Optical control of capacitance in a metal-insulator-semiconductor diode with embedded metal nanoparticles

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This paper describes a metal-insulator-semiconductor (MIS) capacitor with flat capacitance voltage characteristics and a small quadratic voltage capacitance coefficient. The device characteristics resemble a metal-insulator-metal diode except that here the capacitance depends on illumination and exhibits a strong frequency dispersion. The device incorporates Fe nanoparticles (NPs), mixed with SrF₂, which are embedded in an insulator stack of SiO₂ and HfO₂. Positively charged Fe ions induce dipole type traps with an electronic polarization that is enhanced by photogenerated carriers injected from the substrate and/or by inter nanoparticle exchange of carriers. The obtained characteristics are compared with those of five other MIS structures: two based on Fe NPs, one with and the other without SrF₂ sublayers. Additionally, devices contain Co NPs embedded in SrF₂ sublayers, and finally, two structures have no NPs, with one based on a stack of SiO₂ and HfO₂ and the other which also includes SrF₂. Only structures containing Fe NPs, which are incorporated into SrF₂, yield a voltage independent capacitance, the level of which can be changed by illumination. These properties are essential in radio frequency/analog mixed signal applications.

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INTRODUCTION

The combination of insulator sublayers with (or without) embedded metal nanoparticles (NPs) defines the characteristics of metal-insulator-semiconductor (MIS) and metal-insulator-metal (MIM) diodes. Examples include field effect volatile or non-volatile memory (NVM) transistors, linear capacitors, and voltage or illumination tunable capacitors (varactors). Voltage dependent capacitors play an important role in tunable frequency sources and frequency multiplying circuits.¹ The class of optically sensitive capacitors serves mainly as the basis for wireless remote sensing systems, where the capacitor is part of a resonator circuit.^{1–4} A change of the capacitance due to illumination causes a resonance frequency variation in those circuits, which is easily detected. Different optically sensitive varactors have been demonstrated including a planar metal-semiconductor-metal (MSM) diode based on an AlGaAs/GaAs heterostructure^{5–7} and MIS capacitors fabricated on either bulk silicon or silicon on insulator substrates.^{8–10} A photo-impedance sensor comprising photoresistive and photocapacitive sections was constructed on a crystalline CdS substrate, and an optically tunable low-pass filter based on an MIM capacitor made use of polymer CdS as the insulator.^{2,11} Some of the reported devices exhibit a large capacitance photosensitivity but their voltage dependence is highly nonlinear; a fact that limits their application as precision capacitors in different filter circuits and in RF analog mixed signal (RF/AMS) applications. The reported CdS based devices show low photosensitivity; in an MIM

diode, for example, the capacitance changes by only 13% under visible illumination.¹¹

The design of a new optically tunable capacitor as well as its use in a microwave circuit has been presented here. When exposed to 450 nm light, the capacitor shows a change of 13% of its capacitance value associated with the change of the material's relative permittivity. An optically tunable capacitor as well as its use in a microwave circuit has been presented here. When exposed to 450 nm light, the capacitor shows a change of 13% of its capacitance value associated with the change of the material's relative permittivity.

This paper describes the frequency and illumination intensity dependent characteristics of a new capacitor having an MIS structure whose capacitance is insensitive to the applied voltage just as in an MIM device. However, unlike in a conventional MIM diode, the capacitance value is tunable by illumination. The MIS structures employ a multi-layer dielectric stack comprising a thin thermal SiO₂ film and a HfO₂ layer deposited by atomic layer deposition (ALD). The two are separated by SrF₂ sublayers in which Fe or Co NPs are embedded. The devices are fabricated on a bulk silicon substrate.

DEVICE FABRICATION

Three MIS capacitors were fabricated on boron doped p-Si substrates having a resistivity of about 5 Ω cm. 3 nm thermal SiO₂ covers the substrate followed by a 2 nm electron beam gun evaporated SrF₂ film. A thin, 1 nm Fe film was then deposited by sputtering in an Ar plasma environment and was covered by a second, 2 nm thick SrF₂ layer. This stack underwent a rapid thermal annealing (RTA)

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process at 350 °C in a N₂ atmosphere during which the Fe film broke into NPs.¹² Finally, a 20 nm HfO₂ layer prepared by the atomic layer was deposited.

Four additional structures were prepared for comparison. Two lacked the SrF₂ sublayers and one was prepared without the Fe film. The two other structures contained SrF₂; one contained no Co and the second included a 1 nm Co layer. The diodes containing the Fe and Co films as well as the one that used only a SrF₂ sublayer were annealed in the same conditions, as was the main structure. Capacitors with active areas of 1.5×10^{-4} to 5.5×10^{-4} cm² were formed by adding Ti/Pt gates and Ti/Pt/Al back electrodes, deposited by electron beam gun evaporation. Capacitance-voltage (C-V) and impedance-voltage measurements were performed at room temperature in the dark and under illumination at 365 nm wavelength. A HP4192A LF impedance analyzer was used in the parallel or series modes in a frequency range of 5 kHz–5 MHz. The current density voltage (J-V) characteristics were measured using an Agilent 4155C Semiconductor parameter analyzer.

Cross-sectional high-resolution transmission electron microscopy (HRTEM) micrographs are presented in Fig. 1. The TEM samples were prepared using the site-specific *in situ* lift-out method in a dual-beam focused ion beam (FIB) followed by low energy ion polishing.¹³ Prior to the FIB process, the samples were coated with C and Pt to protect them from Ga ion damage. The high resolution micrographs were obtained using a monochromated and aberration-corrected FEI Titan 80–300 kV S/TEM microscope with a measured spatial resolution of 0.07 nm in the TEM mode.

Cross sectional HRTEM micrographs of structures containing iron and a SrF₂-Fe-SrF₂ nanolaminate film are shown in Fig. 1. The low contrast of the material embedded between the SiO₂ and HfO₂ films makes it difficult to distinguish the

NPs from the surrounding matrix, in particular in structure with no SrF₂. Based on the HRTEM images, the estimated diameters of the formed NPs in the MIS and MIM like structures are, respectively, ~3 to 4 and 5 nm, while the thicknesses of the composites are roughly ~4.5 nm and ~5 nm. Figures 1(c) and 1(d) show energy dispersive X ray spectroscopy (EDS) scans of the basic chemical elements in the dielectric stack, measured along the cross sections of Figs. 1(a) and 1(b). The Fe, Sr, and F distribution has smooth maxima and tails that indicate their penetration into both insulator layers. The normalized profile lines [see the inset in Figs. 1(c) and 1(d)] of the Fe and Sr atoms are almost symmetric relative to the maximum, while the F distribution is slightly asymmetric. Note that the average thicknesses of the embedded layers, obtained from HRTEM images, are close to those determined from EDS profile lines at the half width level. Another fact is that the Fe and Sr distribution peaks are located at equal depths, while the fluorine maximum shifts to the left side relative to Fe and Sr curves and hence its penetration is deeper inside the SiO₂ sublayer [see the inset in Fig. 1(d)]. In accordance with Ref. 14 at an elevated temperature, the SrF₂ decomposes and the F part penetrates to the Si. The vacancies of Fluorine are then trapped by oxygen during the HfO₂ deposition process. It is assumed that diffusivities of the Sr and Fe atoms into the SiO₂ are smaller than that of Fluorine.¹⁵

The HfO₂ ALD deposition process causes the oxidation of the Fe films in both structures, while the Fe film which is surrounded by SrF₂ layers is transformed to an oxy-fluoride like compound SrFeO_x-F_y where part of anions (oxygen) sites are occupied by fluorine atoms.^{15,16} The result is fluorine-induced Feⁿ⁺ or mixed Feⁿ⁺/Feⁿ⁺¹ ions, where $n=2$ or 3,^{15,17} while in MIS structures, a Fe_xO_y layer is formed. It is seen from Fig. 1(d) that the Fe/O and Fe/F

FIG. 1. Cross sectional HRTEM micrographs of the (a) MIS structure with embedded Fe and (b) MIM like structure with embedded SrF₂-Fe-SrF₂ nanolaminates; EDS average at. % composition profiles for Hf, Si, O, Fe, Sr, and F along the HRTEM cross sections of (c) MIS and (d) MIM like structures versus distance from the HfO₂ sublayer to the Si substrate. EDS data were acquired at steps of ~0.25 nm along the line-scan. In insets of (a) and (b) are magnified images of the NPs of the embedded materials. (c) and (d) show the normalized elemental distributions in the embedded films obtained by EDS.

atomic ratios near the flat maximum are 1.7 and 2.6, while on the right (close to the SiO₂ layer) and left (close to the HfO₂ sublayer) sides these ratios are 1 and 0.5 and 2.2, respectively.

Experimental results

Figure 2(a) describes measured capacitance density versus voltage measured at 5 kHz to 5 MHz in the dark and under illumination at 365 nm with an intensity of 485 μ W. The capacitance density always increases when the frequency is reduced and the illumination intensity increases; it varies slightly with voltage in the dark, mainly at low frequencies, but is completely voltage independent at any frequency under illumination. The capacitance density value is about twice as large under illumination as compared to the dark value, at any frequency.

The dependence of the zero bias capacitance density on illumination power is shown in Fig. 2(b) for two frequencies: 10 kHz and 1 MHz. The capacitance density increases monotonically with illumination power and is larger for the lower frequency.

The effect of frequency is described in Fig. 2(c), which shows that the capacitance density decreases with increasing frequency in both illumination regimes. The reduction from 10 and 90 kHz is monotonic in the dark and under illumination, respectively, while at lower frequencies, the capacitance dispersion is sublinear. The capacitance rate of change is significantly larger than that of ordinary MIM structures including devices where the insulator comprises single SiO₂, Si₃N₄, or High-k insulator layers or their nano-laminate combinations.^{18–22}

The monotonically decreasing capacitance density versus frequency curves are approximated by a simple power law $k \propto C \propto f^{(m-1)}$ (where $0 < m < 1$) as suggested in Ref. 23 for the dispersion of the dielectric constant (k). Such a fit is shown by solid lines in Fig. 2(c). The average value of the parameter m in the dark and illumination regimes changes from 0.615 to 0.7, respectively. The parameter m enables us to estimate the dielectric loss tangent according to $\tan(\delta) = \cot(m\pi/2)$ (IEEE Transactions on Nanotechnology^{23,24}). In the dark, the loss is determined to be approximately 0.72, while under illumination, it reduces to 0.52. These values are larger than commonly achieved losses in conventional insulator based MIM diodes which are smaller than 0.1.^{18–22}

The change of $\tan(\delta)$ (or m) with frequency can be modeled using a power law approximation, where the value of m is found from $(m-1) = \frac{d \ln(C_{V=0})}{d \ln(f)}$ (Ref. 25) extracted from C- f curves [Fig. 2(c)]. The extracted $\tan(\delta)$ versus frequency is non-monotonic in both the dark and illuminated regimes as seen in solid curves in Fig. 3. Also shown in Fig. 3 (in symbols) are $\tan(\delta)$ versus frequency curves obtained from the impedance measurement.²⁶ The two sets of curves exhibit rather well with the extremum loss value appearing at 90 and 650 kHz, in the dark and under illumination, respectively, similar to Ref. 27.

The C-V data were normalized to zero voltage capacitance (C_0) and fitted by a simple power law $(C(V)-C_0)/C_0 = \alpha V^2 + \beta V$, where α and β represent quadratic and linear

FIG. 2. (a) C-V characteristics of the MIS structure with Fe surrounded by nanolaminate SrF₂ films measured in the dark and illumination regimes at different frequencies; (b) Zero bias capacitance density versus illumination power measured for two values of frequency: 10 kHz and 1 MHz; (c) Zero bias capacitance density versus frequency measured in the dark and illumination regimes. Symbols are experimental, while solid lines are a result of power exponent approximation.

voltage coefficients of capacitance (VCC), respectively.²⁸ The frequency and illumination power dependencies of α and β were extracted from the measured C-V characteristics and are shown in Fig. 4.

The dependence on frequency is shown in Fig. 4(a). The two curves with closed symbols represent α in the dark and illuminated cases. In both α drops, at any frequency, α values

FIG. 3. Frequency dependences of the dielectric loss tangent measured in the dark and illumination regimes and determined in accordance with Refs. 23 and 24 (solid curves) and from impedance measurement.²⁶

are lower under illumination as compared to the dark regime. It changes from 800 ppm/V² to 230 ppm/V² (in the dark) and 130 ppm/V² to almost 5 ppm/V² (under illumination) when the frequency increases from 5 kHz to 5 MHz. The parameter does not depend on the actual capacitance value but rather on the deviation relative to zero bias at both voltage polarities. As this deviation reduces with illumination, so do the α values. The linear VCC β behaves similarly in the dark reducing from 10 000 ppm/V to 3300 ppm/V over the same frequency range. Under illumination, β changes from 3000 ppm/V to 1700 ppm/V. The dependence of the quadratic VCC on illumination power was characterized at 10 kHz and 1 MHz as

FIG. 4. (a) Frequency dependences of the quadratic and linear VCC of MIM like capacitor measured in the dark and illumination regimes; (b) quadratic VCC versus illumination power at two different frequencies.

shown in Fig. 4(b). At both frequencies, α reduces rapidly with illumination power reaching rather low values at a power of 485 μ W. The low values of the quadratic coefficient α , especially under illumination, are crucial to high performance varactors and are significantly more important than the linear coefficient β .²⁹

DISCUSSION

The capacitance density change in MIS or MIM diodes stems from several possible reasons such as an extrinsic series resistance or a non-Ohmic back contact, large interfacial or insulator bulk traps, leakage currents (resulting from tunneling through the insulator layer), and dielectric constant variations with frequency.³⁰ In order to examine the possible influence of any of those factors on the present structure, we consider two additional MIS capacitors, both comprising SiO₂ and HfO₂ films where only one contains also embedded Fe NPs but without SrF₂.

All studied MIS structures exhibit in the dark and under illumination a very small leakage current density of 100 nA/cm², measured at ± 5 V and a breakdown voltage of 12 V, as seen in Fig. 5. The J-V characteristics represent a transient phenomenon typical of MIS or MIM devices that show a hysteresis due to a large density of interfacial or bulk traps within the insulator stack. These characteristics are the same as for the MIM like device considered above; however, their C-V characteristics differ significantly.

C-V characteristics measured at 10 kHz and 1 MHz are shown in Fig. 6. These are classic C-V characteristic of MIS capacitors, which for a sweeping voltage of ± 5 V have a small hysteresis of 0.35 V in the dark when no metal NPs and SrF₂ sublayer are present [see Fig. 6(a)]. The hysteresis is large, 7 V and 1.9 V, respectively, for structures with embedded Fe and Co NPs [see Figs. 6(b) and 6(c)]. These hysteresis values do not vary by frequency.

Under illumination and at small frequencies, the nature of the curves changes in the inversion regime (positive biased). Here, the effect of change of the inversion capacitance density enhances. In the diode with no NPs, the hysteresis increases to 1.3 V at 10 kHz, while it is equal to 0.6 V at 1 MHz. For the structures with Fe and Co NPs, the hysteresis

FIG. 5. Current density versus applied voltage measured in the dark and under illumination regimes of the MIS structure with embedded Fe NPs.

FIG. 6. Capacitance density voltage characteristics of MIS structures comprising a double layer $\text{SiO}_2\text{-HfO}_2$ insulator stack: (a) no and (b) with Fe NPs; (c) with Co NPs surrounded also by SrF_2 layers and (d) with the SrF_2 film, but without metal NPs. Measurement in the dark and under illumination was carried out at 10 kHz and 1 MHz.

becomes, respectively, 7.5 V and 2.1 V independent of the frequency. For this structure, illumination has but a small effect on the hysteresis due to the doping level of the Si substrate, which is larger by an order of magnitude compared to the diode studied in Ref. 31. Finally, the structure that contained a SrF_2 sublayer but no metal NPs also shows an MIS type C-V dependence [see Fig. 6(d)] with a hysteresis width smaller than that when metal NPs are included.

The hysteresis is caused by formation of a transition region between SiO_2 and HfO_2 due to the incorporation of Fe sublayers, which after annealing and following ALD processes transforms to positive Fe ions inside of NPs of different valences that serve as trap charge sites, just as in many non-volatile memories.^{12,32,33} The constant accumulation capacitor (and in turn the dielectric constant) in the 10 kHz to 1 MHz range (independent of illumination) suggests that the series resistance does not play a meaningful role. Illumination does not change the accumulation capacitance [see Figs. 6(a) and 6(b), respectively, no and with Fe or Co NPs]. The change of the accumulation capacitance is known to be due to a reduction of the large series resistance under illumination and is important only at high frequencies.³⁴ Here, the situation is different; the measured accumulation capacitances [see Figures 6(a)–6(c)] of structures with similar semiconductor substrates and electrode material are determined by the effective dielectric constant of the insulator stack, which fits a calculation that uses the series capacitance model considering the measured dielectric constant of the insulator sublayers. At reverse bias, the inversion capacitance varies with frequency and illumination intensity due to a lifetime reduction of the photogenerated minority carriers, relative to the lifetime of thermally excited carriers in the dark regime.^{34,35} This enhances the probability of carriers to follow the applied AC voltage at high frequencies.

Unlike in a MIM diode, a bias level near flat band for a MIS capacitor causes the C-V curves to drop sharply from

accumulation to inversion or vice-versa as the voltage sweeps, independent of frequency and illumination intensity. This is because the depletion region width varies by the sweeping voltage.

Since the C-V curve does not change from accumulation to inversion, we conclude that mixing of the SrF_2 and Fe laminate layers during the RTA process is the main reason for the major change that the C-V curves undergo (compared to the MIS structures at hand or any conventional MIM diode) with illumination and frequency. In the present structures, illumination doubles the value of the voltage independent capacitance; hence, we conclude that the series resistance plays no role even though the device is based on the same substrate, insulator sublayers, and gate electrode as in the MIS structures.

The shift with frequency of the maximum $\tan(\delta)$ seen in Fig. 3 denotes a change of the dielectric relaxation time, which is determined from the frequency corresponding to the maximum.³⁶ The absence of a sharp maximum in the $\tan(\delta)$ versus frequency curves in Fig. 3 with broadened maximum indicates the presence of trap sites with different relaxation times.^{37,38} Namely, the different dielectric relaxation times of traps are the reason behind the observed frequency dispersion of the capacitance density as well as the VCC coefficients.³⁹ Similar $\tan(\delta)$ extrema shifts were observed at different temperatures in Ferrites.²⁷ In accordance with Refs. 24, 27, 36, 40, and 41, temperature variations cause excitation of trap states in ferrite based capacitors, which enables in turn polarization of the local sites. This process is developed by charging/discharging of traps due to injected carriers as well as by a hopping process. In ferrites, such peaking behavior in $\tan(\delta)$ occurs when the hopping frequency of electrons between Fe^{2+} and Fe^{3+} is equal to the frequency of the AC applied field.¹⁷ Finally, the effect of the electric field polarization of the insulator stack and the leakage current are the main factors causing the deviation of the

capacitance density from the zero bias value and hence of the voltage dependence of VCC.³⁹ In the present case, the effect of leakage current on VCC is minor due to its small value (see Fig. 5).

Illuminated CdZnTe crystals, surrounded by two plate electrodes, were discussed in Ref. 42. An enhancement of low frequency capacitance with a strong dispersion was demonstrated resembling the effect of an elevated temperature.²⁷ We postulate that the peculiarities of the MIS structures we describe originate from similar effects as described in Refs. 36 and 42. It was assumed that illumination polarizes local traps within the material, which form an electric field around of the charged sites. The total electric field induced by illumination is proportional to the ionized traps whose density is significantly larger than that of free carriers in the crystal and hence cannot be compensated.

The capacitance density reduction (and consequently of the effective dielectric constant) at frequencies larger than 10 kHz (in the dark) and 100 kHz (under illumination) shown in Fig. 2(c) and the non-monotonic change with frequency of the $\tan(\delta)$ (see Fig. 3) resemble the mechanism governing interfacial (electronic) polarizability in a frequency range of 1 kHz to 5 MHz.⁴³ We propose that following the RTA and ALD processes, the laminate layers SrF₂-Fe-SrF₂ became the dominant component of the entire dielectric stack in determining the polarization in contrast to what is observed in the MIS structure described above including only oxidized Fe molecules, which are embedded into the insulator stack. The EDS profile lines [Fig. 1(d)] indicate that the transition region between the SiO₂ and HfO₂ includes Fe ions of different valence states, while the composition of the embedded sublayer along the cross section is non-uniform. Therefore, the charging ability of Fe ions is high in the vicinity of the SiO₂ film (where the fluorine concentration shows a relative maximum at the same depth where the oxygen peak is minimized) and is low at the boundary of the HfO₂ layer. The displacement of electrons relative to Fe ions can create a dipole (electronic or interfacial polarization effect). Carriers can move towards the ions by injection from the substrate or by hopping from the boundaries of the crystalline regions, which are an efficient source of trap sites. These can cause the non-monotonic behavior of $\tan(\delta)$ with frequency, similar to that described for ferrites.¹⁷ The trap sites at the grain boundaries can induce a strong internal electric field, which attracts minority or majority carriers at the surface of the semiconductor. The RTA and ALD fluoridation-oxidation process of the Fe atoms causes high density positive charge sites of the oxygen reduced Fe ions. The induced traps are polarized toward the semiconductor surface due to an internal electric field even at zero applied bias. Positive charges are pushed out from the traps and induce band bending upward of the p-Si and hence result in the formation of an interfacial inversion layer, even in the dark regime. At positive gate bias, the trap polarization and the consequent band bending are enhanced. Minority carriers are attracted, therefore, from the Si bulk, thereby enhancing the inversion layer population. These processes cause the diminishing influence of the depletion region width on the total capacitance value. Consequently, the MIS structure transforms to an MIM like

device where the capacitance is independent of the applied voltage. The photogenerated minority carrier density in the inversion layer at the semiconductor-insulator interface is increased under illumination. At the same time, their lifetime is decreased so that their response to a high frequency applied AC signal is enhanced.³⁴ More minority carriers are injected, therefore, from the inversion layer toward the NP-boundaries. This intensifies their capture by the positive charge trapping sites, compared to the dark regime. The capacitance increases due to shrinking of the depletion region width,^{34,44} which is caused by compensation of the depletion charge (ionized acceptors) due to photocarrier generation. This is similar to what occurs under illumination in C-V characteristics of the MIS structures [see Figs. 6(a)–6(c)] in the depletion regime (positive bias on the gate electrode). The photocapacitive effect that increases the depletion region capacitance with illumination together with a widening of the hysteresis in the C-V curves of non-volatile memory MIS structures with embedded nanoparticles or presence of the charge trap states of any types is well-known.^{31,45,46}

CONCLUSION

We have demonstrated a new type of varactor comprising an MIS structure with embedded Fe surrounded by SrF₂ nanolaminates whose capacitance is essentially bias independent as in an MIM diode. This originates due to the fluoridation-oxidation process of the Fe atoms that causes the formation of the oxygen reduced Fe ions. These, in turn, induce dipole type traps with an electronic polarization. Illumination increases the photogenerated minority carrier density and reduces the depletion region width. This process is enhanced by the attraction of photo-excited minority carriers from the inversion layer by Fe-SrF₂ NPs, which adds to the band bending of the semiconductor. The varactor exhibits an extremely low quadratic voltage coefficient. Our comparison with other MIS capacitors comprising metal NPs with or without SrF₂ sublayers leads to the unequivocal conclusion that only devices comprising the combination of Fe and SrF₂ turn the MIS structure to a photosensitive MIM-like capacitor. The possible applications of the MIM capacitors are detailed in Ref. 47. The main advantage of the MIM-like device over the MIS/MSM varactors is the fact that they offer high capacitance precision in analog circuits due to their higher linearity. This is extremely critical in RF/AMS applications (in particular filter circuits) as it determines the accuracy of frequency bandwidths. The observed photosensitivity of the structures we report expands their potential in optoelectronic circuits, operating at moderate frequencies, mainly as light sensitive variable capacitors in remote sensing circuits.

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