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Author/Contributor:

Peng, Gang-Ding; Ji, Philip; Chu, Pak

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Electro-Optic Polymer Optical Fibres and Their Device Applications

G. D. Peng¹, P. Ji and P. L. Chu*

Photonics and Optical Communications Group,
School of Electrical Engineering & Telecommunications
The University of New South Wales, Sydney 2052, Australia

* now at the Optoelectronic Research Centre
City University of Hong Kong,
Tat Chee Avenue, Kowloon, Hong Kong

ABSTRACT

This paper will report recent developments in electro-optic polymer optical fibres. We discuss mainly the experimental work done by the Photonics and Optical Communications Group in the University of New South Wales. The relevant issues regarding material processing, design and fabrication of electric-optic polymer fibres and devices will be addressed. In particular, electro-optical polymer fibres that incorporating various electro-optic organic materials, including the well-known disperse red 1, the synthetically attached DR1 side chain monomers, optical liquid crystals, etc, have been fabricated and investigated.

Key words: Polymer optical fibre, electro-optic effect, fibre device

INTRODUCTION

With the rapid development of fibre communication networks, the trend is that electronic components are being replaced by their optical counterparts, and furthermore, the old photonic components give way to the new ones. For example, the existing optical fibre network technology mixes a number of different network protocols (ATM, SDH, SONET, etc.) and it requires the entire capacity of an optical fibre electronically processed at each node in the network [1]. Now it is clear that electronic processing poses a bottle-neck for future expansion of network functions [2]. To avoid this problem, future optical networks will be based on a new physical layer that uses direct optical connections (optical routing) [3]. The novel photonic network technologies under development, eg. Internet Protocol (IP)-over-photonic systems, would be much more cost-effective or bandwidth-efficient by bypassing unnecessary electronic processing at nodes. However, these networks will require various photonic switches (routers) to replace electronic routers to perform configurable optical path cross connection (OPXC) [4].

It has long been recognised that EO waveguide or fibre devices, as fundamental building blocks of photonic switches, modulators or routers, would play an important role in photonic networks [5-7]. Substantial efforts

¹ Email: G.Peng@UNSW.edu.au; Phone: 61-293854014; Fax: 61-2-93855993; <http://www.unsw.edu>; The University of New South Wales, Kensington, Sydney 2052, Australia

have been made on the inorganic (glass/crystal) EO waveguide devices and this has been very successful in achieving optical modulators at very high speeds close to material limits. Various waveguide-type EO devices have now been widely used in current optical networks.

Work on EO devices based silica optical fibres, however, has so far achieved limited success [8]. Since the conventional material of optical fibre, viz. fused silica, is amorphous with a macroscopic inversion symmetry, its second-order nonlinearity $\chi^{(2)}$ is close to zero. The effect could be improved only when a certain poling process is applied. The study of the second-order nonlinear effect in silica fibre has been started since early 1990s [6]. Electric field poling has been found useful to introduce $\chi^{(2)}$ to silica fibre. The EO coefficient between 0.1pm/V and 1pm/V in silica fibre has been demonstrated [9,10].

The search for new materials for better EO waveguide and fibre devices is getting more momentum because of the push for higher and higher network speed. In recent years, research interest has been shifted toward EO waveguide and fibre devices based functional polymers. It has been demonstrated that EO organics have a much higher EO coefficient and faster response than traditional inorganic crystals [11]. Moreover, EO polymers could offer unique and superior characteristics in the design and fabrication of even higher speed devices. Their low dielectric constants allow low capacitance for lumped circuit configuration and provide a nearly ideal velocity matching between optical waves and microwaves for broadband travelling wave structures. It has been demonstrated that polymer EO devices can have modulation bandwidth of 100GHz or more, much higher than that achievable from inorganic EO devices [12].

The recent development of single-mode POFs has also provided new opportunity for fibre NLO devices [13-16]. In terms of material compatibility, many highly nonlinear organics may be easily and uniformly incorporated into the polymer matrices. This makes it possible to produce highly EO active POFs. While devices in waveguide form are suitable for system integration, those in fibre form have the advantage of possible greater length (several meters for fibre devices compared with several centimetres for waveguide devices) to accumulate sufficiently large effects of interest. Furthermore, they are more compatible with silica fibres in other parts of the system. Hence there are considerable interests in R & D of polymer EO fibres or fibre-optic devices [17-20].

Since that appropriate alignment of functional organic molecules in polymer matrices is crucial in achieving desirable electro-optic effects, we systematically studied two relevant processes: mechanical stretching during fibre drawing and electrical poling. The effects related to fibre drawing were investigated by monitoring the transverse birefringence. Experimental results show that the birefringence increases with drawing speed and that it arises from relevant contributing factors: molecular alignment, residual thermal stress and quenching effect. The effects related to poling were investigated using a simple yet accurate measurement system. Optimal conditions that produce the best electro-optic effect have been examined. We will also discuss some key issues that need to be addressed in future research and development.

MATERIAL ISSUE

Various types of nonlinear optic (NLO, $\chi^{(2)}$ or EO in this context) organic chromophores may be doped into POF [18-20]. An EO chromophore molecule is asymmetric with a large molecular hyperpolarisability β and manifests itself molecular electric dipole. When they are doped into a certain polymer matrix, they are randomly oriented. However, when the chromophores are aligned (at least partially) along a certain direction, the polymer will exhibit the 2nd order nonlinear optical effect.

The chromophores used in our experiments include DR1 (the commonly used dye for 2nd order nonlinear applications), DR19, CANS (from Los Alamos National Laboratory), MTPV (from Australian National University), NSPP (from Los Alamos National Laboratory), dimethyl (1S,9S)-5-cyanosemicorrin-1,9-dicarboxylate (DCD, a type of chiral material) and 4-cyano-4'-(2-hydroxyethoxy)-azobenzene (CHAB, a type of azobenzene compounds, courtesy of University of Science and Technology, China). We also tested on side chain polymers, in which one end of DR1 molecule is chemically attached to the PMMA backbone by free radical copolymerisation (courtesy of IBM Almaden Research Centre). The molecular structures of some dyes

listed above are shown in Fig.1. The concentration of the dopants used in POF is usually smaller than that used in optical waveguide structure, because the length of fibre to be used can be much longer than that of waveguide.

Glass transition temperature, T_g of an EO polymer is a very important material parameter. It affects the EO effect of the material in various ways. The most important factor is its relevance to the poling conditions such as poling temperature. Typically, the poling conditions and temperature must be determined related to T_g . In our experiment, we have investigated the poling efficiency vs. the poling temperature. Another important factor is its relevance to operation temperature, thermal stability or long term stability. Typically, the higher the T_g , the higher operation temperature or the better long stability. The reason behind this is the relaxation behaviour of the EO effect in a poled material that is essentially determined by the difference between T_g and the operation or environment temperature.

Below are the T_g values of some of the PMMA-based polymer samples (some with EO dopants) used in our experiment measured using the DSC method:

Polymer Material with Different EO Dopant	T_g (°C, measured)
PMMA (without dopant)	101-106
Poly(DR1-MMA) (PMMA with DR1 side chain, 24% wt)	131
PMMA doped with Poly(DR1-MMA) (4% wt)	81
PMMA doped with DCD (5.3% wt)	109
PMMA doped with azobenzene compound (800ppm)	69
PMMA doped with azobenzene compound (1000ppm)	84

POLING EXPERIMENT

The NLO chromophores as dopants are aligned randomly in polymer matrix. Therefore these doped polymer do not present the desirable EO effects in normal circumstances. To achieve EO effect in the polymer, a certain alignment of the doped EO molecules must be introduced. Direct electric field poling is the conventional method for this purpose.

For EO polymer with planar waveguide structure, corona poling and other modified poling techniques are used to achieve high electric field across the polymer thin film [21-25]. However these techniques cannot be easily applied on fibre because of the fairly different geometry. Therefore the conventional direct electric poling is used in our experiment.

The experimental setup for poling POF and its preform is shown on Fig.2.

The sample shown is a 2 to 3 mm thick slice cut from a twin core POF preform. The 2 cores are in parallel to each other and parallel to the top and bottom surfaces of the slice. Two electrodes (flat surface metal pieces) connected to a high voltage source are placed across the preform slice. The preform slice and electrodes are put in an oil bath to allow temperature control. The transformer oil also helps to reduce the electric breakdown voltage since oil has better electric insulation than air. The dc voltage source provides output up to 50 kV. The current flow in the circuit is constantly monitored through the micro-ammeter. A 30 k Ω resistor is connected in series in the circuit to avoid damage to the circuit in case there is the electric breakdown in the poling sample.

Before the high voltage is applied, the oil bath is heated up to the required temperature. The temperature is maintained at this level, the voltage source is turned on, the voltage across the sample preform slice is raised gradually to the maximum level. Both the high temperature and the high voltage are kept for a period of time to allow the realignment of the doping molecules. Then the temperature of the oil is lowered down to the room

temperature while the high voltage is maintained. This ensures that the alignment of the doping molecules is frozen in situ. Then the voltage source is turned off gradually. Fig.3 shows the poling schemes for the applied voltage and sample temperature.

A few factors that will determine the magnitude of the nonlinearity induced by poling are: poling field strength (which depends on the DC voltage applied and the distance between poling electrodes), poling temperature, poling time and the concentration of dopant.

We have developed a few designs of EO POFs, as shown in Fig.4(a) and Fig.4(b). Fig.4(a) is a slab twin-core EO POF and Fig.4(b) an H-shaped twin-core POF. The flat surfaces of the slab POF and the grooves of the H-shaped POF allow electrodes be made easily. Since electrodes are needed for the functioning of an optical modulator or sensor, the poling electrodes can be incorporate with the sensing electrodes. Besides the advantage of reducing the design of separate poling and sensing electrodes, it has another important advantage -- it allows the poling to be done after fibre drawing. If the poling is done on the preform, when the poled preform (which has then possessed EO effects) is then drawn into fibre, the high temperature required for the fibre drawing (usually above 250°C) will affect the pre-alignment of the doped molecules obtained by poling. But if the poling is done after fibre drawing process, the alignment will maintain.

In order to decrease the distance between the electrodes (so that higher electrode field strength can be achieved at same voltage source), the POF preform is cut into a H shape (Fig.4(c)), with a rectangular groove at each side. The grooves will ensure that the distance between electrodes is small across the doped fibre core, while other uncut section still maintain relatively large distance, which behaves as an insulating layer to avoid electrode breakdown at low voltage. The grooves also help in holding the electrodes applied (either in solid form or using conductive epoxy). The preform is then polished and cleaned.

The H-shape POF preform is then drawn into fibre. The drawing condition is similar to other polymer fibre. The H-shape cross-section is maintained at the drawn POF. Due to the different POF preform and different drawing speed and condition each time, the drawn H-shape POF has different cross-section. Typically the distance between the bottom of 2 grooves is about 100 to 150 μm , the depth of grooves is 80 to 100 μm and the width of grooves is 120 to 180 μm . The diameters of both cores are about 50 μm .

There are 2 method used to apply electrodes onto the H-shape POF. In the direct method, pure silver wire is used as the electrodes, because the diameter of the silver wire is smaller than the width of the grooves, it can be easily inserted into the grooves, epoxy adhesive is used to hold the conductive wire permanently inside the grooves. Fig.5 is the cross-section of the H-shape POF with silver wire electrodes buried inside. The advantage of this method is that it is easy to make and it is clean as it does not require using conductive epoxy, so the maximum breakdown voltage for the doped POF is high. However this method cannot guarantee that the distance between the electrodes is same as the distance between 2 grooves, because it is difficult to ensure that the silver wire is staying at the bottom of the groove. As a result, the electric field for poling is difficult to control, and the grooves are not fully utilized.

In the second method, a conductive epoxy with silver wire is used to construct the electrodes.. The wires also serve as the leads to the poling circuit. This method has overcome the problem of direct silver wire method because it ensures that the conductive material fills the entire grooves, and the distance between the electrodes is exactly same as the distance between the bottoms of 2 grooves. However, electric breakdown still occurs at relatively low electric field strength (about 20 volt/ μm), and so improvement over the first method is not as high as we would expected. It seems that there could have some conducting substances in glue electrodes diffused into the POF.

The poling of EO POF is similar to the poling of the POF preform, except the electrodes are different. For POF, because the distance between electrodes is much smaller (typically 50 - 100 μm , in comparison to 1.5-3 mm for POF preform), the required voltage is much lower. A dc voltage source with maximum output of 3.1 kV is used.

A large number of samples with different material, structure, condition and fabrication procedures are tested. The experimental results show that, for the POF samples with silver wire electrodes, higher electric fields can be achieved (up to 4 kV between the electrodes, which is about 150 μm apart). For the samples with conductive epoxy, the performance and breakdown voltage varies from sample to sample: some breaks down at low voltage, some behaves like a resistor, others can stand high voltage. Apart from the difference between the material and fabrication procedure of these samples, applying an insulating layer seemed an important factor. Usually the super strength Araldite epoxy adhesive can be effective, allowing the poling field up to 30 V/ μm , which is higher than that in the poling of POF preform slices.

MEASUREMENT OF EO EFFECT

The EO effect is usually measured using Mach-Zehnder interferometer scheme [20,26,27]. However this method often suffers from system drifts due to the difference in the disturbing factors affecting the signal and reference paths. We setup a measurement system as shown in Fig.6. The light source is a 633nm He-Ne laser source. A quarter wave plate is inserted to obtain circular polarization light. The circular polarised light is then passed through a polarizer at 45° with regard to the primary optical axis of the test sample. So the light incident into the preform is polarised at 45°. A second quarter wave plate is placed at the output of the sample, it is set at 45° optical axis too. Its purpose is to compensate any intrinsic birefringence that comes from the sample itself. Then the output light is passed through an analyser, which is used to obtain $\pi/2$ bias. The output signal is detected by a photo amplifier, and measured by both a lock-in amplifier (for ac signal) and an oscilloscope (for dc signal). A signal generator generated a sinusoidal wave, which is amplified through a transformer.

For poled polymer which is doped with second order nonlinear materials, the model of Class 3*m* crystal can be used. The EO coefficient can be obtained by:

$$r_{33} = \frac{3\lambda d V_{ac}}{\pi V_{0,rms} L n_0^3 V_{dc}}$$

Where λ is the wavelength of the laser light. d is the distance between 2 electrodes. L is the length of the sample. n_0 is the refractive index of the polymer material. $V_{0,rms}$ is the rms value of the input modulating ac signal. V_{ac} is the ac output from the photodetector, obtained from the lock-in amplifier, and V_{dc} is the dc value of the output from the photodetector.

The advantage of this method is that it is very accurate because the common optical path of the two polarisation components substantially nullifies those disturbing factors affecting the phase measurement. And it takes into account the intrinsic birefringence of the sample and compensates it. This method can achieve high sensitivity and detect EO coefficient as small as $1.0 \times 10^{-15} \text{m/V}$ or less. Also, this measurement system is very straightforward and simple, no complicated signal processing electronics is required.

EXPERIMENTAL RESULT

Samples of POF or POF preform are many times thicker and the achievable electrical field strength is much weaker (for some reasons the breakdown electrical field is found to be considerably lower) as compared with EO polymer film samples reported by other research groups.

Fig.7 shows the EO effect introduced by poling a DCD-doped polymer fibre preform with thickness of 2.7 mm. The poling was performed at 102.5°C at 35 kV for 4 hours (the 35 kV dc voltage was applied on the preform for another 1.5 hours during the process of lowering down the temperature). The horizontal axis is the rms value of the applied ac modulation voltage, and the vertical axis is the signal detected by the lock-in amplifier. The result shows that the output modulated signal is linearly proportional to the applied modulation signal and goes through

the origin. Fig.8 is the EO effect from a POF preform doped with CHAB. It also shows that the linearity of EO response is very good.

The key factors in poling are poling electric field strength, time, temperature and process. They will determine the end result of poling. This EO effect, in terms of the coefficient r_{33} , can be expressed as:

$$r_{33} \propto N\beta \frac{\mu E_p}{k T_p}$$

where β is the molecular hyperpolarisability, N the number density of the EO chromophore, μ the molecular dipole moment, E_p the poling field strength, k is the Boltzmann's constant, and T_p the poling temperature. Note that this expression is only true when poling temperature T_p is above the glass transition temperature T_g of the polymer because under this situation the viscosity of the material is low. Experiments are carried out to evaluate these factors.

Effect of poling temperature: The effect of poling temperature is investigated and the result is shown in Fig.9. The experiment is carried out by poling under different temperature, while all other parameters are kept the same. The sample under test is doped with NSPP (3500ppm), with a T_g at about 100°C. It is shown that at low temperature, poling experiment does not introduce significant EO effect. This is expected since the dopant and base polymer molecules are not very free to move or rotate yet. As poling temperature increases towards the T_g , the EO effect increases, as seen from the increasing value of r_{33} . However one should note that this increasing of EO effect with poling temperature is only true at temperature around or below T_g . When the temperature is above T_g , and when the viscosity of the polymer becomes low, the thermally activated random molecular movement will be dominant, suppressing the electrically activated alignment.

Effect of poling time: For poling experiment on thin films reported by other researchers, it only requires tens of minutes (even as low as 2 minutes for some experiment) to obtain final EO result. Experiments are carried out to pole the samples at same poling condition at various length of time, ranging from 2 hours up to 5 hours (including the cooling time when the high voltage is still applied). The EO results from these experiments are similar, which show that 2 hours is sufficient for the dye molecules in our samples to reach the final alignment state.

RELAXATION OF EO EFFECT

Once the EO effect is obtained in poled samples, it must be able to be maintained under practical conditions. This is essential for device applications where a device is expected to reliably function for a very long period of time. Hence it is important to investigate the stability of the EO effect under various possible circumstances and to ascertain the suitability of EO effect for a certain device application.

When EO effect is introduced by poling, the functional molecules are aligned in a certain way by the poling electric field. When this poling electric field is removed, the aligned molecular system is not in its thermodynamic equilibrium state. Hence naturally there is a tendency that the alignment could be lost, due to external disturbances. In other words, the poled polymer will exhibit thermodynamic instability, which might lead to orientation relaxation of the poled molecules. In fact, the alignment of molecules could be disturbed by various physical factors such as molecular thermal vibrations.

An empirical function to describe the decay in EO effect is the Williams-Watts relaxation function [28]. It describes the non-symmetrical dielectric relaxation behaviour for a distribution of relaxation times. Many past experiments show that this function can describe the relaxation of NLO effect well. The function is given by:

$$r_{33}(t) = r_{33}(0) * \exp\left[-\left(\frac{t}{\tau_0}\right)^\beta\right]$$

where τ_0 is the time constant for the relaxation, β is an order parameter that determines the width of the function, or called the breadth of distribution, t is time after poling of the polymer sample.

We studied the relaxation of the induced EO effect in our POF samples under different conditions.

Long term relaxation of EO effect at room temperature: This experiment is carried out by measuring the EO effect of several poled samples over a long period of time. Some of the samples are kept at room temperature over a few months, with their EO effect tested periodically. Fig.10 shows the normalised curve for the relaxation of EO effect of a DCD-doped POF preform (a) and CHAB-doped POF preform (b) at room temperature over several months. The measured data are fitted with the Williams-Watts function, as shown by the solid lines. It can also be noticed that the noise in these measurements is quite large, this is because the optical path is re-setup for every measurement. This will introduce some differences among each measurement condition. However, the result clearly shows that the EO effect relaxes in a manner in good agreement with a Williams-Watts function. Comparing the relaxation behaviour of the two chromophores, the sample doped with azobenzene compound relaxes faster than that doped with DCD. This is reasonable since the azobenzene compound-doped sample has much lower T_g (measured to be about 69°C, as compared to 109°C for the DCD-doped sample). In general, under a certain room temperature, the lower the T_g , the easier the alignment disturbed by thermal vibrations.

Accelerated relaxation of EO effect at elevated temperature: The relaxation of EO effect is also examined at raised temperatures. In this experiment, the poled POF preform sample was placed in position in the measurement setup and it was heated by a heating element with appropriate temperature control. The EO effect is monitored and measured on-line at varying time intervals. Fig.11 shows the normalised curved for the relaxation of EO effect of a DCD-doped POF preform at 60°C and 40°C respectively. At elevated temperature the relaxation is much faster, each experiment only takes several days. The sample is remained in position in the setup throughout the whole experiment, the measurement condition is kept very much the same. This is reflected at the small noise shown on the measured data.

WORK BY OTHER RESEARCH GROUPS

Work on EO POF has been initially proposed by Kuzyk et al at Washington State University [14,20,26, 29-31]. They have proposed and demonstrated an very good design EO POF with single or twin cores, where the core is straddled by 2 indium electrodes as shown on Fig.12.

They introduce EO chromophore dopants such as DR1 into this type of POF. They have achieved fairly high electric field strength in their experiment. The distance between 2 electrodes of their POF is about 40μm. The poling field is about 25V/μm when the applied voltage is 1 kV. With a 16 cm poled sample of a DR1-doped POF, they obtained an EO coefficient r_{13} about 0.2×10^{-14} m/V (ie, 2fm/V), which is about 2 orders of magnitude smaller than expected for DR1. Their explanation is that it is due to depoling when the device was placed in an oven during fabrication to cure the epoxy securing the electrode leads quickly, the higher temperature allowed the polymer to relax and lead to the depoling [29].

DEVICE APPLICATION

Many device applications are based on the EO effect. First, ultra-high speed EO modulator is in the category of microwave photonics and it is very useful as external modulator for future ultrafast fibre networks. A number of EO organic chromophores (disperse red 1, disperse red 19, etc) have been identified to have the fast speed and large EO coefficient. However, how to incorporate these materials into POFs to construct practical components

remains to be a great challenge. Also, EO fibre optical switch with very low operation voltage may be made. Various photonic switches or routers would be required for future IP-over-photonics systems. These devices will have very large bandwidth, but they may not require very fast operation. For this case, we plan to develop liquid-crystal doped polymer and POFs. This will be a new type of EO POFs. Since the liquid crystals could have very large EO effect, it may provide very low operation voltage and reasonably fast speed.

An example design of these devices is shown in Fig.13. In its design, one of the cores in the twin-core polymer optical fibre is doped with EO material and then electric-poled through applying high voltage at the electrodes at both sides of the fibre. The fibre is then tapered at both sides of the segment so as to introduce coupling between the two cores. In this way, a Mach-Zehnder interferometer is created over the section: taper, electrode segment and two tapers which form 3dB optical couplers. The optical switch is then completed when one of the cores at the input end of the above assembly is connected directly to a laser source which preferably should be a semiconductor laser diode and one of the cores at the output end is connected to a photodetector.

CONCLUSION

Polymer optical fibres doped with various nonlinear optical chromophores have been targeted for future fibre optic EO device applications. Several key factors concerning the creation and maintaining of the EO effect in POF are experimentally examined. For example, the EO efficiency of poling under various poling temperature has been investigated. We have also developed and tested H-shaped POF. EO effect has been observed in POF or POF preform samples with a range of nonlinear chromophores, although the magnitude of the EO effect is not as large as what we would expect. We have observed also EO effect in DCD and azobenzene compound doped POF samples, than larger some published results from conventional material such as DR1.

So far the research in this area are mainly preliminary work. Many important issues must be addressed in further studies. To substantially increase the magnitude of EO effect, thus enabling practical device applications, will be the main and very challenging task. At the moment, the poling efficiency in POF is low. The EO effect achieved in doped POFs is 1 or 2 orders of magnitude smaller than that in film or waveguide configurations. The task in this particular area is to unravel the mechanisms of EO effects in POFs and to apply the effect to the construction of practical devices such as modulators and switches, for optical fibre networks. The dynamics of electro-optic effects under varying poling conditions, material compositions, fibre designs and fabrication processes remain to be investigated and evaluated. Methods of how best to incorporate functional chromophores and/or organic groups into POFs are to be further explored aiming at producing desirable EO responses and better optical qualities. Also the thermal stability will be another important issue. The preliminary results from our experiments on thermal relaxation of the EO effect show that the thermal stability must be improved.

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(a) DR1

(b) DR19

(c) DCD (Dimethyl (1S,9S)-5-cyanosemicorrin-1,9-dicarboxylate)

(d) CHAB (4-cyano-4'-(2-hydroxyethyloxy)-azobenzene)

Fig.1 EO chromophores used in experiment.

Fig.2 Experimental setup for poling POF preform samples.

Poling Conditions

Fig.3 Schematic diagram of typical process of poling POF samples.

Fig.4 An H-shaped, twin-core POF preform.

Fig.5 Cross-section of a H-shape POF with electrode applied using direct method.

Fig.6 Measurement setup for EO effect in poled POF preform samples.

Fig.7 EO effect in DCD-doped POF preform after poling.

Fig.8 EO effect in CHAB-doped POF preform after poling.

Fig.9 Poling-induced EO effect at different poling temperature.

(a) Normalised EO coefficient r_{33} in DCD-doped sample.

(b) Normalised EO coefficient r_{33} in CHAB-doped sample.

Fig.10 Long term relaxation of poled-induced EO coefficient at room temperature.

(a) DCD-doped POF preform, 60 degree.

(b) DCD-doped POF preform, 40 degree.

Fig. 11: Normalised relaxation curves of EO property at raised temperature.

Fig.12 Washington State University design of EO POF [14].

Fig.13 Design of EO POF modulator.