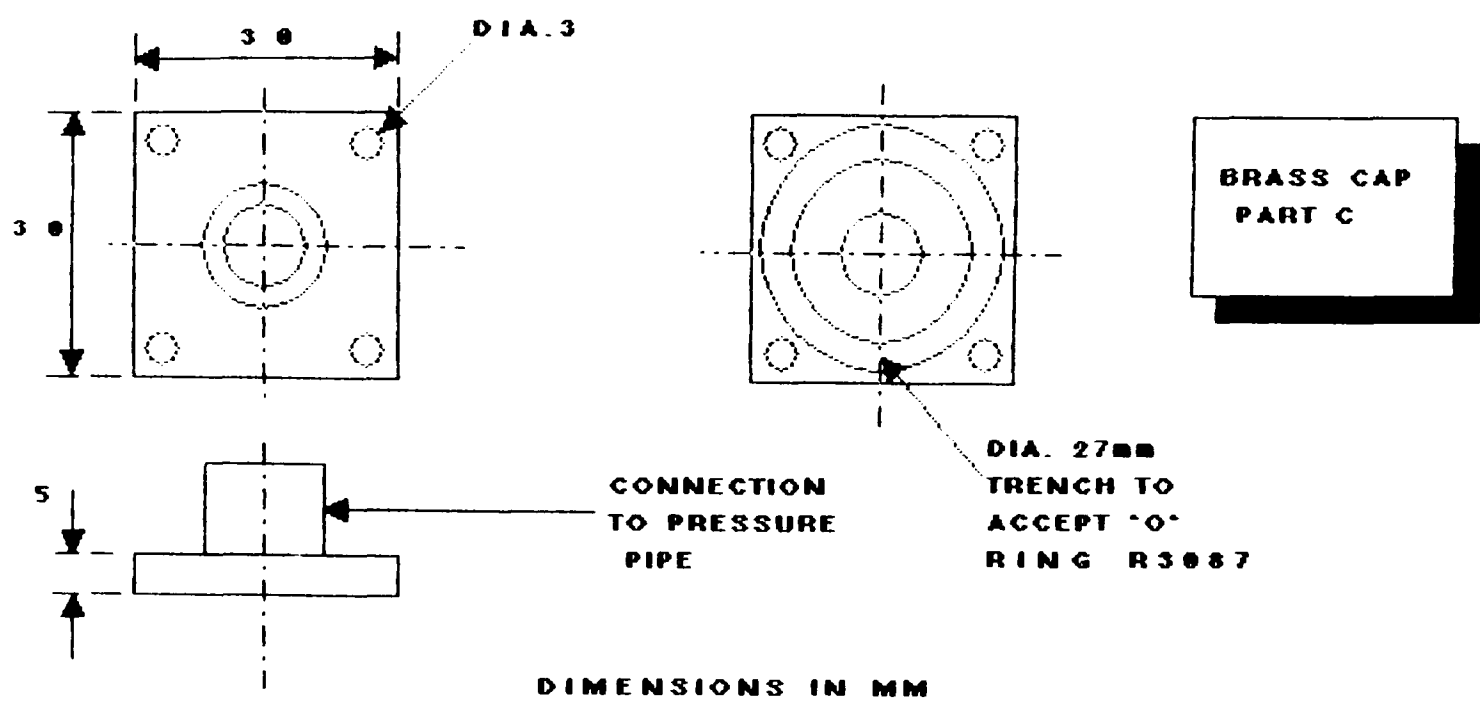


FIGURE 100.(d). Pressure cell design. Brass piston inserted into part B, used to amplify the applied pressure.

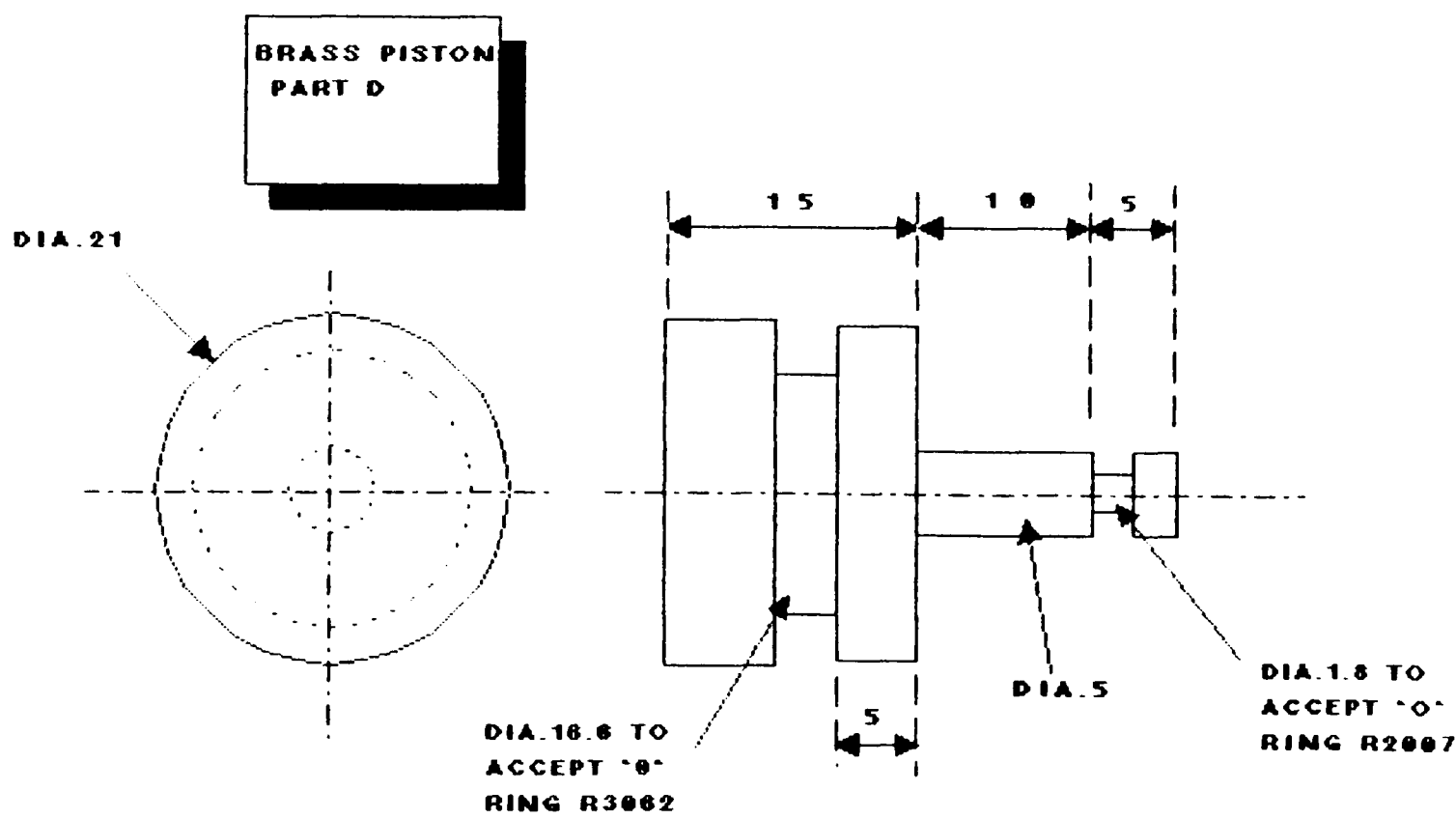
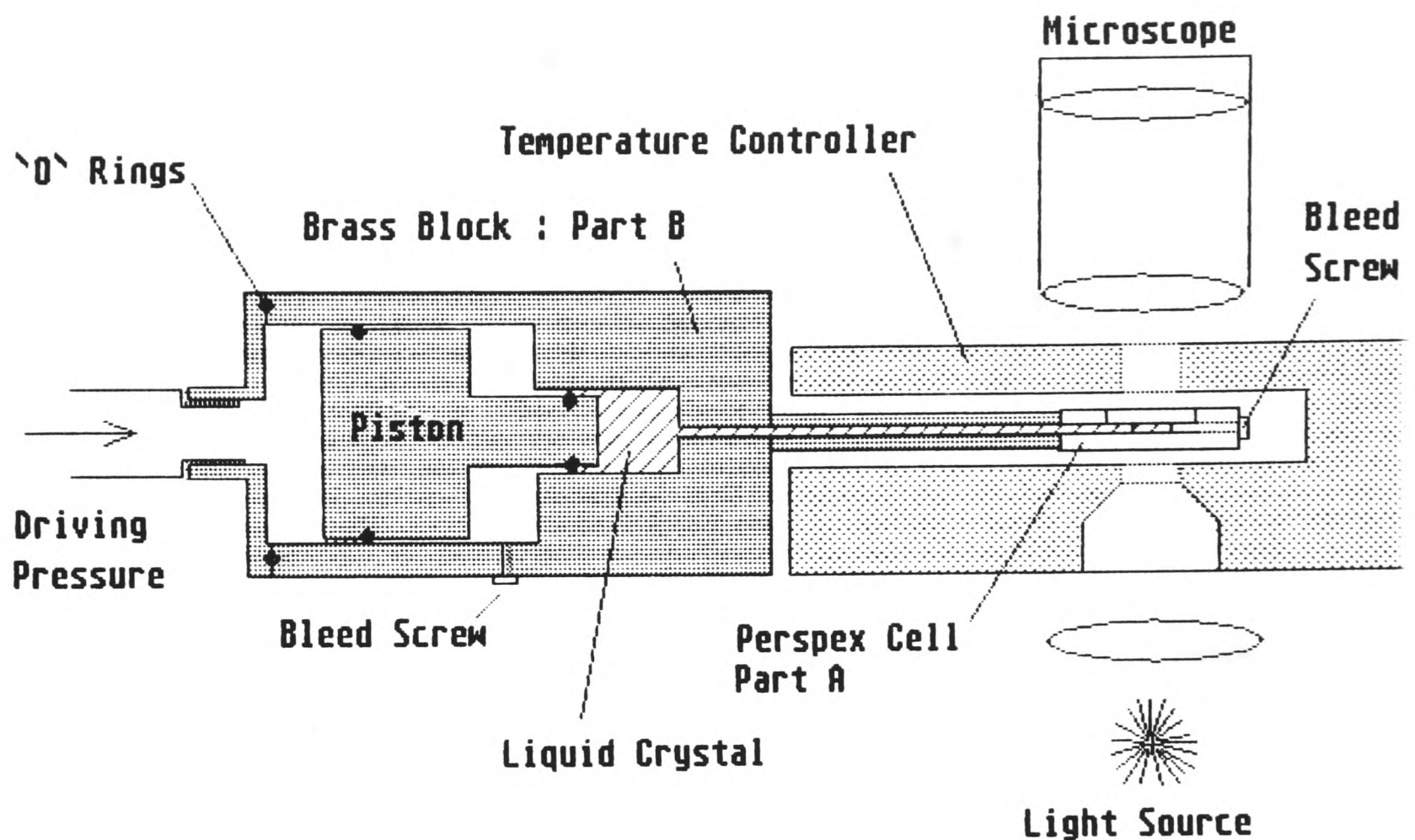


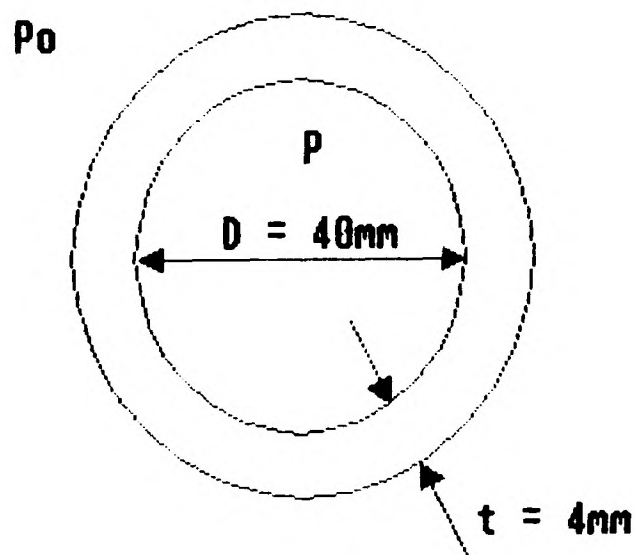
FIGURE 101. Simplified cut away diagram of the pressure cell apparatus, inserted into the microscope hot stage.



Stress Analysis

Safe working stress for brass components is about $55 \times 10^7 \text{ N/m}^2$, while for perspex the yield stress is about $5 \times 10^7 \text{ N/m}^2$.

1. Brass Block - may be considered as brass tubing. Needs to withstand 100psi.



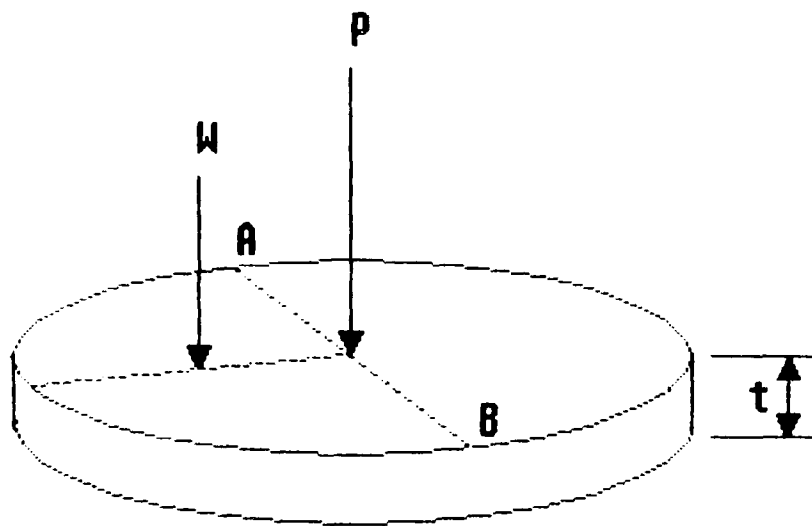
$$S = \frac{P.D.}{2.t} = 3.45 \times 10^6 \text{ N/m}^2$$

$$P = 100\text{psi} = 6.9 \times 10^5 \text{ Pa}$$

$$t = \text{thickness} = 4 \times 10^{-3}\text{m}$$

$$D = \text{diameter} = 40 \times 10^{-3}\text{m}$$

2. Perspex test area - consider lid as flat plate, supported at edges.



For one half. load = $P \cdot \frac{\pi \cdot r^2}{2}$

Assume load concentrated at the semi-centre, $4r/3\pi$ from centre,
then we have a reaction, R, at $2r/\pi$ from the centre.

Bending moment about A-B:

$$M = \frac{R \cdot 2r}{\pi} - \frac{W \cdot 4r}{3\pi} = \frac{P \cdot \pi \cdot r^2 \cdot 2r}{2 \cdot \pi} - \frac{P \cdot \pi \cdot r^2 \cdot 4r}{2 \cdot 3\pi} = \frac{P \cdot r^3}{3}$$

Sectional modulus : $\frac{I}{c} = \frac{2r \cdot t^2}{6}$

Therefore bending stress, $\sigma = \frac{M}{I/c} = \frac{P \cdot r^2}{t^2}$

Now thickness, $t = 1 \times 10^{-3}$, radius, $r = 1.5 \times 10^{-3}$.

Pressure required, $P = 2000\text{psi} = 138 \times 10^5 \text{ Pa}$

Therefore shear stress $\approx \underline{\underline{3.1 \times 10^7 \text{ N/m}^2}}$

This is fairly close to the yield stress of the material, but indicated that 2000psi could be achieved before the cell destructed. In practice the maximum pressure used in tests was 1400psi, a limitation imposed by the nitrogen line available.

The above analysis assumes that the lid is very well adhered in place. In practice it is slightly weaker than the other side of the cell, which has the same dimensions but is continuous perspex. Hence great care was taken that the lid was glued as well as possible all around the lid circumference.

5:2.2. Pressure Testing the Cell

Tests were carried out to determine the maximum pressure which the cell could withstand before breaking.

An air line was used to provide the driving pressure. This pressure just reached 100psi before the cell failed, confirming the accuracy of the stress analysis. The perspex lid of the cell blew off, and since no parts were broken the cell could be reused.

Experimental Tests

A nitrogen line with a pressure of 40psi was used in initial tests with liquid crystal, limiting the maximum pressure on the sample to 800psi.

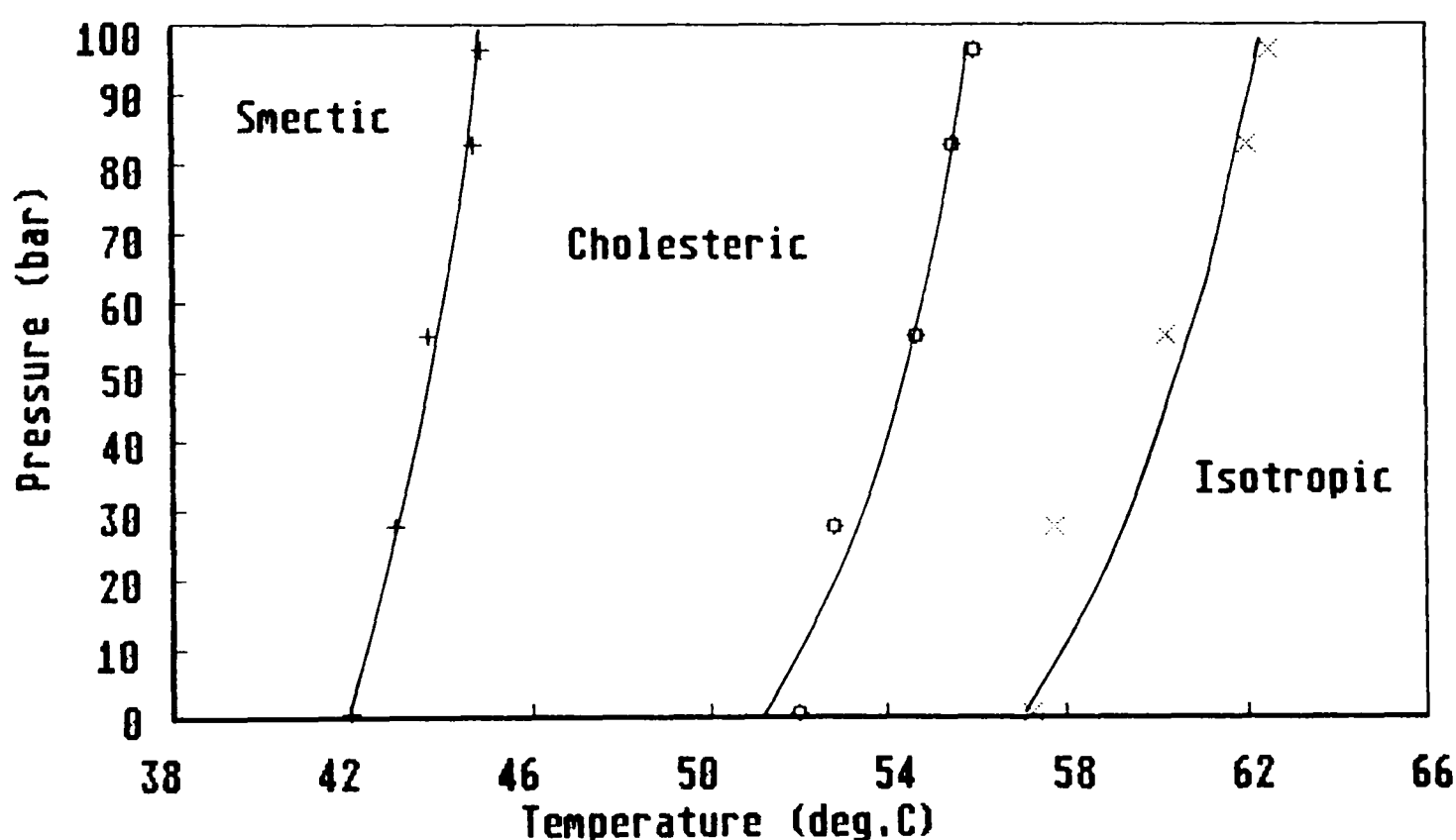
As a consequence of its high thermal mass the thermal response of the cell was slow, so only the slowest heating/cooling rates could be used if accuracy was required. This rate was 0.2°C per minute. The temperature of the transition was measured at atmospheric pressure and then at an elevated pressure.

The first set of tests used TM75B as the test material with a sample that was 1mm thick. It proved very difficult to discern the transition

changes for this thickness of sample, so the initial result of a change of $40^{\circ}/\text{kbar}$ for the cholesteric to isotropic transition needed to be checked with a thinner sample. Tests using TM75A in the reduced thickness cell resulted in a pressure sensitivity of about $60^{\circ}/\text{kbar}$.

Further tests were then carried out using a nitrogen line capable of supplying pressures up to 70psi, which would produce 1400psi at the sample. TM75B was again used, and the variation of the smectic to cholesteric and cholesteric to isotropic transitions were measured. These results are presented in figure 102, which also shows the variation of another textural change inside the cholesteric phase with pressure.

FIGURE 102. Variation of the transition temperatures of TM75B with pressure.



The variations with pressure are not perfectly linear, but the smectic to cholesteric transition temperature changes by about $33^{\circ}/\text{kbar}$, while the cholesteric to isotropic changes by about $50^{\circ}/\text{kbar}$. These figures are of the same order as results reported for cholesteryl esters. For wind tunnel use measurements with a resolution of better than 10 bar are required. This would

correspond to a change in transition temperature of around 0.5°C . This would be discerned as a colour change only if the material were in the temperature sensitive region of its range, in which case the noise from ambient temperature fluctuations would have to be extremely tightly controlled to get an accurate measurement. An alternative method might be to utilise pressure induced mesomorphic phases¹⁸. For example a cholesteric phase might only exist at high pressures, in which case the onset of colour in such a material would indicate a pressure due to the pressure induced phase transition. Limitations of time have meant that the author has not carried out any work in this area, but it may be an interesting field for further study.

6. CONCLUSIONS

The uses for cholesteric liquid crystals in aerodynamic testing appear to be very wide ranging. Within the sphere of aerodynamic testing new applications are being found, some of which have been assessed and developed in this work and a brief overview of the conclusions for each application studied are discussed below.

1. Surface Temperature Measurement

The use of the cholesteric pitch/temperature dependence to measure surface temperatures has been known about and used for some time. Encapsulation of the material provides a practical method of applying the material to a surface so that accurate, real time, reversible surface temperature mapping can be achieved in wind tunnel conditions. This method has been very successful at low heat transfer rates ($<1000 \text{ Wm}^{-2}\text{K}^{-1}$), but at higher rates or in high speed wind tunnels limits are encountered.

(i). Thermal time constant.

Response times for the colour change are dominated by the time needed for the pitch to reach equilibrium, corresponding to a different temperature. This time dependence is material viscosity related. Time constants were measured for chiral nematic and cholesteryl ester based materials, and both fall on the same curve against material viscosity. A chiral nematic material with a colour change at room temperature has a thermal time constant of about 10ms, while a cholesteryl ester will have a time constant of the order of 100 to 200ms.

(ii). Temperature Gradient.

A high heat transfer rate can cause a high temperature gradient to occur across the 10 micron encapsulated liquid crystal layer, of the order of a million degrees per meter. This causes a high pitch gradient in the material,

which badly degrades the selective reflection. Temperature gradients can also cause structural distortion due to convective flow in the material.

These limitations have been overcome by the novel methods developed during the course of this work.

(a). The cholesteric to isotropic transition of a temperature insensitive cholesteric material may be used to visualise an isotherm. This negates the problem associated with temperature gradients by minimising the pitch gradient produced. The phase change also has a much improved time constant as it is not impeded by the material viscosity. The method also offers thermal memory of the transition temperature, which is particularly useful if the model cannot be observed directly during the run. The isotherm position is also viewing and lighting angle independent.

(b). The birefringence colours of an aligned nematic layer were also investigated. The nematic material is held in a porous plastic material called Celgard, which serves to align the nematic and provide a method of obtaining an even, flat nematic layer, adhered to a test surface. The layer, when viewed between crossed polarisers, displays bright birefringence colours which are temperature, and angle, dependent. The intrinsic time constant of the colour change is negligible. Response time is only limited by thermal conduction into the layer.

Further work could be done to further assess and develop these two new methods. The effect of heating or cooling rate on the cholesteric to isotropic colour change could be assessed. Also it may be that it would be easier to trade off some of the colour/temperature insensitivity in exchange for a material which could be easily realigned by cooling it into a smectic phase below the operating temperature. The birefringence method has not been widely tested, and its limitations need to be assessed. Ideally a thinner celgard substrate is required, and the method would find wider application if its use

in reflective mode were developed. This could be done by sticking the layer to a suitable reflective background.

There are many temperature dependent properties of nematic, smectic and cholesteric liquid crystals, some of which may be developed for use as temperature transducers, for example selective reflection colours from chiral smectics, though at the present time no other effect appears to offer as much scope for exploitation in aerodynamic testing as selective reflection from cholesterics.

2. Shear Stress Measurement.

Cholesteric liquid crystals have been used for surface shear stress visualisation only quite recently. Theory predicts numerous complex effects resulting from flow in cholesterics, hence great care must be exercised when analysing experimental results as many effects may be present when only one is wanted.

Reflected colour change with shear may offer a qualitative indication of shear levels over a surface, but it is plagued with difficulties which make exact measurement almost impossible. Among these are the viewing and lighting angle effects and the fact that the material itself flows off the surface under shear, and may form rivulets which themselves disturb the flow pattern.

A new method with improved performance and great potential has been invented during the course of this work. This utilises the shear induced texture change in a cholesteric material from the focal conic to the Grandjean texture. This method gives a clear indication of the shear level from the boundary between the two textures produced when the material flow aligns. This alignment is produced by a flow driven displacement of the layer. Various levels can be measured and additives can be used to extend the range

by changing the mixture viscosity. The method not only provides a means of quantifying surface shear stress levels, but also provides very good flow visualisation.

Further work could be done in this area to develop and test the method further and evaluate its accuracy. As before, there are probably many untried methods that could be utilised using liquid crystals to evaluate surface shear stress. A method of making the shear induced colour changes reversible by including a focal-conic promoting agent within the material would be very useful. Further studies with various forms of Aerosil may make progress in this direction.

3. Pressure Measurement

A liquid crystal method has not yet been produced to accurately measure pressure differences over a surface. Reflected colours from cholesteric liquid crystals can be made fairly sensitive to pressure near the materials transition temperature, but this is the region where the colour is also at maximum temperature sensitivity, and this is likely to predominate. Transition temperature variations of the order of 30 to 50°/kbar have been found for chiral nematic materials, which may not be a great enough sensitivity for accurate measurement, although further work could be carried out on maximising this figure and investigating the use of pressure induced mesomorphism.

APPENDIX A

MEASUREMENT OF CHOLESTERIC REFLECTION SPECTRA

Reflection spectra need to be measured in order to determine the dependence of the reflected colour from the cholesteric material on influences such as temperature, shear and pressure.

For the purposes of this thesis the measurements were carried out using a Rofin Sinar spectrum analyser, which scans the visible spectrum every 80ms using a rotating diffraction grating and a photo-multiplier tube. The results of up to 200 scans can be averaged to improve signal to noise ratios. The signal may be referenced to a white standard reflector to normalise the samples reflection spectrum with respect to that of the lamp. The spectral processing was handled on a BBC micro-computer. Additional programs were written by the author to improve the output graphics and remove errors in the commercial software package. The program plots reflected intensity against wavelength and so allows accurate analysis of reflection from the ultra violet into the infra red.

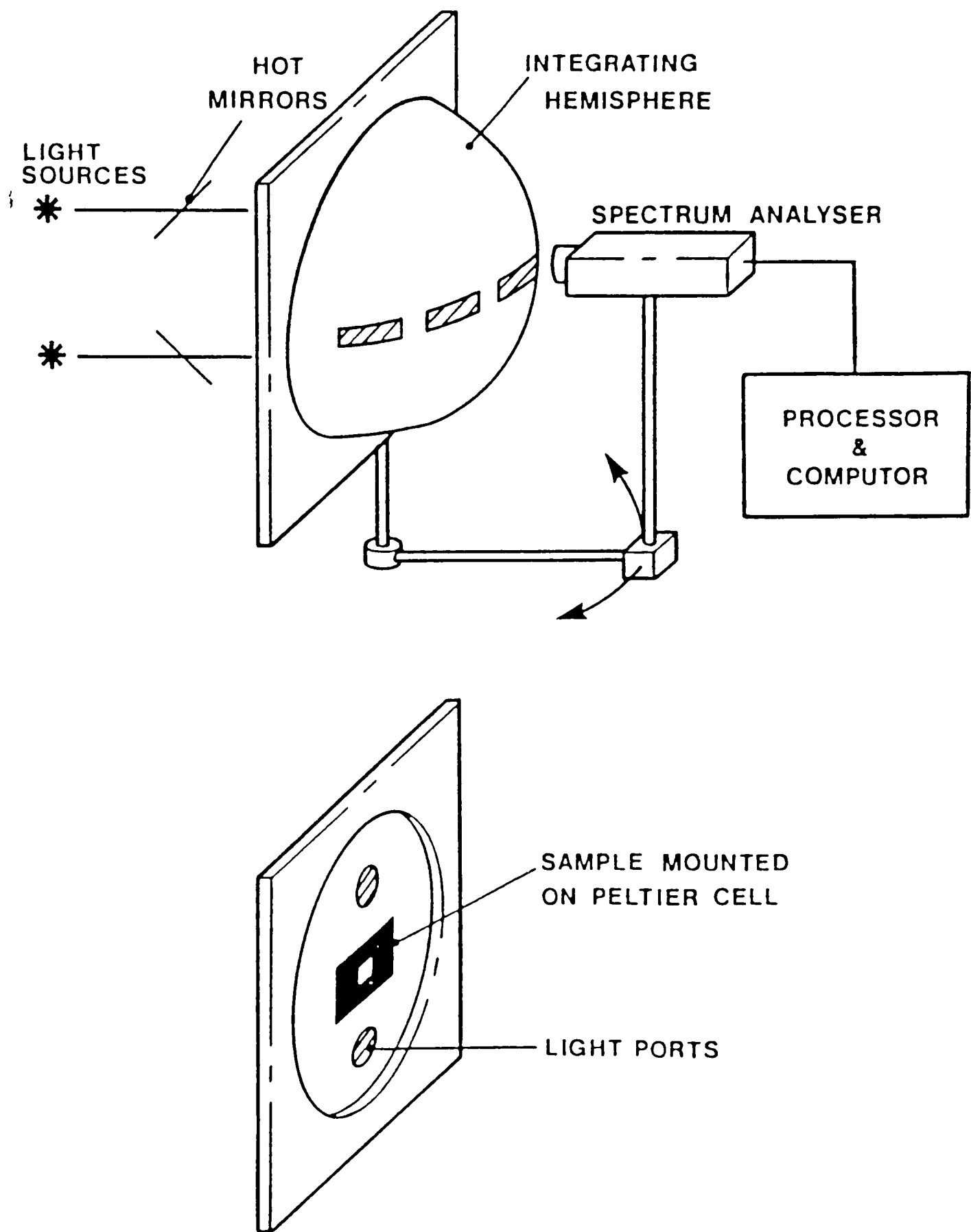
The spectra were referenced to a Kodak reflectance standard, Kodak 6091, a white powder, which was sprayed onto the substrate in ethanol, and dried to form a good diffuse reflector. As some of the liquid crystal samples used were on glass or plastic, and in some cases behind an ITO film, care had to be taken to ensure that the reflectance standard was measured behind the same type of front surface, to eliminate the effects of this from the final spectrum.

In order to allow direct comparison of both neat and encapsulated materials, and to take into account as far as possible the part specular, part diffuse nature of the reflection, an integrating hemisphere was used. It was

assumed that this would give a better approximation to how the eye perceived the reflected brightness. The hemisphere used had a diameter of 12 inches, and is shown schematically in figure A.1. Illumination was provided by two 300 watt projector lamps, which were placed behind the hemisphere and the light passed through two hot mirrors before entering the hemisphere to limit the heating of the hemisphere interior from infra red radiation. The inside surface of the hemisphere was coated with the white Kodak reflectance standard, and very uniform diffuse lighting conditions were achieved inside the hemisphere. The liquid crystal sample to be measured was mounted on a temperature controlled platform at the back of the hemisphere. The sample temperature could be controlled to 0.1°C over the temperature range -30 to $+80^{\circ}\text{C}$ by thermostated liquid from a circulator.

The hemisphere also had viewing ports cut around it to enable the angle dependence of the reflected colour to be measured. This arrangement gave clear, low noise reflection spectra, and so the method was used to measure all the reflection spectra referred to in this thesis.

FIGURE A1. Schematic diagram of the integrating hemisphere used to obtain reflection spectra from neat and encapsulated liquid crystal samples.



APPENDIX B

THERMAL MODELING

Thermal modeling of pulse heating situations was necessary to calculate the temperature of the liquid crystal layer over time. This information was required for the calculation of the thermal time constants.

Values for the thermal properties of the material have to be known to do the calculation. The values used are shown below. Measurements described in chapter 3.4. indicate that these are good estimates.

Thermal conductivity : 0.2 W/mK

Diffusivity : $1 \times 10^{-7} \text{ m}^2/\text{s}$

Both analytical and numerical programs may be used. All the equations used may be found in Carslaw and Jaeger³⁰. Programs were then written for a BBC micro computer which could plot temperature against distance at various times or temperature against time at various distances through the heated layer.

1. For simple step function heating of a single layer an analytical solution may be used.

For the layer $0 < x < L$, zero initial temperature

$x = L$ held at V , no heat flow at $x = 0$.

$$\varphi = V - \frac{4.V}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{2n+1} e^{-k.(2n+1)^2 \pi^2 t./4.L^2} \cdot \cos.\frac{(2n+1).\pi.x}{2.L}$$

This may be used if an encapsulated layer is subjected to a step rise in temperature at one surface.

2. Step function heating of a two layer system, e.g. may be used for step function heating of neat material in a glass cell.

Material 1, $-L < x < 0$, Material 2, $0 < x < \infty$

Zero initial temperature, $x = -L$ at V

$$\varphi_1 = V \cdot \sum_{n=0}^{\infty} \alpha^n \cdot \left[\text{erf.c.} \left(\frac{(2n+1) \cdot L - x}{2 \cdot \sqrt{k_1 t}} \right) - \alpha \cdot \text{erf.c.} \left(\frac{(2n+1) \cdot L + x}{2 \cdot \sqrt{k_1 t}} \right) \right]$$

$$\alpha = \frac{\sigma - 1}{\sigma + 1} , \quad \sigma = \frac{K_2 \cdot \eta}{K_1} , \quad \eta = \sqrt{\frac{k_1}{k_2}}$$

3. For the case of a parabolic rise in temperature of one surface, as occurred in the pulsing of an ITO film on a glass substrate in my experiments, it is easier to use a numerical method to calculate the temperature changes.

A finite difference approximation method was used. A step in time and distance is used. If the initial conditions are known, the progress of the temperature rise through the layer may be found from:

$$T_{t+1,x} = \frac{k \cdot \Delta t}{\Delta x^2} (T_{t,x+1} + T_{t,x-1}) - \left(\frac{2 \cdot k \cdot \Delta t}{\Delta x^2} - 1 \right) \cdot T_{t,x}$$

The temperature is calculated at each position and time through the layer. If the initial temperature is known at $t=0$, for all values of x , it is possible to construct the temperature profiles through the layer knowing the temperature profile at $x=0$. In our case the profile at $x=0$ is parabolic. $T = \text{const.} \cdot \sqrt{t}$. The only constraint on the calculation is that:

$$\frac{k.\Delta t}{\Delta x^2} \leq 0.5$$

The value of diffusivity, k , refers to the material at x .

At a boundary, a heat transfer coefficient may be defined, or a second material may be used. At the boundary:

$$T_{x+1} = \frac{T_x + (h.\Delta x./K).T_{\infty}}{1 + h.\Delta x./K}$$

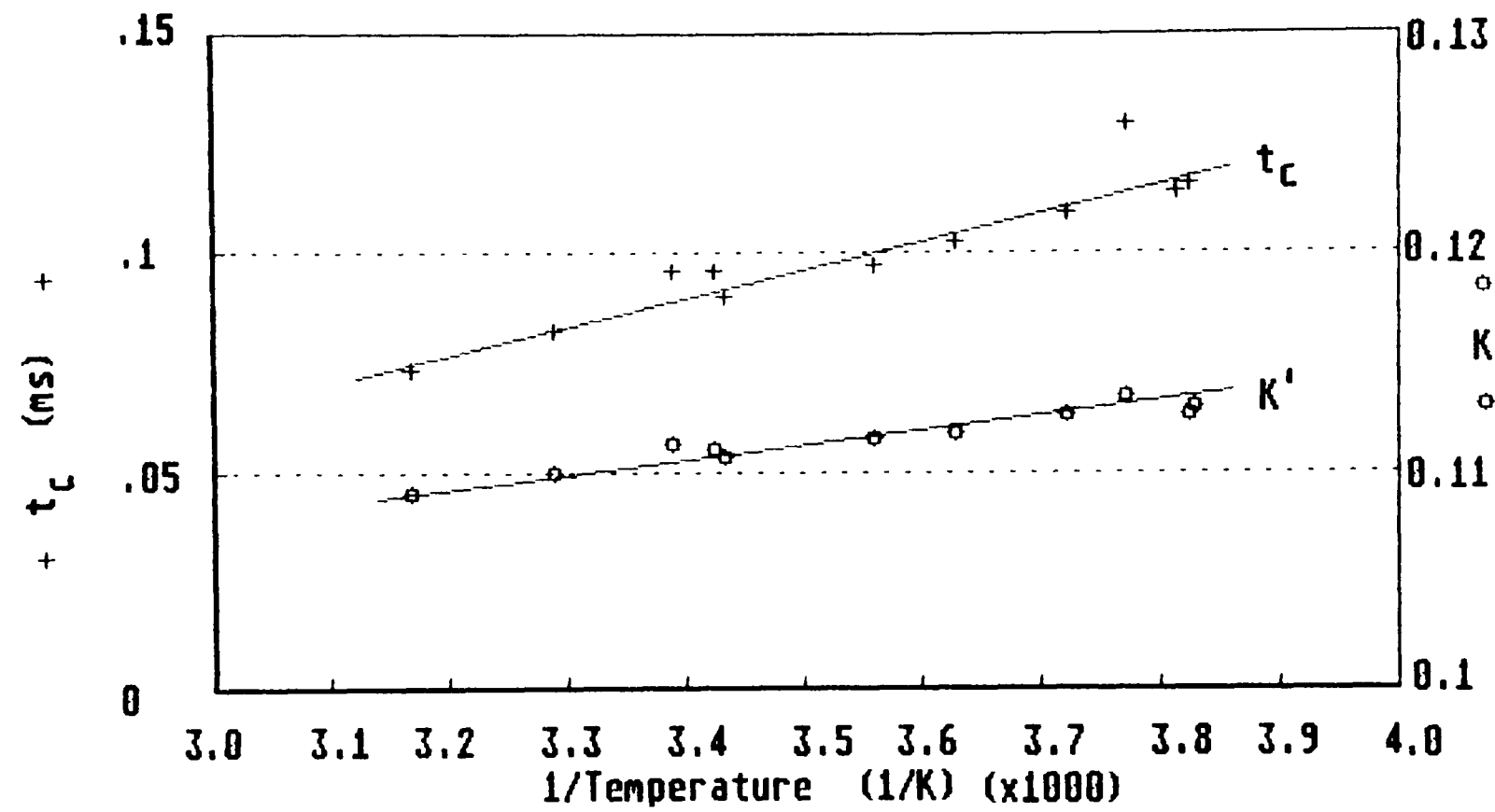
K is the thermal conductivity. For an air boundary with only natural convection present, a value of h of the order of $5 \text{ W/m}^2\text{K}$ may be used.

This method was used to calculate the rate of rise of temperature of the liquid crystal layer. It was assumed that the rise could be thought of as parabolic from a time t_c (see figure 2D), with a temperature given by

$$T_{lc} = K'.P.\sqrt{t - t_c}$$

. Using the above equations K' and t_c may be calculated as a function of layer thickness and material diffusivity. This information was used to plot figure A.2. Then values of K' and t_c could be read from this curve and used in the time constant calculation equation. This could be done because the rate of rise of the ITO film temperature was almost identical for each gauge used. A variation of diffusivity with temperature was assumed though this made little difference to the results.

FIGURE A2. Plots obtained from thermal modeling programs used to determine parameters for the calculation of the thermal time constant. K' pertains to the heating rate of the liquid crystal layer, while t_c is the extrapolated starting point of that heating, assuming it to be parabolic at small times.



APPENDIX C

LIQUID CRYSTALS FOR FLOW VISUALISATION

Although flow visualisation was not studied as a separate issue in the present work, the rivulet flow of liquid crystal material in the shear stress work is seen as an important development for this application.

Both surface temperature measurement and surface shear stress measurement are useful for flow visualisation.

Tests have been conducted by Professor Jones at Oxford into using an encapsulated cholesteric liquid crystal layer for flow visualisation by measuring temperature differences between air from different sources. The tests were very successful. Room temperature air was made to flow down a channel. Hot air was then released from a side channel. With liquid crystal covering the channel wall it was possible to visualise the flow direction of the hot gas and compare the results with theory.

Neat liquid crystal applied to a surface has also proved to be useful for flow visualisation. Apart from the tests mentioned with the shear induced texture change method, Lawrence Moreland has conducted some tests under my supervision at Oxford. The same geometry as above was used, but in this case air flowing down the large main channel was sucked at right angles down a small side channel. Liquid crystal material on a perspex sheet in the flow was used to visualise the shear levels and for flow visualisation. The shear level results did not tie in so well with theory, due to a lack of sharpness of the low shear/high shear edge, but the method certainly provided some interest and declared its potential.

There are already many excellent and interesting techniques for visualising flow⁸⁸ but as can be seen from plates 13, 14 and 15 the liquid crystal method provides a beautiful addition to the armoury, as well as providing information on the surface shear stress level at the same time.

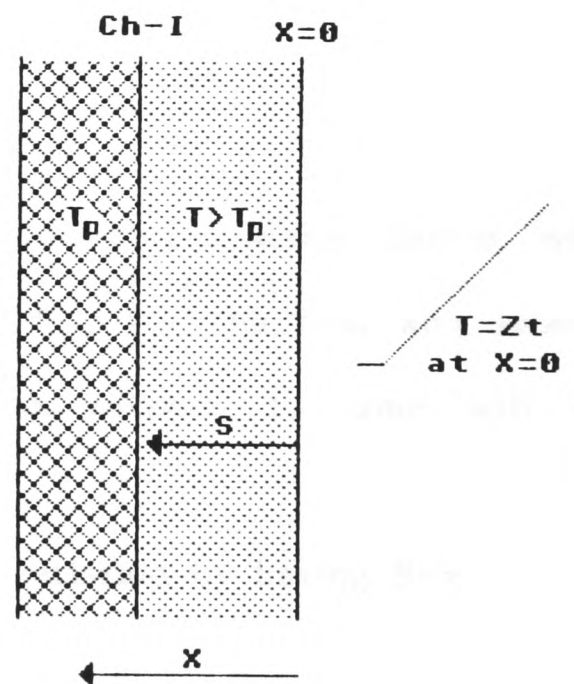
APPENDIX D

ESTIMATION OF TIME TAKEN FOR PHASE CHANGE TO PASS THROUGH A L.C. LAYER

The following is an estimation of the time required for a phase change to pass through a liquid crystal layer in a driving temperature gradient

Consider the time taken for the phase change line to pass through a layer of liquid crystal material when one face is heated.

Assume that the layer is initially at a constant temperature T_p , the phase change temperature, but that the phase change has not yet occurred. If one face, at $x=0$, is subjected to a linear temperature rise, such that $T_{x=0} = Z.t.$, the phase change line, s , will move through the layer. The aim is to calculate the time taken for this line to travel the layer thickness.



The heat transfer into the surface at $x=0$ may be given by the differential equation:

$$\frac{d^2T}{dx^2} = \frac{1}{\alpha} \frac{dT}{dt}$$

where α is the thermal diffusivity.

Also, at the phase change point, energy is absorbed due to the latent heat of the material. Therefore:

$$-K \frac{dT}{dx} = \rho L \frac{ds}{dt}$$

where ρ is the density and L the latent heat of the phase transition.
 K is the thermal conductivity.

A solution of the following form is chosen to satisfy these equations:

$$T(x,t) = A \cdot \sum_{n=1}^{\infty} \frac{1}{(2n)!} \frac{d^{2n}}{dt^{2n}} \left[\frac{(x-s)^{2n}}{B^{2n}} \right]$$

where A and B are constants. The relevant boundary conditions are:

$T_p = 0$, At $x=s$ $T(x,t) = 0$: this is clearly satisfied by the above equation as the summation is zero when $x=s$.

$$\text{At } x=s \quad -K \cdot \frac{dT}{dx} = \rho \cdot L \cdot \frac{ds}{dt}$$

To simplify the equations in the following, differentiation with respect to distance will be denoted by T_x and T_{xx} for the first and second derivatives, respectively, and differentiation with respect to time will be denoted by $\dot{T}$ and $\ddot{T}$.

The heat conduction equation may be satisfied by having $B=\alpha$.

If the solution is expanded:

$$\frac{T}{A} = \frac{1}{2} \frac{d}{dt} \frac{(x-s)^2}{B} + \frac{1}{4!} \frac{d^2}{dt^2} \frac{(x-s)^4}{B^2} + \dots$$

$$\frac{T}{A} = -\frac{1}{2} \frac{2(x-s)\dot{s}}{B} + \frac{1}{4!B^2} + \frac{4 \cdot 3 \cdot (x-s)^2 \dot{s}^2}{4!B^2} - \frac{4 \cdot (x-s)^3 \ddot{s}}{4!B^2} + \dots$$

$$\frac{T}{A} = -\frac{(x-s)\dot{s}}{B} + \frac{(x-s)^2 \dot{s}^2}{2 \cdot B^2} - \frac{(x-s)^3 \ddot{s}}{6 \cdot B^2} + \dots$$

Now find the derivative with respect to x :

$$\frac{1}{A} T_x = - \dot{s} \frac{1}{B} + \frac{2(x-s) \dot{s}^2}{2 \cdot B^2} - \frac{3(x-s)^2 \ddot{s}}{6} + \dots$$

At $x = s$

$$T_x \frac{1}{A} = - \dot{s} \frac{1}{B}$$

This result must satisfy the form $K T_x = -\rho L \dot{s}$, therefore:

$$\frac{A}{B} = \frac{\rho L}{K} \quad \text{and hence} \quad A = \frac{\alpha \rho L}{K}$$

Now a solution for the form of s must be assumed, which will satisfy the condition that $T(0,t) = Z.t$, where Z is a constant. (i.e. the face at $x = 0$ has a linear ramp in temperature. A simple series solution will be assumed for s , of the form:

$$s = at + bt^2 + ct^3 + \dots$$

At $x=0$:

$$T(0,t) = A \sum_{n=1}^{\infty} \frac{1}{(2n)!} \frac{d^{2n}}{dt^{2n}} \frac{(-s)^{2n}}{B^{2n}}$$

expanding this gives:

$$A \left[\frac{1}{2} \frac{d}{dt} \frac{s^2}{\alpha} + \frac{1}{4!} \frac{d^2}{dt^2} \frac{s^4}{\alpha^2} + \frac{1}{6!} \frac{d^3}{dt^3} \frac{s^6}{\alpha^3} + \dots \right]$$

$$= \frac{A.s.\dot{s}}{\alpha} + \frac{1}{4!} \left(\frac{4.3.s^2.\dot{s}^2}{\alpha^2} + \frac{4.s^3.\ddot{s}}{\alpha^2} \right) + \dots$$

To satisfy the linear relationship only the linear part of the assumed solution will be used:

$$T(0,t) = \frac{A.a.t.a}{\alpha} + \frac{A}{2} \frac{1}{\alpha^2} a^2.t^2.a^2 + \frac{1}{6} a^3.t^3.a^3 + \dots$$

As t is small it is reasonable to ignore terms with high powers of t , hence the solution may be approximated with:

$$T(0,t) = \frac{A \cdot a^2 \cdot t}{\alpha} = Z \cdot t$$

therefore:

$$a = \left(\frac{Z \cdot K}{\rho \cdot L} \right)^{1/2}$$

Using $s = a \cdot t$ a time may be found from:

$$\underline{\underline{t = \left(\frac{\rho \cdot L}{Z \cdot K} \right)^{1/2} \cdot s}}$$

Typical values for the heat pulse test on the chiral nematic materials used are:

$$\rho \sim 1000 \text{ kg.m}^{-3}, L \sim 4000 \text{ J.kg}^{-1}, Z \sim 1000, K \sim 0.2 \text{ Wm}^{-1}\text{K}^{-1}, s \sim 24 \times 10^{-6} \text{ m}$$

This results in a value for t of 3.4 ms

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Switzerland.
- C.5.** Degussa A.G., Geschäftsbereich Anorganische Chemieprodukte,
Weissfrauenstrasse 9 Postfach 11 05 33,
D.6000 Frankfurt 11, Germany.

