Field-Effect Optical Modulation Based on Epsilon-Near-Zero Conductive Oxide

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Abstract:

Epsilon-near-zero (ENZ, dielectric constant $\epsilon_r \approx 0$) materials have attracted significant research interest, however, their applications in the near-infrared regime are very limited. Conductive oxide (COx), owing to its moderate carrier concentration, is a candidate for ENZ material at telecom wavelengths based on the Drude model. Herein, we report an indium tin oxide (ITO) thin film as an ENZ material with cross-over wavelength, where real part of permittivity crosses zero, and enhanced light absorption at telecom wavelengths. We also report the investigation of electro-absorption modulation based on a metal-oxide-semiconductor (MOS)-like structure, more specifically a metal-oxide-ITO stack, where ITO works around ENZ. Based on the attenuated total reflectance (ATR) configuration, our test shows great promise for future electro-optical (EO) modulators. The operation speed of the MOS-like structure is limited mainly by RC delay.

Key words:

Electro-optical Modulator, Epsilon-Near-Zero (ENZ) Material, Indium Tin Oxide (ITO).

1. Introduction

Research on metamaterials has shown that the dielectric constants of materials can be engineered to be almost any arbitrary value (positive, zero, or negative). One example is ENZ (or low-index, index-near-zero in literature) materials [1-3], which have attracted significant interest

in the microwave regime and found applications in squeezing electromagnetic energy through very narrow channels [4-7], reflectionless sharp bends [8], design of matched zero-index materials [9], zero-index resonators [10, 11], enhancing optical nonlinear effect [12-14], as well as shaping the radiation pattern [1, 15-18]. Two widely used approaches to fabricate ENZ materials are (1) metal-dielectric stacks or nanoparticles based on the effective medium theory [1, 19-22], and (2) metamaterials based on periodic arrangement of special unit cells [23-25]. Most of reported applications are limited in the microwave spectrum regime due to the complicated fabrication processes or large unit size. The progress on ENZ materials for visible or near-infrared (NIR) light applications is very limited [18, 26-28]. Optically, ENZ is a critical point, where the optical property is transiting between the “dielectric state” (with positive dielectric constant) and the “metallic state” (with negative dielectric constant). Any slight change in the dielectric constant may result in “optical property inversion” or the switch between these two distinct states. Therefore, ENZ provides a unique point to enhance the tunability of optical properties.

Recent works on the optical and EO properties of conductive oxide (COx) in the NIR regime provide a new insight into the fabrication of ENZ materials. The effect of free carriers on an optical material can be approximated by the Drude model, $\epsilon_r = \epsilon_\infty - \frac{\omega_p^2}{\omega(\omega + j\gamma)}$, where ϵ_∞ is the high frequency dielectric constant, ω_p is the plasma frequency, and γ is the electron damping factor. Thus, the ENZ effect can be seen in many materials at $\omega \approx \omega_p / \sqrt{\epsilon_\infty}$. However, the plasma frequencies of most metals are located in the ultraviolet or deep ultraviolet regime owing to their high carrier concentration. Note plasma frequency $\omega_p = \sqrt{\frac{Ne^2}{\epsilon_0 m^*}}$, depending on carrier concentration N , and the effective electron mass m^* . To make ENZ located in the NIR regime, the carrier concentration should reduce to $10^{20} \sim 10^{22} \text{ cm}^{-3}$, which coincides that of COx. The specific value of

carrier concentration can be controlled by the growth/deposition processes and post-annealing conditions [29]. The feasibility of fabrication of COx as ENZ materials has been verified in previous works from different groups [14, 30, 31]. Reference [14] reports the dielectric constant of aluminum-doped zinc oxide (AZO), $\epsilon_r=0.22+j0.26$, at $\lambda=1310$ nm. By controlling the substrate temperature and oxygen flow ratio during the sputtering, the dielectric constant of ITO can be engineered to be negative, zero, or positive for a given wavelength.

2. Epsilon-Near-Zero Conductive Oxide

We fabricated an ITO/HfO₂/Al concentric structure on quartz substrate as shown in the insets of Fig. 1(a). HfO₂ is known for its high DC/RF dielectric constant. It also has high dielectric strength so that a thin insulating layer could withstand a large electric field without breaking down. As a result, HfO₂ may be used to enhance the field effect in the device. The ITO film is deposited by PVD75 RF sputtering [32] on 200 °C substrate with 2% oxygen flow ratio. A 60 s deposition time yields ~10 nm thick ITO film. The sheet resistance of the ITO film on quartz substrate was measured to be ~200 Ω /sq. Following the ITO film, a 10-nm HfO₂ film is produced by the atomic layer deposition (ALD), and finally, 150-nm Al is deposited by evaporation. As illustrated in the inserts of Fig. 1(a), the structure is composed of concentric layers and the dimension of each layer is defined by photolithography and lift-off process, where AZ® nLOF 2020 negative photoresist and Shipley 1165 remover are used, respectively.

To test the optical properties of the ITO film, as shown in Fig. 1(a), the device is placed in a Kretschmann ATR setup, where quartz matching liquid is applied between the substrate and a hemicylindrical prism. The light source is an NIR tunable laser with working wavelengths spanning from 1260 nm to 1620 nm. The laser is first collimated by a GRIN lens and propagates through a linear polarizer (P), to choose the desired polarization for the experiment. A beam splitter

(BS) is used to sample the input power level for monitoring the laser power fluctuation by detector D2. An adjustable aperture (A) is used to control the input light beam diameter. Input beam is then incident into a hemicylindrical BK-7 glass prism and interacts with the ITO/HfO₂/Al structure to be tested. The reflected light is then detected by detector D1. The prism (together with the sample) and D1 are controlled by two motorized rotation stages to allow for different incident angles.

In our previous work [32], the reflectance as a function of incident angle is measured for a given wavelength. In this work, at a specific incident angle $\theta = 70^\circ$, the reflected light power as a function of working wavelength for the transverse magnetic (TM) polarization of light is first measured and plotted in Fig. 1(b). Note that the input light power is kept the same for all wavelengths. As can be seen, there is a dip around $\lambda=1280$ nm in the R - λ plot. This dip corresponds to the maximum absorption, and ENZ should be in the vicinity of the dip. For a TM mode, where the magnetic field is parallel to the multi-layers, the normal electric flux density D is continuous at ITO-HfO₂ interface: $\epsilon_{ITO}(E_{ITO})_n = \epsilon_{HfO_2}(E_{HfO_2})_n$. For lossy medium, the dielectric constant can be formulated as $\epsilon_{ITO} = \epsilon' + \epsilon''$, where $\epsilon'' = \frac{\sigma}{\omega\epsilon_0}$. The dissipation power density, $p_d = \frac{1}{2}\sigma E_{ITO}^2 = \frac{1}{2}\omega\epsilon''\epsilon_0 E_{ITO}^2 \propto \frac{1}{2}\epsilon''/|\epsilon_{ITO}|^2$, is inversely proportional to the magnitude of material dielectric constant when $\epsilon' = 0$. When $|\epsilon_{ITO}| \rightarrow 0$, the electric field in ITO can be greatly enhanced and power dissipation p_d reaches the maximum. As a result, ENZ represents an absorption resonance for TM light. Figure 1(c) shows similar result by measuring the R - λ relation of pure ITO on quartz sample, also demonstrating an ENZ effect of ITO film around $\lambda = 1280$ nm. To quantitatively verify this, we measured the reflectance (R)-incident angle (θ) relation of pure ITO on quartz sample at $\lambda = 1280$ nm and fit the dielectric constant of ITO, as shown in Fig. 1(d). The fitting parameter $\epsilon_{ITO} = 0.209 + j0.477$. Here, if we adopt $\epsilon_\infty = 3.9$, $m^* = 0.35m_e$ [33], then

based on the Drude model, other parameters of ITO can be extracted as $\omega_p = 2.85 \times 10^{15} s^{-1}$, $\gamma = 1.9 \times 10^{14} s^{-1}$, and $N = 8.94 \times 10^{20} cm^{-3}$.

3. Field-Effect Optical Modulation Based on Epsilon-Near-Zero Conductive Oxide

Furthermore, we investigate the field-effect optical modulation based on the ENZ COx. Field effect in metal-oxide-semiconductor (MOS) capacitors plays a key role in field-effect transistors (FETs). A MOS structure functions roughly as a parallel plate capacitor. When a bias is applied across the oxide layer, surface charge can be excited and the induced charge can alter the electric properties of the semiconductor at the oxide-semiconductor interface. Recent works [32, 34, 35] showed that the field effect can also be used to change the optical properties of semiconductors, and gives rise to “field-effect optical modulators” (FEOMs). To enhance the impact of the field effect on light propagation, the ENZ COx is introduced as the active material in a metal-insulator-COx (MIC) structure, where the optical properties of COx can be significantly altered by modulating-electric-field-induced charge.

In the MIC structure, light absorption is controlled by the electric-field induced charge in the active ITO film, which is adjacent to the gate oxide. As shown in Fig. 2(a,b,c), there exist three states for the device, namely original state (O), depletion (D), and accumulation (A). The whole ITO film stays at original state with no applied bias. When positive bias is applied on the ITO film, the free electrons in the ITO film close to insulator-ITO boundary will be depleted. The depletion layer (ITO- d) thickness, l_d , is about few nanometers. Without free carriers, the absorption by the depletion layer is negligible; equivalently, the ITO absorption layer thickness decreases. Thus, the structure becomes less absorptive. On the contrary, if negative bias is applied on the ITO, more free electrons would accumulate on the ITO film surface. The result is that the accumulation layer

is more absorptive. The accumulation layer (ITO-*a*) thickness, l_{ac} , is about few Debye lengths (≤ 1 nm).

In our previous work, we performed DC test on the fabricated device [32], where ITO was fabricated at room temperature and its cross-over wavelength was out of our investigated spectral range. Previous experiment revealed the bias polarity-dependent modulation effect: the absorption of the EA modulator could be increased by negative bias or decreased by positive bias. In particular, the light absorption to DC bias is composed of significant ratio of slow response. The fast response which happens within few milliseconds from the moment of switching on DC bias only contributes to a small portion of modulation depth. The majority of modulation depth is accumulated over a long period of time (over 30 minutes). Similarly in this work, we observed, for the same positive or negative bias, the modulation depth increases with time (up to several minutes), as shown in Fig. 3(a). The slow response may be attributed to the unstable property of ITO under a large DC bias. Similar phenomenon is found more recently in the investigation of a plasmonic memristor based on an MIC structure from another group [36]. The authors attribute the large but slow modulation to the formation of a reversible conductive path in the dielectric layer induced by applied DC voltage, which results in resistive switching. Although a relatively large modulation depth is achieved, the slow response does not contribute to the modulation working at high speed. To this end, we test the FEOM in response to AC sine signals. All tests are performed in the ATR setup as shown in Fig. 1(a).

In this work, a function generator is used to apply an AC sine signal on the device. The photodetector D1 (Thorlabs SM1PD5A) collects the reflected light power and converts it into an electric signal in an oscilloscope. A 50- Ω load is used to convert the photocurrent into voltage, and we record the reflected power as R_o . In this paper, the modulation depth is defined

as $M = (R_{O_{max}} - R_{O_{min}})/R_{O_{max}}$. We first applied a 500 Hz, $V_{pp}=12V$ sine signal on the MIC modulator at a specific wavelength, and an output wave with the same frequency was observed on the oscilloscope. Then, we rotated both the prism and D1 to find the optimal incident angle where it exhibited the largest modulation depth. After the optimal angle was set ($\theta = 70^\circ$ in this work), we examined the output waveform at different wavelengths. Note that the AC bias results in a switch between depletion ($V_b > 0$) and accumulation ($V_b < 0$) states.

In the test, the input light power maintains constant for all wavelengths between 1260 nm and 1410 nm. The output waveforms for different wavelengths are plotted in Fig. 3(b). Three factors define each waveform: the average, $\bar{R}_O$, peak-to-peak variation, $R_{O_{pp}}$, and frequency, f . Our experiments shows the results as follows.

- (1) $\bar{R}_O$ changes with working wavelength. $\bar{R}_O$ decreases from 1260 nm to minimum at 1280 nm, and then increases. This is consistent with the results shown in Fig. 1(b), where a dip exists at 1280 nm.
- (2) $R_{O_{pp}}$ is minimized at 1301 nm, which corresponds to the cross-over wavelength. Starting from smaller wavelength, we can see $R_{O_{pp}}$ reduces to a minimum at $\lambda_c=1301nm$, and then bounces back. In detail, the crests and troughs of the waveform are squeezed as λ approaches λ_c . At the cross-over wavelength, the crests low down to become troughs, while the troughs rise up to become crests. Consider that light absorption would be either increased by electron accumulation or decreased by electron depletion according to the working principle. Here, if the troughs correspond to accumulation and the crests correspond to depletion, the modulation effect must have undergone an inversion at the cross-over wavelength.

(3) When R_{opp} goes to very small, the frequency f nearly doubled. An interesting phenomenon here is that at the cross-over wavelength $\lambda_c=1301\text{nm}$, the frequency of the output wave is double that of the applied signal.

Conceptually, the closer ϵ_{ITO} is to zero, the larger the light absorption will be. Imagine an extreme case that the maximum absorption is achieved at a certain wavelength, both accumulation and depletion would decrease absorption. When the applied voltage runs for one cycle, (A)→(O)→(D), the power reflectance will be (increase)→(normal)→(increase) or “ $\cap_ \cap$ ”, which is roughly similar as the waveform of $|\sin(2\pi ft)|$. Thus, the frequency seems doubled. In detail, in the output waveform in Fig. 3(b), the crests, which correspond to the decrease in absorption would remain; while the troughs, would turn into crests, indicating a conversion from absorption increase to decrease. This conversion is observed when the working wavelength is scanned through the cross-over wavelength, which can be seen by comparing the output waveforms at $\lambda=1298\text{nm}$, 1301nm and 1303nm . The double-frequency waveform is an interaction of accumulation and depletion at the cross-over wavelength, where ITO is at an extreme ENZ state.

According to the observation, the effect of carrier depletion and accumulation on increasing or decreasing absorption is opposite below and above the cross-over wavelength. In Fig. 3(c), when $\lambda < \lambda_c$, the reflectance by depletion is greater than that by accumulation; when $\lambda > \lambda_c$, the reflectance by accumulation is greater than that by depletion. The reflectance curve by the ITO film without bias is located between the curves for depletion and accumulation. This can be explained by how carrier accumulation or depletion quantitatively changes optical property of ITO. When $\lambda < \lambda_c$, ITO layer is at dielectric state with positive dielectric constant. Carrier accumulation would push the ITO-*a* layer towards the ENZ state, resulting in absorption increase. On the contrary, when $\lambda > \lambda_c$, ITO layer is at metallic state with negative dielectric constant. With excess

free carriers, ITO-*a* layer would deviate further away from ENZ state, which decreases absorption. Therefore, the cross-over wavelength λ_c witnesses the inversion of optical property of ITO layer from dielectric to metallic, as well as the inversion of modulation effect in active ITO layer brought by carrier accumulation or depletion.

Despite the ENZ effect shown at the cross-over wavelength $\lambda_c=1301\text{nm}$, the maximum absorption for the MIC modulator is found around $\lambda=1280\text{nm}$. This shift could be owing to the working mechanism of the MIC structure and the ATR measurement setup. To verify this, we calculate $|\epsilon_{\text{ITO}}|$ as a function of wavelength with extracted ITO parameters $\epsilon_\infty = 3.9$, $\omega_p = 2.85 \times 10^{15} \text{s}^{-1}$, $\gamma = 1.9 \times 10^{14} \text{s}^{-1}$. From Fig. 4(a), the minimum value of $|\epsilon_{\text{ITO}}|$, which indicates the cross-over wavelength, is identified at $\lambda=1300\text{nm}$. Then, with the same set of parameters, we calculate the reflectance of the MIC structure based on transfer matrix method (TMM) [37], where both the incident angle θ and wavelength are swept. As seen in Fig. 4(b), at the optimal incident angle ($\theta = 70^\circ$) used in the measurement, the minimum reflectance is located near $\lambda=1280\text{nm}$, which matches the experimental result. In addition, according to the Fig. 4(b), the incident angle where maximum absorption is found varies with wavelength.

Furthermore, modulation can be achieved between any two states among “original”, “depletion”, and “accumulation”. We combine the 500Hz, $V_{\text{pp}}=12\text{V}$ AC signal with a 0V, +6V or -6V DC offset in order to examine A-D, O-A, and O-D modulations, respectively. Figure 5 plots the modulation depth as a function of incident light wavelength for the three modulations. Minimum “A-D” modulation occurs at cross-over wavelength. Peak “A-D” modulation depths are located at about $\pm 30 \text{ nm}$ from cross-over wavelength. The spectrum shift in the three curves is caused by the DC offset-induced optical property change of the active ITO film.

4. Working Bandwidth and Modulation Speed

In the measurement, we have observed similar modulation effect all the way from 1260nm to 1620nm, however, the modulation depth becomes trivial when the wavelength is far away from the ENZ region. For example, as can be seen in Fig. 5, the modulation depth for A-D modulation drops to less than 3% when $\lambda > 1400\text{nm}$. As expected, the ENZ region of the modulator depends on the property of ITO layer, which can be controlled by fabrication process. We have obtained ITO films with sheet resistance from 100 to $400\Omega/\square$, and the resulting cross-over frequency λ_c varies within, or even beyond the wavelength range we investigate. Despite the difference in λ_c due to ITO film property, the EO modulators all behave in a similar manner as illustrated in this paper. Promising applications could be achieved by this property; for example, one could make EO modulators working at a desired wavelength.

Due to a limited working bandwidth of Ge photodiode D1, absorption modulation could hardly be observed at frequencies over 10 kHz. In order to well investigate the working speed of the EO modulator, we utilized an AC-coupled InGaAs photodetector (Newport 818-BB-30A). Figure 6(a) illustrates the reflected power fluctuation of MIC EO modulator under an AC sine electric signal of $V_{pp}=12\text{V}$ working at 1 MHz. The incident light wavelength $\lambda=1280\text{nm}$. Though the EO modulation has been achieved at MHz scale, however, the rapid decrease of modulation depth obviously shows the speed limitation of the MIC EO modulator. As shown in Fig. 6(b), starting from over 10% at 1 kHz, the modulation depth quickly drops below 1% at 500 kHz.

We believe that RC delay is the factor that limits the speed of the device. In order to verify this, we examined the electric circuit of the MIC structure, shown as Fig. 6(c). Here R is a series resistance brought by the function generator and device, $1/G$ is a resistor parallel to the capacitor, and r is a $1\text{ k}\Omega$ resistor externally added to the circuit. We measured the applied voltage on the device V_i as a function of modulating frequency f on condition that the total applied voltage V is

normalized to 1. Plotted in Fig. 6(d), the results show agreement with Fig. 6(b), which indicates that only a very small portion of voltage is applied across the HfO₂ layer at higher frequencies. Based on the equation $V_i = V \frac{1+R(G+j\omega C)}{1+(R+r)(G+j\omega C)}$, we fit the V_i - f curve (Fig. 6(d)) and extract the circuit RC constants as $C=4.13$ nF, $R=111.7$ Ω , and $1/G=32$ k Ω . According to the actual dimension of the sample, the capacitance is calculated as $C = \frac{\epsilon A}{d} \approx 25$ nF ($\epsilon = 25$ is used here for HfO₂). The measured value is several times smaller than the theoretical value, probably due to the over estimation of dielectric constant of insulating layer as well as capacitor fringe effect. However, the RC constants are still too large to allow for a fast modulator. We are optimizing the design and fabrication process for better device performance, and we are expecting a speed boost of several orders by shrinking the size of the modulator from millimeter scale to micro- or even nano-scale [38].

6. Conclusion

To summarize, we have experimentally demonstrated multi-layer MIC EA modulators based on ITO. Our previous investigation on the modulators has shown broadband EA modulation with a relatively large modulation depth. We further explored the tunable property of ITO, and verified its ENZ behavior in the MIC structure around telecom wavelength. Light absorption of the MIC structure can be modulated by both carrier accumulation and depletion. The modulation effect by accumulation and depletion in changing light absorption is opposite below and above the cross-over wavelength, near which the maximum modulation depth can be found. The ENZ working region depends on ITO film property, which can be tuned in fabrication process. The modulation speed is mainly limited by device RC constant, which should be solved by more compact

dimensions and optimized design. The results in this paper may find promising applications in making efficient and broadband EO modulators.

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Figure captions:

Fig. 1. (a) Illustration of the ITO/HfO₂/Al structure and experimental setup. The dimensions are not in scale. Quartz substrate is about 0.5mm thick and the diameter of the hemicylindrical prism is about 100mm. Insets: 3D schematic of the device (upper left) and picture of the fabricated device (upper right). (b) At incident angle $\theta = 70^\circ$, the reflected power as a function of working wavelength at original state (no bias) for the ITO/HfO₂/Al sample. (c) At incident angle $\theta = 70^\circ$, the R - λ measurement for a 10-nm ITO film on quartz. (d) The R - θ measurement for a 10-nm ITO film on quartz for TM polarized light at $\lambda=1280$ nm and the fitting curve.

Fig. 2. (a) The MIC structure under no bias (original). (b) The MIC structure under negative bias (depletion). (c) The MIC structure under positive bias (accumulation).

Fig. 3. (a) The R - θ measurement under DC biases at $\lambda=1280$ nm with increased time (b) Output waveforms of the reflected light power for different wavelengths when the modulator is under $V_{pp}=12$ V, 500 Hz sine electric signal. (c) The reflected power as a function of incident light wavelength for A, O and D states. The inset shows the minimum value of the three curves, which correspond to ENZ.

Fig. 4. (a) Drude model calculation for $|\epsilon_{ITO}|$ as a function of wavelength. (b) Reflectance calculation of the MIC structure by sweeping incident angle and wavelength.

Fig. 5. M - λ relations for O-A, A-D, and O-D modulations under 500Hz, $V_{pp}=12$ V AC signal.

Fig. 6. (a) The output waveform for modulating frequency $f=1$ MHz. (b) Modulation depth as a function of modulating frequency. (c) The circuit diagram for the MIC structure. (d) For a given V , the normalized applied voltage on the device as a function of modulating frequency.

Figures:

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

Figure 6.