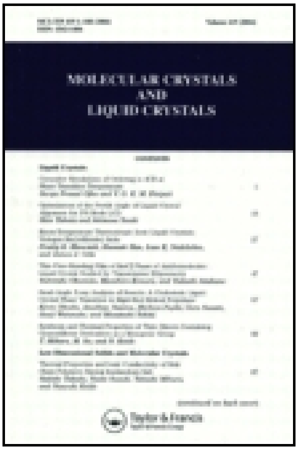

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Cholesteric Liquid Crystal Sensing of High Hydrostatic Pressure Utilizing Optical Fibers

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A new method of sensing high hydrostatic pressure utilizing cholesteric liquid crystals (ChLCs) as a sensing element and optical fibers for communication with the high-pressure region is proposed and demonstrated the concept exploits the effect of pressure-induced changes in the peak light-reflection wavelength observed in ChLCs. Measurements were performed on an encapsulated ChLC sample obtained by dispersion of a mixture consisting of nematic-host phase with cholesteryl oleyl carbonate and cholesteryl nanoate in a polymer matrix. We used multimode optical fibers for guiding the light to the ChLC sensing element and for transmitting the selectively reflected signal from the cholesteric layers to a detector. The ChLC sample with the optical fibers was placed inside a high-pressure chamber equipped with a fiber-optic leadthrough system, designed to sustain pressure up to 200 MPa. As a result we obtained a set of pressure and temperature characteristics of the device clearly demonstrating its pressure-sensing potential. A brief discussion of the high-pressure and temperature effects in the ChLC sample is also presented in the paper. The construction of a high-pressure sensor based on advanced technology for preparing of ChLCs and exploiting the attractiveness of fiber-optic techniques is proposed.

Keywords: cholesteric, pressure effect, optical fibers

1. INTRODUCTION

Over the past two decades cholesteric liquid crystals (ChLCs) have been intensively investigated from both theoretical and practical points of view. They have been successfully applied as single detectors of temperature, radiation and organic-compound vapours; as monitors of temperature fields and ionization radiation; and as infrared-visible transducers. The newest generation of liquid-crystal displays utilizes two electro-optical effects: dynamic scattering with the storage mode and the cholesteric-nematic phase transition. Other applications of ChLCs include various types of light beam modulators.

Significant progress has been made in overcoming some of the serious disadvantages of classical cholesteric compounds such as chemical, photochemical and

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electrochemical instabilities which strongly limited possible applications. In addition to pure cholesterol esters, so-called induced ChLCs (or induced chiral nematic systems) have been obtained which are composed of a nematic liquid crystal and an optically active dopant not necessarily a cholesterol derivate. Another giant step forward towards long-term stability was development of a new method of microcapsulation of ChLC films dispersed in a polymer matrix which increased stability of the so-obtained ChLC layers to several years.

High-pressure effects in liquid crystals remain an underexplored area, though their study could give new insight into the nature of molecular interactions responsible for liquid crystalline ordering and from the practical point of view they have a great potential for application in high-pressure metrology. To date few high-pressure experiments with liquid crystals have been reported,[1,2] since measurement under high pressure requires a specialized apparatus for communicating with the high-pressure chamber containing the liquid-crystal sample. The influence of high hydrostatic pressure on thermo-optical properties of ChLC was initially investigated by Pollmann[1] who determined the wavelength of maximum reflection of light λ_R of cholesteric mesophases at high pressure up to 500 MPa. His results show that in a definite pressure range (above 50 MPa) the sensitivity of λ_R to pressure is extremely large, with a red shift of λ_R, and suggest that this could provide a new method of pressure measurement. The red shift under pressure can be explained in terms of pretransition effects occurring in the vicinity of the smectic A-cholesteric phase transition,[1] for example by an increase in the transition temperature due to decreasing of intermolecular distances. In another experiment, pressure dependence of the linear birefringence of a ChLC[3] was investigated which made it possible to calculate the pressure dependence of the order parameter representing the degree of parallel orientation of the molecules. It appeared that at the transition point to the isotropic phase the order parameter was independent of the transition pressure. All the experiments were conducted by using the high-pressure equipment described in Reference 1 with a ChLC sample placed between quartz windows to ensure uniform distribution of pressure.

Application of optical fibers for communication with a liquid-crystal sample placed in a high-pressure chamber is an alternative approach in studies of pressure-induced effects in liquid crystals, one that opens up the possibility of constructing a liquid-crystal fiber-optic high-pressure sensor which can exploit all the attractiveness of fiber-optic techniques. In this paper we introduce a new method of sensing high hydrostatic pressure based on pressure-induced changes in the peak light-reflection wavelength observed in a ChLC as a sensing element, and using multimode optical fibers for communication with the high-pressure region.

2. OPTICAL FIBER SENSORS OF HIGH PRESSURE

Over the last decade, there has been extensive research and development activity in the design and production of fiber-optic sensors for the measurement of physical and chemical variables. Examples of typical measured parameters include pressure; temperature; flow; vibration; electric, magnetic and acoustic fields; linear and rotatory displacement; velocity; chemical concentration; pH; and the partial pressure of gases. Fiber-optic sensors are being used increasingly in a number of areas, including industrial process control; the electric power industry; the military sector; the biomedical and chemical sector; aerospace; robotics; nondestructive testing and evaluation; and in many other fields. These newly emerging fiber-optic devices offer important advantages in comparison to conventional sensors: since they are fabricated from dielectric materials (silica or plastic), they are suitable for use in electrically dangerous, hazardous, noisy, or explosive environments; they are completely immune to the effects of electro-magnetic interference, and they have far greater sensitivity. Adding to their effectiveness is the fact that they are directly compatible with fiber-optic telemetry, long-distance optical data-transmission systems, and optical multiplexing/demultiplexing technology.

There are two general categories of optical-fiber sensors: extrinsic and intrinsic sensors. In extrinsic sensors, the optical fiber is simply used to guide light to and from the place in which a fiber optic head is located. The sensor head is designed to modulate the properties of light in response to changes in the measured parameter of interest. In this way, optical fibers transmit optical energy to the sensor head, where the light exits from the fiber, then is appropriately modulated and coupled back via a second, receiving fiber, which guides it to the optical detector. Intrinsic sensors operate through direct modulation by the measured parameter of the light guided in the fiber. Optical fiber sensors, either extrinsic or intrinsic, may be classified into one of the four general categories: amplitude (intensity) sensors, wavelength or frequency sensors, interferometric sensors, and polarimetric sensors.

To date only a few papers[4,5] have dealt with fiber optic sensing of high pressure, i.e. pressure of at least 60–70 MPa. We have recently developed a new fiber-optic sensor capable of measuring pressures up to at least 100 MPa using the direct effect of hydrostatic stress on the polarization mode coupling which occurs in highly birefringent polarization-maintaining single-mode optical fibers.[5] This intrinsic polarimetric sensor can be regarded as a one-fiber Mach-Zehnder interferometer, carrying two modes differing in polarization and whose phase delays exhibit different responses to the external pressure. Its pressure coefficient is nearly two orders of magnitude higher than current high-pressure sensors. It also offers unprecedented flexibility to any required range of pressures up to 200 MPa. Another type of high hydrostatic pressure measurement has been proposed based on displacement of the absorption edge in a semiconductor under the effect of high pressure.[6] This extrinsic intensity sensor for a pressure range up to 100 MPa was composed of a GaAs single crystal attached to multimode optical fibers.

The proposed new high hydrostatic pressure sensing method is based on extrinsic intensity modulation of light reflected from an encapsulated ChLC which act as a sensing element. The method exploits the effect of pressure-induced changes in the wavelength of maximum light reflection observed in ChLCs. The sensing element is attached to two multimode optical fibers to transport the optical signal in and out of the high-pressure region. The low cost of commercially available multimode fibers and of different ChLC mixtures for various temperature and pressure ranges enhances the attractiveness of this sensing method.

3. CHOLESTERIC LIQUID CRYSTALS FOR PRESSURE SENSING

Since ChLCs are formed by optically active molecules introduced, in general, to a nematic host phase, the nematic uniaxial structure undergoes a helical distortion: the direction of the long molecular axes in each successive layer (made up of molecules which are oriented in parallel and moving freely in two directions) forms a given angle with the direction of the axes of molecules in the preceding layer. In this way a helix is formed, whose pitch (Z_0) is dependent on intermolecular and external fields and forces.

Optical properties of ChLCs are very specific and are determined by the pitch Z_0, the arrangement of the axis of the helix and the polarization of the incident light. When an external field (electric, magnetic, optical) or mechanical deformation is applied, changes occur in both the direction of the axis of the helix (texture transition) and its pitch (untwisting of the helix). It has been well established that temperature can strongly affect the optical properties of a ChLC. In most ChLCs, Z_0 is a decreasing function of temperature,[7] that is $dZ(T)/dT < 0$ and the order of magnitude of dZ/dT is often surprisingly large. However, other kinds of thermal behaviour: $dZ/dT \approx 0$ and $dZ/dT > 0$ are also possible, especially in induced chiral nematic liquid crystals and some explanations of these phenomena have been proposed.[8]

When a light beam is incident normally on a ChLC sample arranged in a planar Grandjean texture, it can be reflected selectively from it only if the following condition is met:[7]

$$\langle n \rangle Z_0 = \lambda \tag{1}$$

where $\langle n \rangle$ is the mean refractive coefficient of the cholesteric LC and λ is the wavelength used. This is valid in the case of a Bragg reflection from planar ChLC layers and explains why any change of helical pitch under the influence of temperature causes a change in the wavelength (colour) reflected selectively from the cholesteric layer. The spectral band of the Bragg reflection is proportional to the optical anisotropy, Δn of the ChLC:

$$\Delta\lambda = Z_0 \Delta n \tag{2}$$

and its typical values—depending on the ChLC mixture used—vary from about 20 nm to about 80 nm. Thermal dependence of the helix is the basis of all the ChLC sensing devices and monitors for detection of various physical (thermal) fields and chemical phenomena in which temperature plays a significant role.

The other thermodynamic parameter which reveals some interesting effects in liquid crystals is pressure. Measurements under high pressure can generate additional experimental data on basic thermodynamic quantities, specially at and in the vicinity of phase transitions between different liquid crystal phases.[9] Pressure plays also an important role in monitoring the character of the phase transitions. Some transitions between different liquid crystal phases are usually discontinuous, but it should be possible to make them continuous by increasing either the transition pressure or the transition temperature or both, since coexisting liquid crystal phases are thermodynamically very similar. Moreover, there are known compounds which at atmospheric pressure do not form liquid crystals but as the pressure is raised they exhibit mesophase.

For typical cholesteric mixtures, application of hydrostatic pressure causes a red shift of the wavelength of maximum light reflection, hence—according to Equation (1)—helical pitch increases,[1] and in the vicinity of the smectic A-cholesteric phase transition a divergence of the cholesteric helical pitch may even be observed.[9] This behaviour of the pitch was theoretically expected[10] and was explained in terms of pretransition effects arising from the increase of interactions between molecules due to the decrease of intermolecular distances. The typical quasi-absorption spectrum caused by selective light reflection from cholesteric mixtures is shown in Figure 1, together with a schematic representation of the influence of pressure and temperature on its position, where we assumed a 50 nm spectral band of the selective Bragg reflection. The position of the light source used (a He-Ne laser, 633 nm wavelength with a half-power bandwidth less than 1 nm) is also shown in Figure 1.

The thermo-optical properties of ChLCs have been successfully used for the measurement of superficial temperatures and in the construction of liquid crystalline temperature sensors.[11,12] In the earliest procedures, thin (10–100 μm) cholesteric layers were applied to the surface to be measured, which had first been covered with black substrate in order to improve the colour contrast of the light selectively

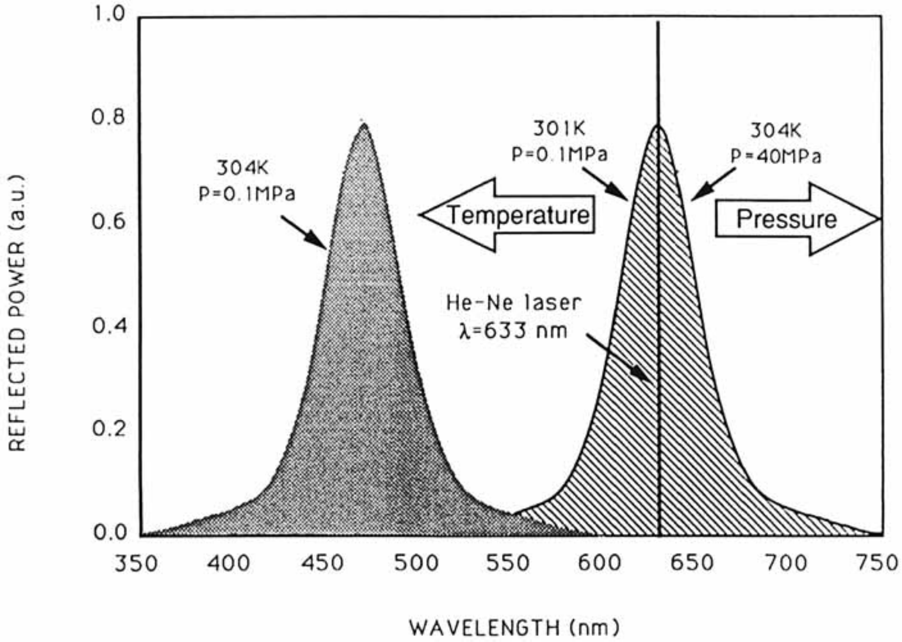

FIGURE 1 Typical quasi-absorption spectra for ChLCs (data for the Ch-A sample) far away from phase transitions, with shifts caused by influences of presence and temperature. Also shown is emission line of a He-Ne laser at 632.8 nm.

reflected from the surface. This procedure assured only about one week's stability of the liquid crystal layer. In a later method, a ChLC was placed between two elastic sheets, producing considerable improvement in the stability of the layer up to some months' duration The best results—stability of several years—were obtained with microcapsulated ChLC layers dispersed into a polymer matrix and that is why we have chosen this form of semi-rigid cholesteric liquid-crystal film packaging in constructing our fiber optic head for pressure sensing.

4. EXPERIMENTAL

The experimental setup for high-pressure measurement up to 200 MPa is presented in Figure 2, with the sensor arrangement represented in inset. The light source was a He-Ne laser at 632.8 nm modulated using a standard chopper technique and coupled to the source optical fiber. The signal reflected selectively from the cholesteric sample was transmitted through the receiving fiber and coupled to a Newport 818-SL planar-diffused PIN photodiode with a large active surface and a broadband responsivity from 400 to 1100 nm. The setup included a synchronous detector system composed also of a lock-in amplifier and a computer-controlled digital DC voltmeter.

The ChLC sample was glued to a plexiglass spacer and then to three multimode optical fibers: one source and two receiving fibers with a large core diameter (core 50 μm and cladding 125 μm) using a UV adhesive compound. The fact that the UV fiber optic resin had a refractive index compatible with the index of input/output fibers assured an optimal coupling of reflected light. The thickness of the plexiglass spacer between the fiber tip and the ChLC was subject to an elaborate optimization procedure[12] and as a result, a 1.3 mm plexiglass spacer was chosen,

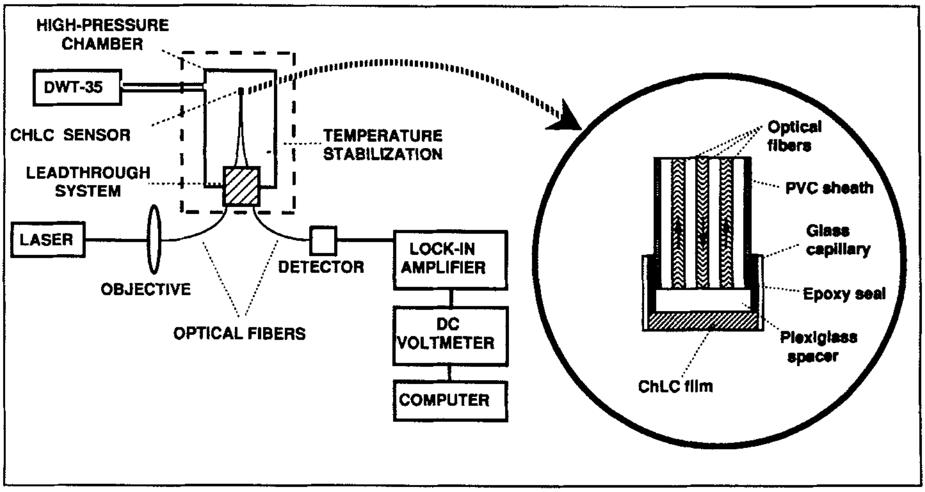

FIGURE 2 Experimental setup and fiber-optic for ChLC pressure sensing.

assuring a good insensitivity to small distance fluctuations during operation of the sensor.

The cholesteric films were obtained from dispersion of a ChLC in the polyvinyl alcohol (PVA), polymer matrix and then deposited on the Dupont black solvent. The ChLC was a mixture of nematic host phase 2-pentyl-azoxy-benzen (nematic range from 27°C–57°C) with cholesteryl oleyl carbonate and cholesteryl nonanoate and were described by us earlier[12] (referred to here as Ch-A). Alternatively, we applied also commercially available ChLC films from Davis LC, Inc. (Ch-B), designed for temperature ranges from 20°C to 45°C. All the ChLC films used were completely black, opaque and semi-rigid films with bright colours. Full colour range (from blue to red) responded to about 3 degrees' change of temperature for Ch-A films and about 5 degrees for Ch-B films.

The sensor assembly was placed inside a standard high-pressure chamber designed to sustain pressure up to 500 MPa. The chamber was thermally stabilized using an automatic controller with 0.01% accuracy of reading and a range of operation from − 15° to 150°C. A Harwood DWT-35 deadweight tester was used to generate and calibrate high pressures up to 105 MPa inside the chamber with a reading accuracy of 0.01% traceable to the U.S. National Institute of Standards and Technology. For higher pressures, the deadweight tester could be disconnected and the chamber could be fed directly from a high-pressure pump equipped with a secondary Bourdon gauge.

Insertion of the sensor into the chamber was accomplished using a fiber-optic leadthrough system described elsewhere.[13] This leadthrough system allows the passage of up to 20 separate optical quartz fibers of any kind into a high-pressure chamber. To assure better mechanical protection and reliability of the device, the sensing element should be embedded in a hydrostatic liquid, then closed and hermetically sealed using a miniature nickel bellows. The whole assembly can then be inserted directly into a high-pressure chamber, autoclave or pipeline depending on the specific application.

5. RESULTS

The experiment was conducted for pressures up to 100 MPa and temperatures ranging from 10°C to 50°C depending on the ChLC film used. Figure 3a presents data of the optical energy reflected from the Ch-A sample (full colour range corresponding to temperatures from 28.7°C to 31.7°C) for three typical isothermal pressure runs. An increase of temperature shifts a peak position corresponding to the maximum light reflection at 632.8 nm towards higher values of pressure for the Ch-A sample. There is a quasi-linear signal output *vs.* pressure in the certain range of pressure, depending on the actual temperature of the sensing element. A similar shift is observed for izobaric temperature runs (Figure 3b), for the same sample, indicating its highly sensitive dependence in narrow temperature range.

Figure 4 shows similar isothermal pressure (a) and isobaric temperature (b) runs obtained for the Ch-B sample (full colour range for temperatures from 20°C to 25°C). There is a significant improvement in both sets of characteristics for the

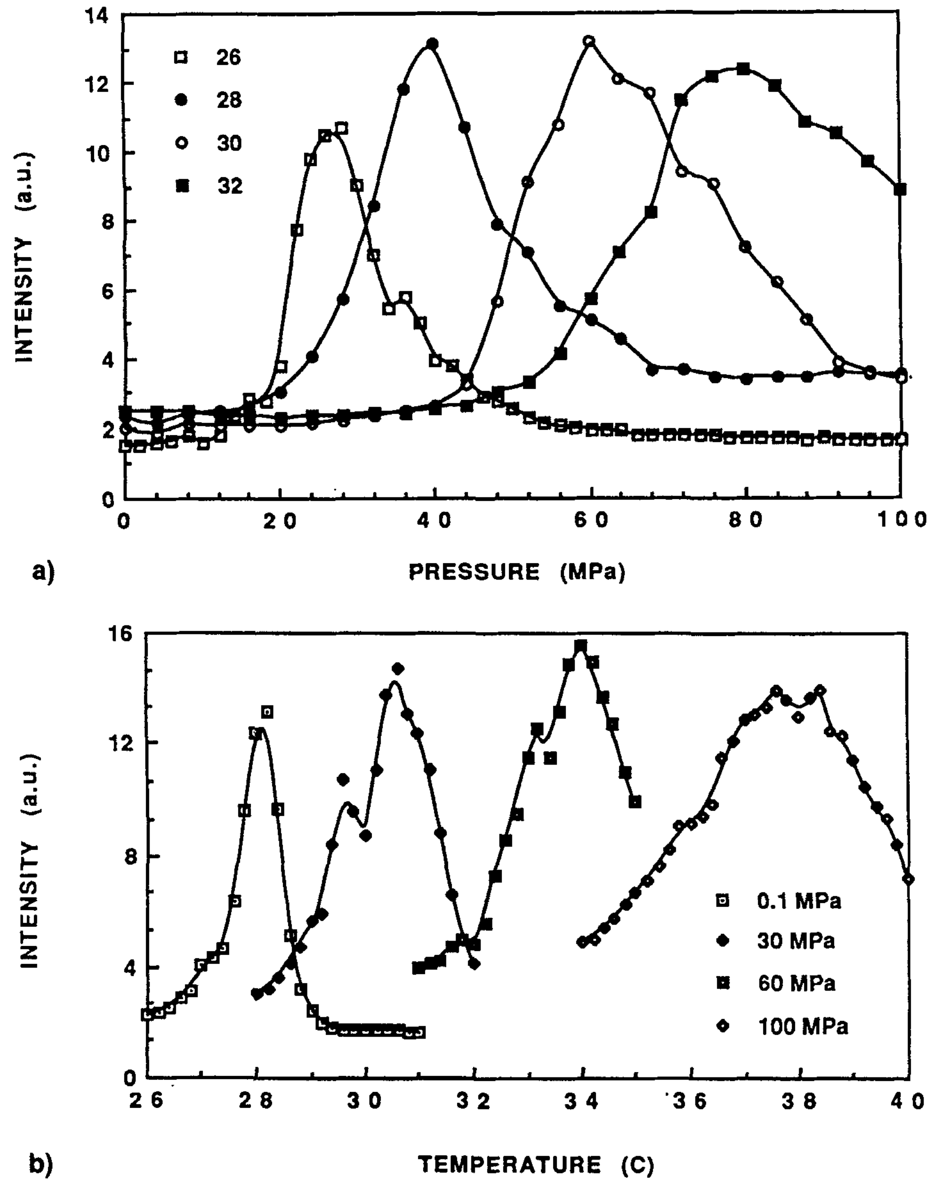

FIGURE 3 Light reflection at 632.8 nm from the Ch-A sample *vs.* pressure at various temperatures (3a) and *vs.* temperature at various pressures (3b).

Ch-B sample consisting in quasi-independence from temperature of our pressure sensing device and in quasi-independence from pressure when the device acts as a temperature sensor. A comparison of both ChLC films used for pressure sensing in the experiment is presented in Figure 5 in which pressures corresponding to the peak values at 632.8 nm have been plotted against temperature, for both sets of runs: with increasing and decreasing pressure. We observed a slight hysteresis

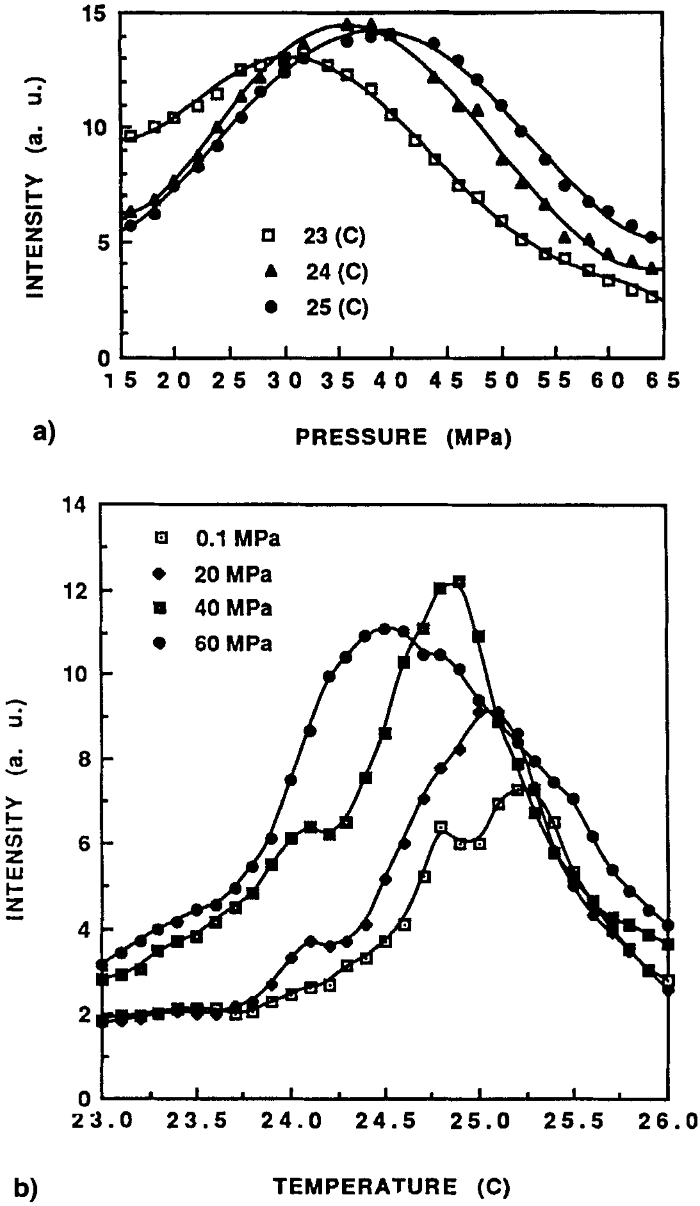

FIGURE 4 Light reflection at 632.8 nm from the Ch-B sample *vs*. pressure at various temperatures (4a) and *vs*. temperature at various pressures (4b).

(specially for the Ch-A sample) due to the sweeping rates chosen and the presence of adhesives. At an elevated range of pressure and at some values of temperature the peak broadens and a second peak may appear (Figures 3 and 4). This can probably be explained in terms of pretransition phenomena which arise in the vicinity of the smectic-cholesteric phase transition and can suggest a pressure-induced re-entrant cholesteric phase behaviour which was recently reported.[14]

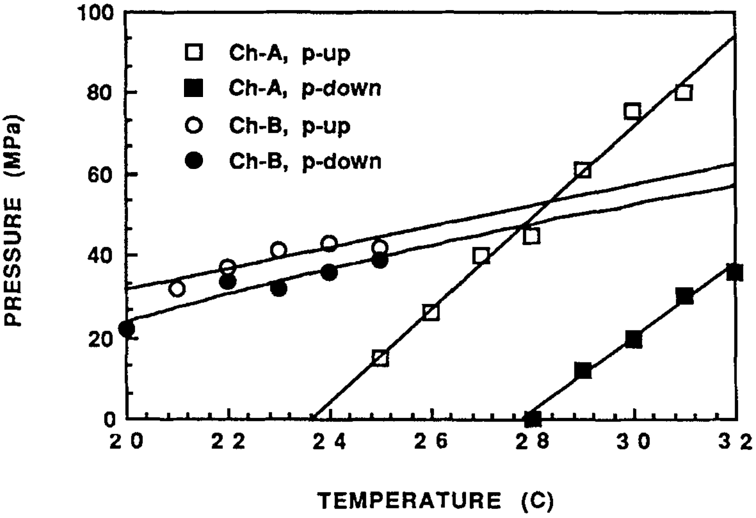

FIGURE 5 Plot of pressures corresponding to the selective light reflection at 632.8 nm against temperature, for both sets of runs: with increasing (p-up) and decreasing (p-down) pressure, and for both samples: Ch-A and Ch-B.

From Figures 3 and 4, the output signal (I) mean pressure-sensitivity expressed as:

$$\alpha_p = (d \ln I/dp)_{T=\text{const}} \qquad (3)$$

can be easily calculated. For the Ch-A sample the pressure sensitivity depends strongly on ambient temperature and varies from 4.2×10^{-2} MPa^{-1} (T = 26°C) to 2.0×10^{-2} MPa^{-1} (T = 32°C). However, for the Ch-B sample the measured sensitivity was found to be about 1.5×10^{-2} MPa^{-1} in the whole temperature region (22–25°C). Both sets of data clearly demonstrate pressure-sensing potential of the proposed sensor which has the pressure coefficient nearly two orders of magnitude higher than current high-pressure sensors.

6. CONCLUSIONS

We have introduced a new fiber-optic pressure-sensing technique which utilizes a cholesteric liquid-crystal film as a sensing probe and is based on the effect of a pressure-induced shift of the peak light-reflection wavelength observed in ChLCs. This technique, inherently immune to electromagnetic interference and safe in electrically dangerous, hazardous or explosive environments, is directly compatible with fiber-optic telemetry and optical data transmission systems. The probe embodies the principle of microcapsulation of ChLCs dispersed in a polymer matrix—an advanced technology which overcomes some of the serious disadvantages of classical cholesterics such as chemical, photochemical and electrochemical instabilities. Alteration in the composition of the components in the liquid-crystal probe can assure a good linear response and sensitivity for specific ranges useful in different types of industrial applications such as oilfield and mining instrumentation, underwater devices, and general process-control and manufacturing technologies. Our pressure sensing technique can be easily applied to construct over/under indicators preset for a required range of pressure.

We believe that through elaborate optimization which will include application of two cost-effective light-emitting diodes in reference configuration instead of a laser source, appropriate choice of ChLC sensing elements, adhesives, and the operating wavelength (wavelengths) we will be able to construct a highly sensitive liquid-crystal fiber-optic pressure sensor. The future sensor should be immune to any external distortions, including temperature and should be characterized by a wide range of linear response. The relatively low cost of producing multimode optical fibers and preparing liquid crystalline components is an additional advantage of the sensing method described in this paper.

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