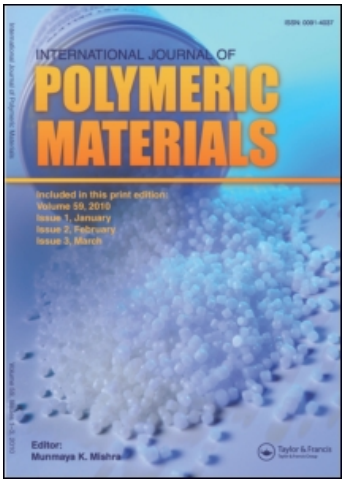

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Preparation and Characterization of a Polymeric Bulk Electro-Optic Modulator

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Preparation and Characterization of a Polymeric Bulk Electro-Optic Modulator

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The basic theoretical concepts on which the electro-optic linear effect are based and the optic modulation are briefly described in this work. A novel method for obtaining a doped bulk polymer with electro-optic properties by means of the incorporation of an organic pigment called 2-Methyl-4-Nitroaniline (MNA) in epoxy resin followed by a poling technique of the MNA molecules by means of the application of a strong electric field is proposed. Also, the construction of an optic modulator cell is described showing the experimental arrangement and the results obtained, with the corresponding calculation of their main parameters. A simplified mathematical model is also proposed.

Keywords: Electro-optics; linear effect; modulators; 2-methyl-4-nitroaniline

1. INTRODUCTION

The rapid growth of optical fiber communication systems has stimulated the search for new materials for fast and efficient processing of

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optical signals. Fiber optics voltage sensors or modulators based on the electro-optic effect (Pockels effect) in electro-optic crystals are well known from experimental high voltage techniques [1,2]. However, devices based on electro-optical crystals may have difficulties finding their way into the market for advanced future applications, where relatively low cost devices are required [3,4]. The crystals with the current technology are too costly to be used for this application. As a potential alternative, a new class of optically nonlinear organic polymers have been studied. The electro-optic effect is not restricted to inorganic single crystals, but it is also found in certain organic materials. This material has the ability to alter the propagation properties of light opening the possibility of manipulating optical signals. Operations such as optical switching, amplitude optic modulators, electric field sensors, etc., are important in the evolving field of optical telecommunications and optical instrumentation [1].

In this work, the basic concepts and the actual development of a polymeric bulk modulator made with an epoxy resin matrix doped with organic optically nonlinear materials are described.

2. ELECTRO-OPTIC EFFECT

The electro-optic effect is related to displacements of charged particles as a function of the applied field. The centers of gravity of the positive and negative charges will be displaced and this produces the so-called induced electric polarization (Pi), which can be expressed as

$$P(i) = \varepsilon_0[\chi^{(1)} * E + \chi^{(2)} * E^2 + \chi^{(3)} * E^3 + \cdots\cdots] \tag{1}$$

in which ε_0 is the permittivity in a vacuum, $\chi^{(i)}$ represents the dielectric susceptibilities and E the electric-field strength.

The linear dielectric susceptibility $\chi^{(1)}$ is connected to the dielectric constant and the refractive index n by

$$\chi^{(1)} = \varepsilon_r - 1 = n^2 - 1 \tag{2}$$

The others terms in eqn. (1) are also related to the refractive index. These field dependences are the basis of the observed nonlinear optic

effects. The term $\chi^{(2)}$ gives the nonlinear optical effect such as frequency doubling, linear electro-optic effect or Pockels effect. The term $\chi^{(3)}$ produces the quadratic electro-optic effect (Kerr effect) and stimulated Raman scattering. Only non-centrosymmetric materials show second (in general odd terms) and nonlinear effects [1]. In other words, the linear electro-optic effect manifests itself by a change in refractive index only if non-centrosymmetric materials are subjected to an electric field. Organic polymers offer the freedom to induce non-centrosymmetry in macroscopic or bulk systems. In particular, organic molecules like 4-dimethylamino-4′-nitrostilbene (DANS) or 2-methyl-4-nitroaniline (MNA) exhibit a very large second order nonlinear value and do not have centrosymmetry.

Polymers doped with non-centrosymmetric molecules (guest/host system) with large molecular nonlinearities do not always yield nonlinear bulk materials. It is clear that the orientation of nonlinear molecules in bulk samples is very important with respect to the magnitude of the second-order nonlinear properties. However, inactive polymeric systems can be converted into a second-order nonlinear medium by electric field-induced poling process [6, 5]. In non-crystalline materials, the anisotropy induced by an external electric field in the polymers could remain as long as the electric field is present, temporarily (relaxation time) after withdrawing the field or settle down permanently. This behavior is related to the alignment of certain molecules characterized by their chemical rigidity and their structure which determines how long the induced anisotropy will remain.

Polymeric materials are intrinsically isotropic, but when they are doped with polar molecules and subjected to intense electric fields during the production process, they acquire anisotropy and electro-optic effects. The stability of this depends on many factors but certainly temperature is one of the main ones which also determines the relaxation time.

3. OPTIC MODULATORS

Electro-optic modulators have as an operating principle, the change in light propagation characteristics in a material, when an electric field is applied. In other words, the applied electric field produces changes in

the material's refractive index (induced birefringence). This variation produces changes in the light polarization state when the light goes inside the material, and an array of polarizers is used. The electro-optic material could be either a crystal or a polymeric material and acts like a transducer, converting the electric signal changes and modifying the light characteristics when passes through the material. An ideal linear electro-optic modulator requires a material that, besides having a defined optical axis (anisotropic), also has a high electro-optic coefficient (relationship between the induced anisotropy or induced delay in one of the components of the light and the electric field applied), does not possess optical activity and has a high dielectric rigidity.

The basic schematic diagram of an electro-optic traverse modulator, and its components is shown in the Figure 1. Initially, the light goes through a linear polarizer where it acquires a lineal polarization state with an angle of 45° relative to the optical axis of the material. Then, the light passes through a 1/4 λ retardation plate, where the polarization state changes to circular polarization. This allows a linear operation point in the modulator to be obtained. Later on, the light penetrates the material with electro-optic properties, where a phase delay of its components, generated by the applied electric field, takes place. This delay is proportional to the electric field applied between the electrodes, producing an elliptic polarization state. This modulation of the polarization state becomes amplitude modulation when the

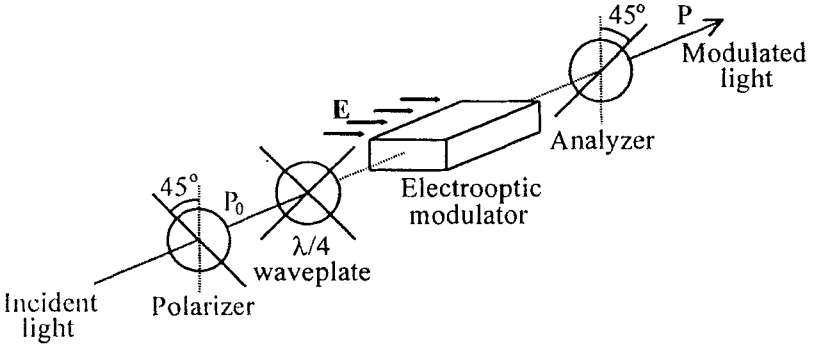


FIGURE 1 Schematic diagram of an electro-optic transverse modulator.

light goes through a second lineal polarizer (analyzer) in the optical modulator setup. In order to evaluate the modulated light characteristics, the optical power that emerges from the modulator is detected by an electronic circuit, which provides an electric signal proportional to amplitude variations of the optical signal.

The transfer function of an electro-optic modulator with polarizers perpendicular to each other is considered [2]: where P_0 is the input optical power and $\Gamma(t)$ is the delay between

$$P = P_0 \mathrm{Sin}^2 \frac{\Gamma(t)}{2} = \frac{P_0}{2}(1 - \mathrm{Cos}\ \Gamma(t)) \tag{3}$$

parallel and orthogonal to the optical axis components of the light in the electro-optic modulator. When a 1/4 λ retardation plate is added, then $\Gamma(t) = \Gamma(t) + \pi/2$ and

$$P = \frac{P_0}{2}(1 + \mathrm{Sin}\ \Gamma(t)) \tag{4}$$

The previous equation is formed by a component that is a function of the input optical power (P_{dc}) and another that is affected by modulation signal (P_{ac}).

$$P_{dc} = \frac{P_0}{2} \quad P_{ac} = \frac{P_0}{2}(\mathrm{Sin}\ \Gamma(t)) \tag{5}$$

This can be seen graphically in Figure 2.

The relation between the P_{ac} and P_{dc} components is a measure of the applied electric field and it is independent of the input optical power P_0. This relation is known as the modulation index $m(t)$ and it is represented by

$$m(t) = \frac{P_{ac}}{P_{dc}} = \mathrm{Sin}\ (\Gamma(t)) \tag{6}$$

When the induced delay in the light components is very small, it can be considered that $\mathrm{Sin}(\Gamma) \approx \Gamma$ and thus

$$m(t) \approx \Gamma(t) \tag{7}$$

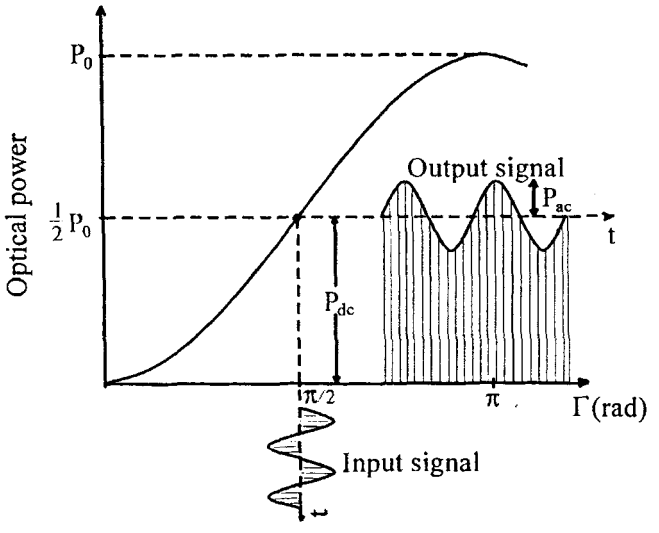


FIGURE 2 Optical array of light electro-optic modulation with an operation point of $\Gamma = \pi/2$.

For the case of a polymeric modulator with a traverse configuration [3]

$$\Gamma(t) = \frac{\pi n_0^3 (r_{33} - r_{13}) V_m(t)}{\lambda} \cdot \frac{L}{d} \tag{8}$$

where

n_0 is the ordinary refractive index of the transducer,
V_m is the applied voltage to electro-optic modulator (Vrms),
λ is the light wavelength,
L is the distance that the light travels inside the modulator,
d is the distance between the electrodes.

Considering $r_{33} = 3r_{13}$ [3][4], and $\Gamma(t) \approx m(t)$ on the equation (8), we finally obtain

$$r_{33} = \frac{3m\lambda}{2\pi n_0^3 V_m} \frac{d}{L} \tag{9}$$

$$r_{13} = \frac{m\lambda}{2\pi n_0^3 V_m} \frac{d}{L}. \tag{10}$$

4. FABRICATION TECHNIQUE

4.1. Construction of the Modulator

The type of mold used to deposit the doped polymer consists of a glass cell with metallic electrodes parallel to each other. The cell construction characteristics are shown in the Figure 3.

4.2. Chemical Composition and Polymer Preparation

Polymeric material acts basically as the host. First, polyester resin, methylmethacrylate and polystyrene were used, but epoxy resin showed the most appropriate properties for the desired function. The resin used in the process is the DER 331 Bisfenol-A type (DOW) and a cicloalifatic amine, AMICURE-PACM (AIR PRODUCTS), was used as a catalyst.

These polymers (DER series 300) are epoxy resins of low viscosity with a high content of epoxy groups. Their curing could be carried out through several types of groups of epoxy hardeners, forming thermo-fixed polymers with high net density and with a T_g of the order of the

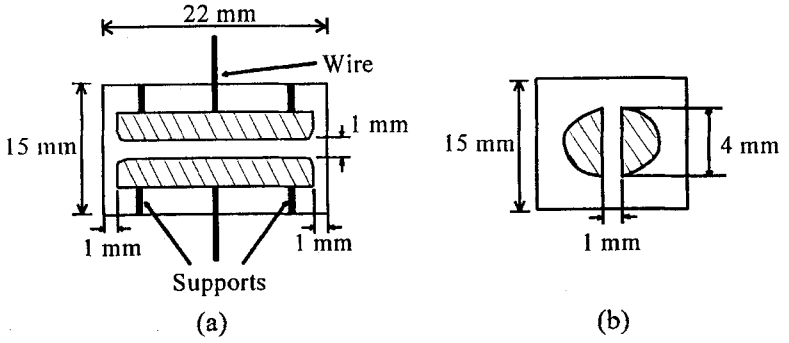


FIGURE 3 Cell characteristics used as a mold in the electro-optic modulator fabrication, top (a) and frontal (b) view.

140°C. Once cured, these resins have high thermal stability, good resistance to aging by heating and good chemical resistance to solvents.

The 2-Methyl-4-Nitroaniline (MNA) was selected as a guest. It is a highly polarizable yellow pigment, which makes it ideal for obtaining electro-optic materials. In its crystalline state, an electro-optic coefficient up to 500 pm/V has been obtained [6], but unfortunately, the growth process for obtaining bulk crystals is very difficult and expensive [7,8].

The first step of the production process consists of the incorporation of the organic pigment (MNA) in the epoxy resin. We tested several dopant concentrations. At concentrations higher than the 5%, the dopant modifies the structure of the resulting polymer, thus altering the curing time and the rigidity of the material. The concentration that was appropriate for the process and for the results described below, was from 3% in weight upwards.

The amounts of resin and catalyst used were 75% by volume of resin and 25% by volume of catalyst. In order to dope the epoxy, the MNA is added to the epoxy resin at 60°C to reduce the viscosity. Then, it is agitated until the pigment incorporates totally, and a uniform mixture is observed. Thus, the corresponding part of catalyst is added and mixed again until a homogeneous mixture is obtained. Later, this is deposited in the test cell and subjected to vacuum for 60 minutes to extract the air bubbles. Once the air has been extracted, the sample remains for the following 24 hours at room temperature to reach a partial curing where it acquires a certain mechanical hardness that allows the electrodes to remain soaked and fixed inside.

4.3. Poling and Curing Process

The following step in the production process is the poling of the molecules of MNA incorporated in the epoxy resin. This process consists in the arrangement of the molecules of MNA present in the epoxy resin by means of a strong electric field applied during the polymerization of the material. To control the electric field applied, a special device was designed. It allows the electric current that goes through the processed sample to be monitored. A schematic diagram of this device is depicted in Figure 4.

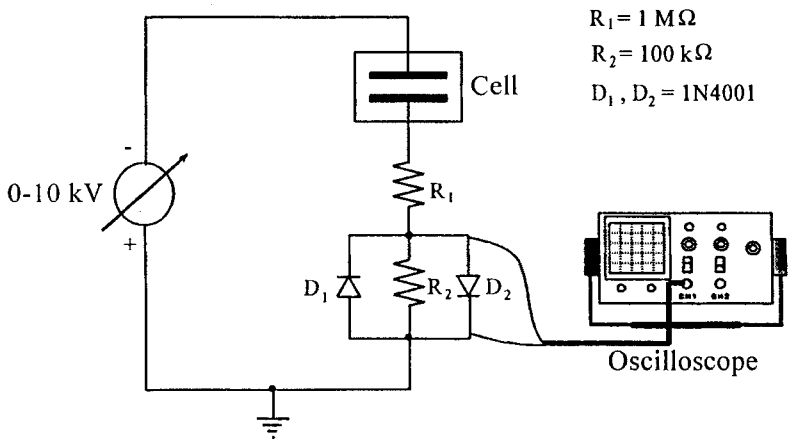


FIGURE 4 Monitoring device used for the applied voltage control for the cell poling.

The material sample is placed in an oven whose frame should be properly grounded, so the above described monitoring device can be connected. Then, the cell is subjected to the thermal cycling that is shown in Figure 5. The oven is programmed with this cycle in which the epoxy will be completely polymerized.

The poling of the doped molecules of MNA will be made by means of the application of an intense electric field between the electrodes

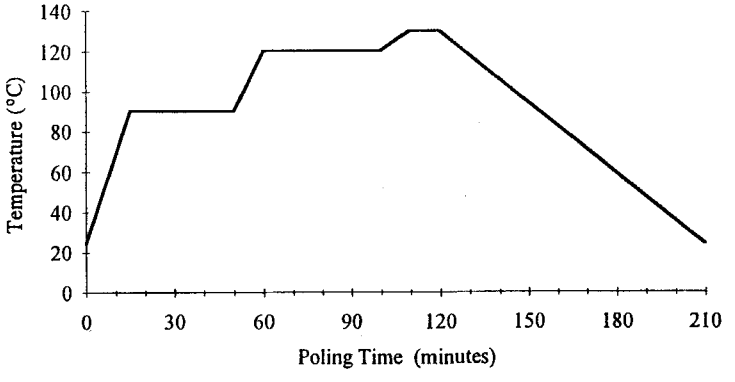


FIGURE 5 Thermal cycle for the cured and the poling of the doped epoxy resin in the modulator fabrication process.

installed in the cell. When the resin begins curing, the cell possesses a very low resistance and it could be damaged if we apply a strong electric field.

The poling of the doped polymer begins when, in the thermal cycle, the temperature reaches 130°C. A high voltage source was connected to the cell to control the voltage applied during the poling process. This voltage is continuously adjusted during the whole process in order to maintain a maximum current flow of 0.8 μAm throughout the sample; by applying to the cell, the biggest potential that is required without exceeding the current limit. This adjustment is repeated during the temperature drop until the applied voltage reaches 10 kV. The electric resistivity behavior is shown in the Figure 6. As the temperature falls, a gradual reduction of the current in the cell is observed, and this remains until the current reaches a value of the order of nAm. When the oven reaches room temperature (24°C), the poling voltage is diminished to zero and the cell is withdrawn for evaluation of its optical properties.

To obtain an appropriate cell production, it is necessary to take care of the following aspects.

- Purity of the resin and catalyst.
- The dimensions and separation between the electrodes.
- Air extraction of the resin before curing.

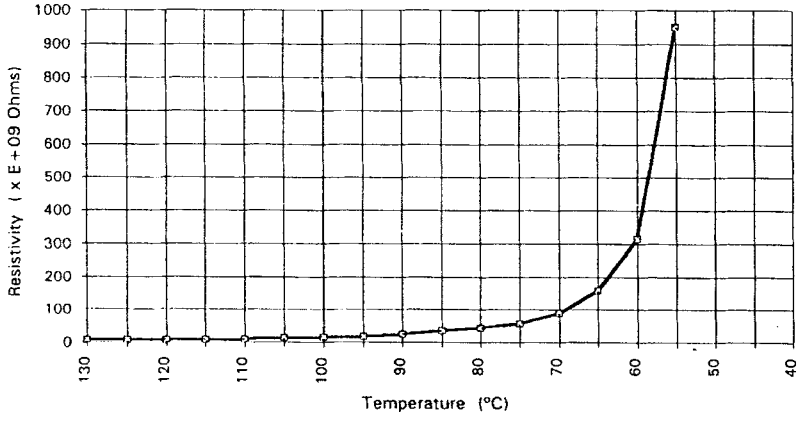


FIGURE 6 Electric resistivity behavior of the cell in the poling process.

- The finishing of the surfaces where light enters and leaves.
- Control of the humidity in the manufacturing.

5. CHARACTERIZATION

5.1. Polymeric Material

Once the doped polymer is processed, the following step is to evaluate some of its characteristics and optical properties.

5.1.1. Optical Absorption

This analysis allows the absorption that the electro-optic polymer presents when specific light wavelength is used to be measured. For this test, a Bentham M300 monochromator was used. The test was carried out sweeping in wavelength from 300 up to 1000 nm (2 nm steps). The analysis of a sample was carried out in the polymer with and without dopant. The absorption graphs of both samples are shown in the Figure 7.

It can be observed that, when the organic pigment (MNA) is incorporated in the epoxy resin, an absorption peak appears with a maximum at 470 nm. This doesn't produce sensitivity problems on the

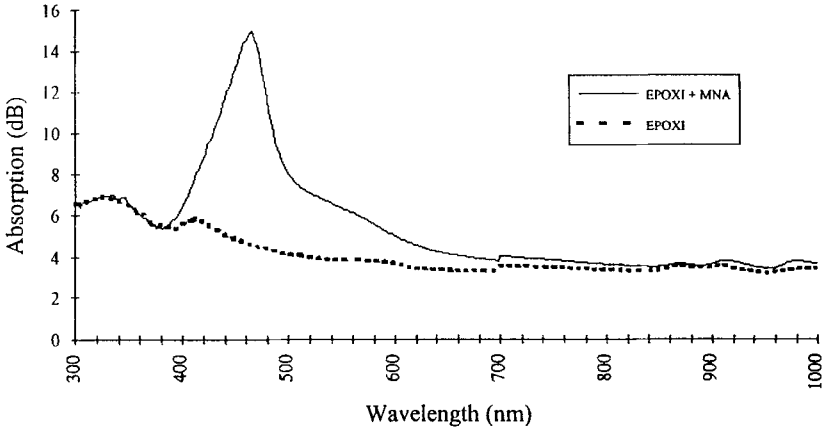


FIGURE 7 Absorption spectrum of the epoxy resin with and without MNA.

electro-optic effect of a doped polymer. The optical attenuation of the light source used (633 nm wavelength) is not significant. If a light source with a greater wavelength is used, according to the Figure 7, it will not represent a problem to observe the electro-optic phenomenon because the absorption is very small at wavelengths of up to 1000 nm.

5.1.2. Optical Activity

The optical activity was determined by measuring the change of the angle of the polarization plane that takes place in doped polymer samples with several materials or cell lengths. The optical activity of the doped polymer was measured with a polarimetric array. A shift in the polarization plane of 3 degrees per millimeter approximately was found, for the 633 nm wavelength used. For the length of the cell of two centimeters, the whole rotation of the polarization plane is of the order of 60 degrees. The optical activity sensitivity which the cell produces does not increase proportionally to the cell length, because the ideal conditions of Figure 1 are altered and the polarization plane changes along the cell.

5.2. Electro-Optic Modulator

In this evaluation, the processed material is placed in a polarimetric modulator to determine its electro-optic properties. The elements that form this device are illustrated in Figure 8. The light source used is a He-Ne laser with a 633 nm wavelength and a 15 mW emission power.

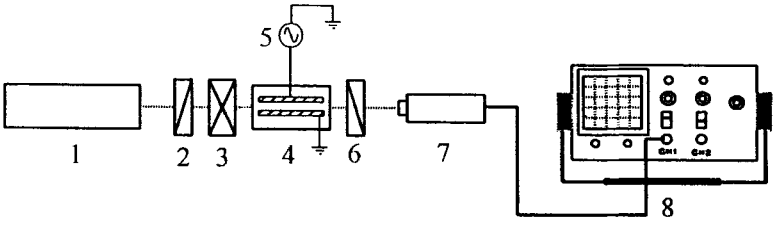


FIGURE 8 Optical arrangement for evaluating the properties of the electro-optic modulator made with the doped polymer prepared. The blocks are: 1 He-Ne Laser, 2 Polarizer, 3 Quarter wave retarder, 4 Electro-optic cell, 5 Modulation voltage source, 6 Analyzer, 7 Receiver and 8 oscilloscope.

The polarizer and analyzer are 3 FPG 003 Melles Griot lineal dichroic polarizers. The 2 WRM 003 1/4 of wavelength Melles Griot retardation plate is used. The optical detector is a low noise and high gain (10,000) amplifier, with 1 KHz band width, designed especially for electro-optic effect measurement. Figure 9 shows a typical cell manufactured as described above. A 60 Hz high voltage signal test (0–2kV) is applied to the cell electrodes. At the same time, a high voltage measurement tip is used, for monitoring simultaneously the test signal and the signal coming from the electro-optic modulator behavior. Once the optical array is adjusted to obtain maximum sensitivity, two sinusoidal signals are observed in the oscilloscope showing great similarity.

5.2.1. Cell Poling and Mathematic Model

The MNA molecules' alignment in the epoxy resin matrix is the main factor in the linear electro-optic response of the material. When the cell has not been poled, a non-linear response is observed as in Figure 10(a). In other words, if for example a 60 Hz signal is applied to the cell, the light amplitude that leaves the cell has a small modulation precisely of double the frequency. However, when the cell has

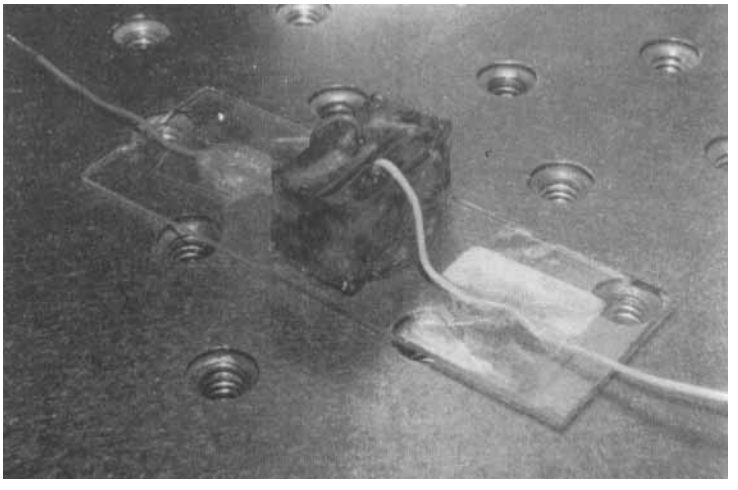
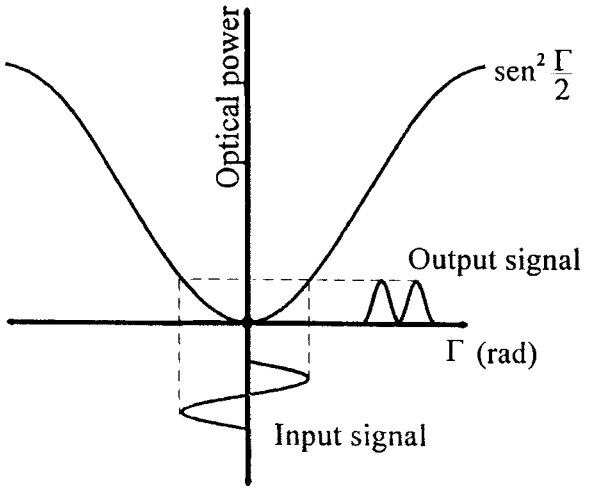
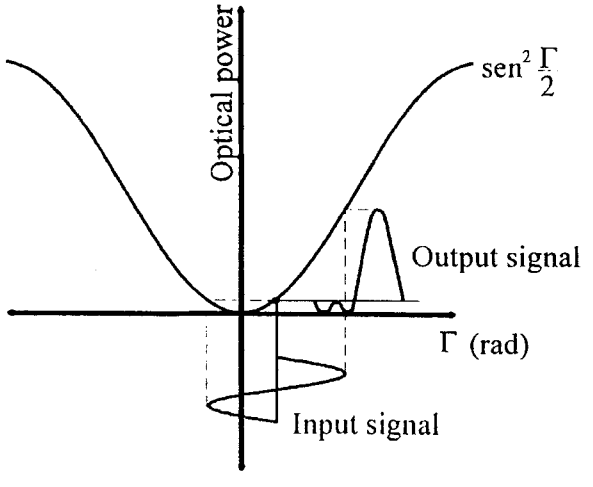


FIGURE 9 Photograph of the electro-optic modulator cell manufactured.

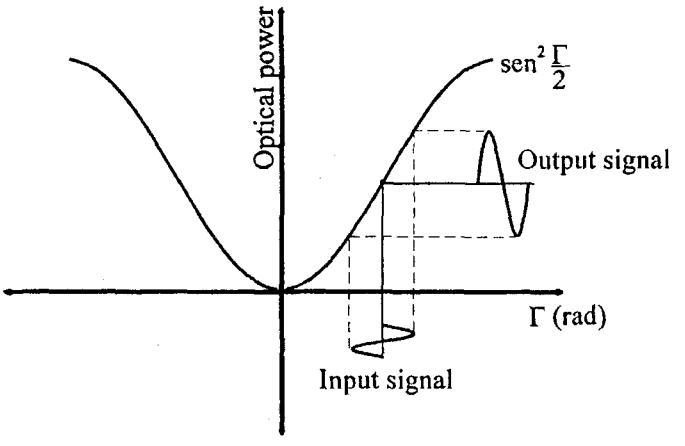


(a)



(b)

been previous poled or the input signal has an added dc voltage (they behave like equivalent inputs), the output signal from the cell has the waveform showed in Figure 10(c). Partially poled cell behavior is shown in Figure 10(b) and its respective measured experimental



(c)

FIGURE 10 Schematic cell behavior without poling (a) partially poled (b) and strongly poled (c) polymer.

curves are shown in Figure 11. The observed waveform is the result of the adding of two components, the first is proportional to the input signal and the second, is nearly proportional to the quadratic function of the input signal. Therefore

$$V_o = k_1(V_i \pm k_2)^2 \tag{20}$$

where V_i is the input voltage applied to cell, V_o is the optical signal modulated in magnitude, k_1 is a factor associated with the sensitivity of the cell and it is function of the physical parameters of the material and the cell dimensions. The k_2 factor is associated with the dc voltage applied to the cell, which produces the MNA molecular alignment. This behavior was experimentally confirmed, measuring the relationship between the input and output signals. The function that represents the measured behavior is

$$V_o = 9.645 * 10^{-5} * (V_i \pm 81.938)^2 \tag{21}$$

from the equation (21) we have simply

$$V_o = 0.647 + 0.0158\ V_i + 9.645 * 10^{-5} * V_i^2 \tag{22}$$

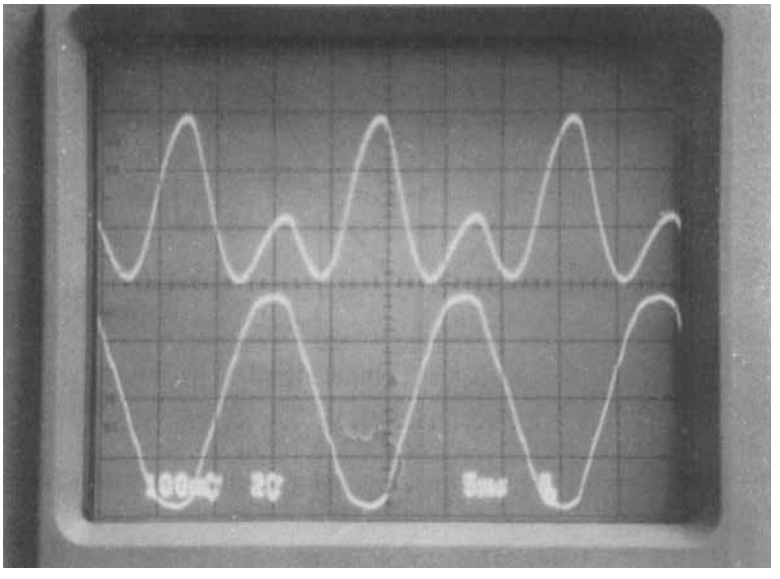


FIGURE 11 Photograph of the waves forms when the cell is partially poled, output (upper) and input (below) signals.

Analyzing each term of this function, we have that the first term doesn't have the input signal and it produces only an offset in the output signal. The second term is proportional to the input signal and the coefficient represents the molecular alignment in the cell. The last term is a quadratic term and its coefficient depends only on the cell physical characteristics. This simplified mathematical model was tested and its error was less than 2%. The measured curves are presented in Figure 12 and it shows the amplitude of the sinusoidal peaks behavior of Figure 11.

5.2.2. Sensitivity, Linearity and Dynamic Range

Once the cell was correctly poled and the electro-optic modulator was working properly, the modulator behavior was characterized. The signal that leaves the cell was correlated with the input test signal. The result obtained from this measurement is shown in the Figure 13. The best sensitivity obtained was approximately 1 Vpp in the output when in the input is 1400 Vpp.

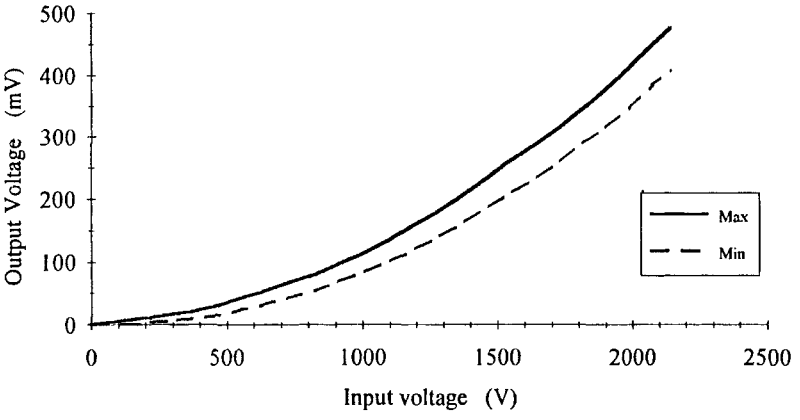


FIGURE 12 Transfer function curves measured from partially-poled cells.

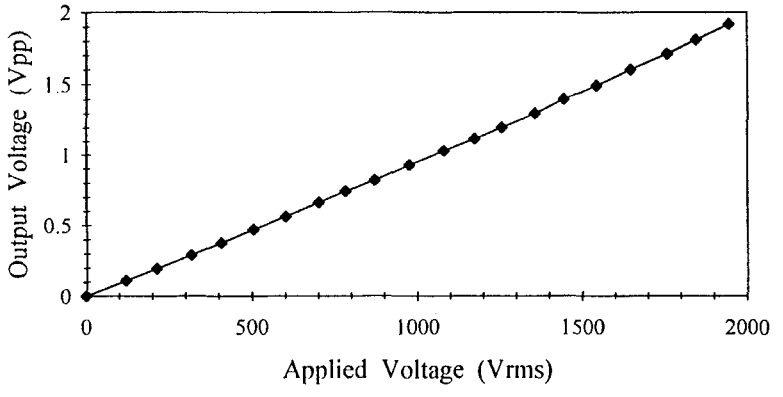


FIGURE 13 Polymeric electro-optic modulator response when it is used as a high voltage sensor.

We observe that the evaluated cell shows good high voltage signal shape reproduction, because it has good linear behavior with respect to the input test signal in the range of 0 to 1500 V (dynamic range).

5.2.3. Electro-optic Coefficients

If we use the equations (9) and (10) obtained previously, with the experimental data it is possible to obtain an approximation of the

coefficients r_{33} and r_{13} of the polymer manufactured. In order to obtain the value of the modulation index, we applied a 560 V_{rms} test signal to the cell and the values of the components P_{ac} and P_{dc} in the receiver from the coming signal of the electro-optic modulator were measured. The data obtained were 1.414×10^{-3} V_{rms} for the P_{ac} signal, and 0.55 Volts for the P_{dc} signal. The modulation index calculated is 2.571×10^{-3}. The measured refractive index is 1.58, with which the electro-optic coefficients result in

$$r_{33} = 17.59 \times 10^{-15} \quad \text{and} \quad r_{13} = 1/3\, r_{33}.$$

5.2.4. Modulator Capacitance

The modulator capacitance is one of the decisive factors in the frequency response of the modulator. For its measurement, a HP impedance bridge was used and the value of $C = 4$pF was obtained for the cell configuration used.

6. CONCLUSIONS

From the results obtained it could be concluded that the employment of polymeric doped materials with optically active substances, such as MNA, is a good alternative for electro-optic modulator production. The polymeric doped materials have, as main their advantages, low cost of production and a large dynamic operation range. As disadvantages, they have a small electro-optic coefficient and poor thermal stability, when we compared with inorganic crystals like KDP and $Bi_{12}SiO_{20}$.

The optical modulators developed will be used as optical sensors for electric field measurement in high voltage laboratories where a band width of at least 20 MHz and high electric isolation from the measurement point are required.

At this point, we are working on the optimization of the production process and on the integration of a complete measurement system, using optical fibre to carry the optical information. On the other hand, other materials such as an inorganic doped matrix are being tested, with the purpose of developing new materials with better electro-optic properties.

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