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► **To cite this version:**

Kremena Makasheva, H. Wang, Bernard Despax, Laurent Boudou, F. Coccetti, et al.. New materials versus new design: study on the physico-chemical and electrical properties of thin SiO_xN_y layers for their use in RF-MEMS switches. MGE'2010: 5e Colloque sur les Matériaux du Génie Electrique, Aug 2010, Montpellier, France. pp.104-109. hal-03942707

HAL Id: hal-03942707

<https://hal.science/hal-03942707>

Submitted on 18 Jan 2023

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New materials versus new design: study on the physico-chemical and electrical properties of thin SiO_xN_y layers for their use in RF-MEMS switches

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Abstract: In this work we present results from an experimental study on the physico-chemical and electrical properties of thin dielectric layers (SiO_xN_y) deposited using a plasma assisted process. An application of those dielectric layers in RF-MEMS switches is aimed at. We look for a better understanding of dielectric charging phenomenon as it is known to be the main cause for the failure and low reliability in the actuation of RF-MEMS switches. In order to improve the performance of dielectric layers used in RF-MEMS we have tested a new concept: instead of elaborating new materials we assembled a dielectric multi-layer structure that gains from design rather than from composition. To achieve this we have started with a characterization of the mono-layers deposited singly. The multi-layer structure was found to be a promising candidate for suitable modulation of the conductive properties of the deposited dielectric layers for their use in RF-MEMS switches.

INTRODUCTION

Dielectric materials are regularly exploited in a large variety of solutions in electrical engineering. Quite often they make part of these systems as components on different scale levels: from millimeter to nanometer size. The insulating materials in use require a deep knowledge on their composition, physical and electrical properties [1]. For example, the dielectric charging phenomenon is one of the challenges to meet because of the necessity to control or to avoid it. The dielectric charging is an obstacle for many electrical engineering solutions as it modifies the control parameters and in certain cases it can even lead to complete loss of command of these systems. This is also the case with the performance of MicroElectroMechanical Structures – RF-MEMS capacitive switches. Currently, the dielectric charging of RF-MEMS switches is the main cause of failure and low reliability of capacitive switches with electrostatic actuation [2]. In general, in our work, we are interested in revealing the mechanisms at the origin of dielectric charging phenomenon and in this case, our effort is directed to an improvement of the reliability of technological devices containing thin insulator layers. The purpose of this work is to elaborate thin dielectric layers with adequate performance for their use in

RF-MEMS capacitive switches. To achieve this, instead of elaborating new materials, we choose to check a new concept: we initiated a study on the properties of a system of dielectric materials that will gain from design rather than from composition. Our research effort is directed towards the implementation of thin dielectric layers either constructed as multi-layer systems or with gradual variation of their properties. Both steps the deposition and the characterization (structural and dielectric ones) of those dielectric systems are envisaged in our work. Before going to the assembly, we should know well and to be in command of the properties of the dielectric mono-layers. Thus we first characterized mono-layers with different chemical compositions, deposited singly on a substrate before evaluation of the performance and reliability of the multi-layer systems.

In this work we present results from experimental study on the physico-chemical and electrical properties of thin dielectric silicon nitride-oxide layers (SiO_xN_y). These dielectric layers are deposited by using pulsed plasma-enhanced chemical vapor deposition (PPECVD). Details on the deposition technique and the experimental conditions are given in the next section. A brief description of the applied diagnostic methods is then presented. Later on in our paper, we will report the obtained results, which will be immediately followed by a discussion. Finally, we will give our concluding remarks.

EXPERIMENTAL DETAILS

Materials

To elaborate our dielectric layers, we use pulsed plasma-enhanced chemical vapor deposition (PPECVD) [3, 4] as a deposition technique. PPECVD is known to be technologically compatible, highly efficient, performing, reproducible and quite flexible so as to elaborate materials with different composition and high quality. Thin sub-stoichiometric silicon nitride-oxide layers (SiO_xN_y , $x < 2$, $y < 1$) are deposited on Au/Ti (200nm/50nm) metalized Si (100) wafers in the plasma of radiofrequency ($f = 13.56$ MHz) capacitively coupled discharge sustained with power of 5 W. The CW (continuous wave) signal is modulated by square pulses with period $T = 400$ ms. The pulse duration is 100 ms (duty cycle $\tau = 0.25$). The gas mixture contains SiH_4 , N_2O

and He and the total gas pressure in the discharge reactor is $p = 65$ Pa. The total gas flow is 210 sccm. Helium component in the mixture is fixed to 40%. The gas flow ratio $\gamma = \text{N}_2\text{O}/\text{SiH}_4$ allows adjusting the silicon concentration in the film. In our case, it varies between $\gamma = 100$ and $\gamma = 2$. The lower the gamma ratio, the richer in silicon the dielectric film is. High γ value ($\gamma = 100$) means that the dielectric layer owns properties close to that of a SiO_2 layer. The metalized silicon substrates are clamped on the grounded electrode. The substrate temperature is kept to $T_s = 573$ K during the deposition process. Different times for deposition have been used according to the envisaged layer composition and thickness.

Single layers with different compositions (different γ values; $\gamma = 100, 25, 10$ and $\gamma = 2$) have been deposited separately on Au/Ti metalized Si wafers. A multi-layer structure (4 layer structure) containing layers with the same γ values as for the single layers, was deposited on an intrinsic silicon (100) substrate for simplicity of the following material characterization.

Applied diagnostic methods

Several diagnostic methods have been applied to determine the physico-chemical and electrical properties of the deposited SiON layers. The results presented in next section are from diagnostic of these layers in ellipsometry, transmission electron microscopy TEM, X-ray photoelectron spectroscopy as well as from electrical measurements consisting of transient DC charging/discharging current measurements. Details on each diagnostic technique are specified when the results obtained from that type of diagnostic are discussed.

RESULTS AND DISCUSSIONS

Some of the results from our study on thin SiON layers have already been disclosed elsewhere [5]. In this paper we will complete the study with new results. We have revealed in Ref. 5 that the as-deposited SiON layers, singly or arranged in a multi-layer system, are in an amorphous phase. A significant concentration of amorphous Si-nanoparticles (a-Si = 26%) have been found in the SiON matrix of the layer with $\gamma = 2$ compared to the composition of the layer with $\gamma = 25$, where the a-Si nanoparticles are less than 1%. There is not a-Si nanoparticles in the $\gamma = 100$ thin layer. The SiON layer in that case is close as composition to a SiO_2 layer. These results are consistent with the results for the volume resistivity obtained from electrical characterization. The variation of the calculated volume resistivity as a function of γ shows a significant decrease of resistivity when γ is decreased. Furthermore for γ values under 10, the decrease of volume resistivity is sharp, and the value for $\gamma = 2$ is much than 6 orders of magnitude lower compared to that for $\gamma = 100$.

Before going to the presentation of our further results on the study of silicon nitride-oxide thin layers we will pay

attention to some peculiarities in the layer's characterization when metalized substrates are utilized.

Key steps to do when using metalized substrates

This part is especially devoted to the characterization of dielectric layers deposited on metalized substrates. It is necessary step because of some particular points to survey during the diagnostic that can influence the procedure and induce uncertainty in the obtained results. As the reliability of RF MEMS capacitive switches is targeted in our study we use Au/Ti metalized substrates. In the current RF MEMS technology the transmission lines are golden made [6]. The reasons for using gold are common: low resistivity, which allows minimum transmission losses and compatibility with planar microfabrication processes. The Ti layer is to guarantee adherence between the Si substrate and the Au layer. The PPECVD process that we use for deposition of SiON layers demands certain time for evacuation of the discharge reactor. We have estimated that an overnight pumping with simultaneous heating ($T_s = 300^\circ\text{C}$) assures well enough high vacuum ($p = 2 \times 10^{-4}$ Pa) as initial conditions for the deposition process. Moreover it implements annealing of the golden layer. To perform a good diagnostic of the deposited dielectric layers we should first study the modifications that the deposition technique induces in the Au/Ti layers deposited on the substrate and then adapt the diagnostic protocol.

Spectroscopic ellipsometry

Fig. 1 shows light penetration depth in the Au layer for as-deposited Au-layer and one after annealing. It is obtained from spectroscopic ellipsometry measurements. We have used a SOPRA GES-5 spectrometer in the spectral range 250-850 nm at incident angle of 69° degrees. One can see that the long time pumping of the plasma reactor not only assures good initial conditions for the deposition process but improves the transmission properties of the golden layer.

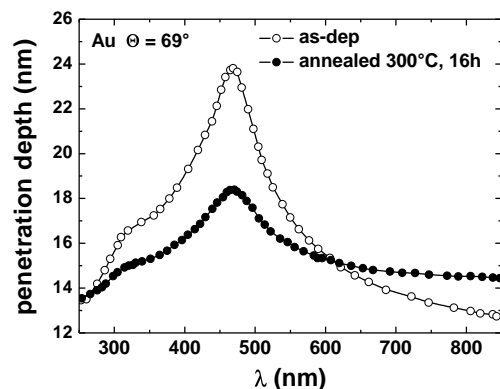


Figure 1. Light penetration depth in the golden layer for the same wavelength range as used in the ellipsometry measurements: as-deposited golden layer (open symbols); golden layer after annealing at 300°C during 16 hours (closed symbols).

To adapt the procedure in ellipsometry we had first to determine the angle of incidence, which is the most

appropriate one to perform the measurements. In general, it is the Brewster's angle. The Brewster's angle for Au varies strongly as a function of the wavelength and this variation determines the need of such assessment.

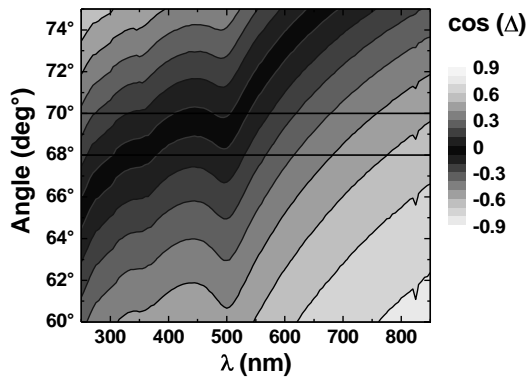


Figure 2. $\cos(\Delta)$ -measurements as a function of the wavelength for different incidence angles of the golden layer after annealing.

In ellipsometry the measured quantities are: $\tan(\Psi)$, which represents the ratio between modulus of the reflection coefficients and $\cos(\Delta)$ that gives the phase difference induced by the reflection. $\cos(\Delta)$ is extremely sensitive to the angle of incidence. To decide at what angle the measurements must be done it is important to have a good sensitivity all along the wavelength range, which is equivalent to a zero value of $\cos(\Delta)$. Figure 2 presents $\cos(\Delta)$ measurements as a function of the wavelength for different incidence angles of the golden layer after annealing. It can be seen in Fig. 2 that the best choice of incident angle lays in the range $68^\circ - 70^\circ$. Our choice was 69° .

After the spectra acquisition, it is the simulation procedure of the recorded spectra to be performed. We have used WinElli, software provided by SOPRA LAB [7]. The small light penetration depth (18 nm is the upper limit) in the annealed Au-layer (Fig. 1), compared to the Au-layer thickness (200 nm), means that we can assume the Au-layer as bulk material, consequently as substrate in the simulation of the recorded spectra. Because of the modifications in the properties of Au-layer induced by annealing during the pumping time, the optical indices n and κ (n – refractive index and κ – extinction coefficient) proper to the annealed Au-layer must be used in the regression of the measured spectra of SiON layers. To extract the needed n , κ values that will be further used in the regressions we apply the bulk calculation, which assumes that the sample (the Au-layer in this case) is a bulk material.

Transmission Electron Microscopy

Another step to do before characterization of the SiON layers is to verify that there is not diffusion of Au atoms into the deposited layer. This additional check comes out from the deposition conditions as the substrate temperature is kept to 300°C during the pumping time and the deposition one; a value close to the eutectic temperature for Au, which is 363°C [8].

We have used transmission electron microscopy (TEM) to observe the SiON layer deposited on Au/Ti Si-substrate in a cross sectional view. To achieve the observation we have prepared specimens transparent to electrons in accordance with the standard procedure: mechanical polishing and Ar^+ ion milling. The observation in bright field mode [9] is performed on a field emission TEM, PHILIPS CM30.

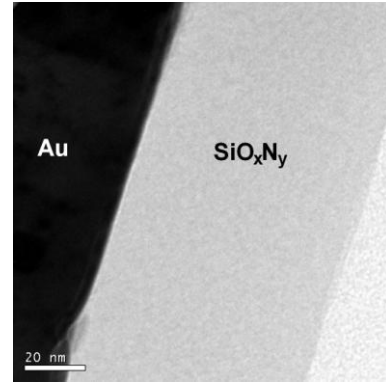


Figure 3. Bright field TEM image of $\gamma = 100$ layer.

A Bright field TEM image of SiON layer deposited with $\gamma = 100$ is shown in Fig. 3. It can clearly be seen that there is not Au-clusters penetrating in the deposit. Hence the substrate temperature used in the deposition does not provoke contamination of the SiON layers with Au-atoms. A layer thickness of 78.0 ± 1.0 nm was obtained from this technique. In ellipsometry, following the procedure described above and applying the Forouhi-Bloomer approximation for the optical dispersion relation [10] in the modeling, we have obtained a value of 79.6 ± 1.1 nm for the same layer, which is close to the value obtained in TEM. Thus, it justifies and confirms all the steps in the procedure of ellipsometry characterization of dielectric layers deposited on metalized substrates.

To observe the multi-layer structure we have used a field emission TEM, FEI TecnaiTM F20. This microscope is equipped with a corrector for spherical aberration and the Gatan Imaging Filter (GIF) TRIDIEM. The operation voltage is 200 kV.

For EFTEM, we have detected the Plasmon signal (at 17 eV) associated with the Si nanoparticles [9]. All the Si-nanoparticles with diameter larger than 1 nm, either amorphous or crystalline, are visible and appear as bright in a dark background (the SiON matrix). EFTEM image of the multi-layer structure is shown in Fig. 4. The Si substrate appears in bright. We can easily distinguish the 4 layers ($\gamma = 100, 25, 10$ and 2) deposited on it, starting with the layer that does not contain a-Si. The silicon rich layer ($\gamma = 2$), is on top of the structure.

The assembly of the multi-layer structure containing layers with different conductivities (Si-concentration) implies the option to be in command of the dielectric charging in RF-MEMS switches.

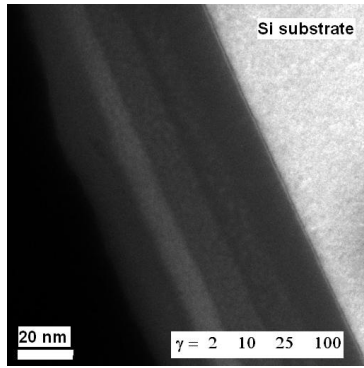


Figure 4. EFTEM image of the multi-layer structure.

X-ray photoelectron spectroscopy

To enlarge the structural analysis of the SiON dielectric layers and complete the results [5] obtained from Fourier Transform InfraRed Spectroscopy (FTIR), we have performed X-ray photoelectron spectroscopy (XPS) measurements by using a VG ESCALAB 220-iXL spectrometer. Here it should be pointed out that the XPS technique is sensitive to elements except hydrogen or helium. The layer surfaces of the deposits were excited by energetic Bremsstrahlung radiation produced in the MgK α anode (1253.6 eV). The kinetic energies of the electrons emitted from the surface were collected at a constant energy resolution of 1 eV. The energy step used to record the spectra was 0.2 eV. Two SiON layers ($\gamma = 100$ and $\gamma = 2$) have been characterized. Since the thin film with $\gamma = 100$ is rather nonconductive, electron compensation was used during the measurement.

First the surfaces of as-deposited layers have been analyzed. Then they were sputtered with accelerated argon ions (Ar^+) under a pressure of $p_{\text{Ar}} = 2.5 \times 10^{-5}$ Pa at theoretical rate of about $v = 0.1$ nm/s. Under the low-energy Ar^+ sputtering in a zone of about 150 μm in diameter and depth of 3 – 5 nm, the surface contaminant species were completely removed and the individual atomic concentrations in the bulk were obtained from the integrated peaks after calibration of the background level. We have used the C1s photoelectron peak area as reference. The deconvolution was done by AVANTAGE of ThermoFisher Scientific with Gaussian/Lorentzian functions (G/L ratio: 25% to 70%). The composition of each layer was evaluated from the intensity ratio of the Si2p, N1s and O1s photoelectron lines by using their sensitivity factors.

The chemical composition of the as-deposited SiON layers with $\gamma=100$ and $\gamma=2$ are summarized in Table 1. Then, we provide information about the different chemical bonds and valence states on the SiON surfaces (Fig. 5) and in the bulk (Figs. 6, 7) after sputtering. The thicknesses of the SiON layers were found to be 86.7 nm and 82 nm for $\gamma=100$ and $\gamma=2$, respectively.

The results in Table 1 show that the surfaces of both SiON layers were carbon and oxygen contaminated due to their exposure to air. Furthermore, the composition of the layer with $\gamma=100$ is close to SiO_2 , while the one with

$\gamma = 2$ is silicon rich. These results coincide with the results for the same layers obtained from FTIR [5].

It is now generally accepted for a SiO_x layer ($0 < x < 2$) that the Si2p spectra can be interpreted by five oxidation states Si^0 , Si^{1+} , Si^{2+} , Si^{3+} and Si^{4+} [11]. Thus the SiO_x matrix is formed by Si-($\text{Si}_{4-n}\text{O}_n$) tetrahedras with $n = 0-4$. In this scheme the transition from a pure amorphous Si (a-Si, $n = 0$) where the Si-atom is bounded to four adjacent Si-atoms to a pure a- SiO_2 ($n = 4$) where the Si-atom is bounded to 4 oxygen atoms is done by consequent replacement of Si-atom by O-atom for each oxidation state. The binding energy range of the transition $\text{Si}^0 \leftrightarrow \text{Si}^{4+}$ is between 99.3 eV to 103.3 eV with a shift of approximately 1 eV per oxidation state [11].

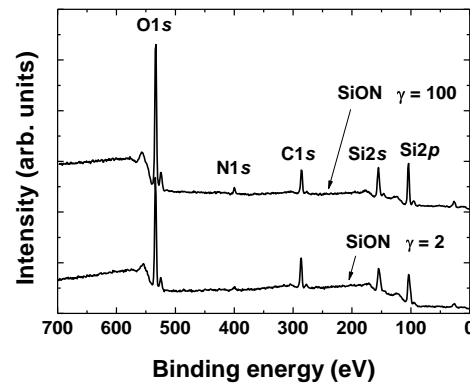


Figure 5. XPS spectra of the layer surfaces: in SiON with $\gamma = 100$ and $\gamma = 2$.

SiON layer	type	C	O	N	Si
$\gamma=100$	surface	16	52.1	3.4	28.5
$\gamma=2$	surface	20	42.9	2.8	34.3
$\gamma=100$	depth	×	60	4	36
$\gamma=2$	depth	×	35	5	60

Table 1. Summary of XPS measurements (at.%) for SiON thin layers deposited on a Au/Ti Si substrates.

Figure 6 presents the results for the SiON layer with $\gamma = 100$. It can be seen (Fig. 6(a)) that the Si2p photoelectron peak includes only two components: the two highest oxidation states, the peak at 102.6 eV with FWHM = 2.0 eV and the one at 103.6 eV FWHM = 2.2 eV. Both peaks are probably slightly perturbed because of the small amount of N-atoms present in the layer composition. This perturbation can be imposed by replacement of an oxygen atom in the Si-($\text{Si}_{4-n}\text{O}_n$) tetrahedral structure by a nitrogen atom [11]. The large FWHM, in this case, confirms that the layer with $\gamma = 100$ is close as structure to a stoichiometric SiO_2 layer for which the FWHM is always 2.3 eV. The spectra in Fig. 6(b) is about N1s core level and indicate that there is more than one type of chemical environment for nitrogen [12]. However, the presence of N-atoms in the SiON network remains minor.

The Si2p and N1s core levels of the two samples ($\gamma=100$ and $\gamma=2$) appear very different.

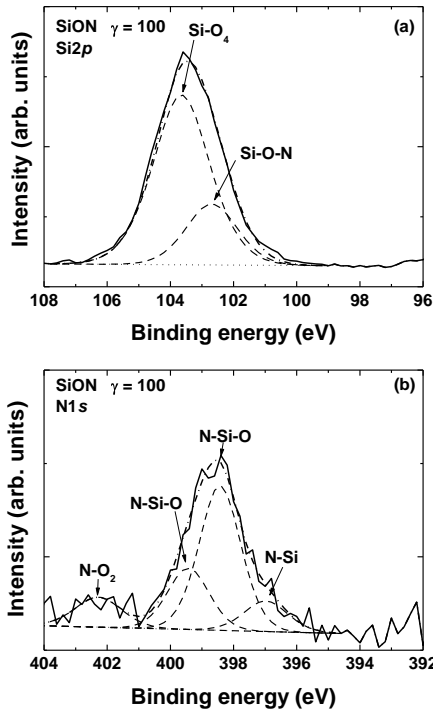


Figure 6. Binding energy of SiON layer with $\gamma = 100$ after sputtering: (a) Si_{2p} spectrum, (b) N_{1s} spectrum.

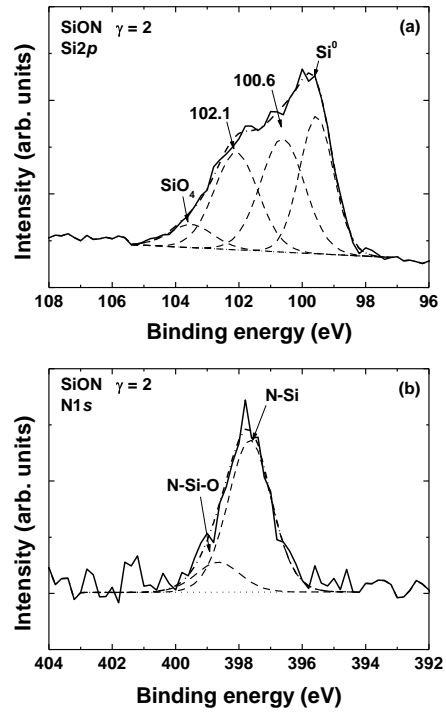


Figure 7. Binding energy of SiON layer with $\gamma = 2$ after sputtering: (a) Si_{2p} spectrum, (b) N_{1s} spectrum.

Figure 7 presents the results for the SiON layer with $\gamma = 2$. Figure 7(a) reveals the existence of Si⁰ at 99.5 eV with FWHM = 1.33 eV. Thus we can confirm the silicon richness in this layer. The splitting of the Si_{2p} photoelectron peak in three other components validates the presence of oxidation states. The first two components are to be attributed to the presence spin orbital splitting Si_{2p}3/2. They can be perturbed by the availability of the small amount of nitrogen (Table 1). The third component, which has a small contribution to the Si_{2p} photoelectron peak, is at 103.6 eV (FWHM 1.67 eV). It characterizes the Si-O₄ state; the silicon highest oxidation state. Figure 7(b) shows the N_{1s} core level (fitted to two Gaussian components) and suggests different environment for the N-atoms [12]. Similarly to the layer with $\gamma = 100$, the contribution of N-atoms to the layer network is small.

From the above observations we can conclude that Si-Si≡Si chemical bonds are significantly present in the Si-rich film ($\gamma = 2$) whereas in the layer with $\gamma = 100$ the highest oxidation states Si-(Si-O₃) and Si-O₄ are the only present ones.

Electrical characterization

For electrical characterizations, a circular golden top electrode of 4 mm in diameter was deposited on the SiO_xN_y thin film surface by sputtering, while the ground electrode was the Au/Ti layer, deposited on the Si substrate. The conduction current measurements were carried out under secondary vacuum (10^{-5} - 10^{-6} mb) at room temperature. The current flowing through the volume was recorded at different electric fields by means

of a Keithley 617 electrometer. The polarization time was fixed to 1000 s and can be considered as leading to a representative conduction current. After each polarization step, the sample was short-circuited for 1000 s and the transient current was recorded.

Fig. 8 shows an example of the current transients obtained for SiON layer with $\gamma=25$. This gamma value is representative for the layers with high volume resistivity ($\gamma = 100, 25$ and 10). The layer thickness was 193 nm as deduced from spectroscopic ellipsometry [5]. Except for the lowest applied voltages, the charging current reaches a steady state within less than 1000 s. However, in the depolarization stage, much longer time would be necessary to obtain a complete discharge of the samples. Besides, the transient contribution to the charging current is not symmetrical to the discharging current, all the more that the field is high. Finally, it can be seen that the shape of the depolarization current depends on the applied field; meaning that non linear phenomena are at play. Indeed, considering the transient discharging current corresponding to 20V polarization voltage, the current decays with a slower rate at long time. The feature is even more clearly revealed considering depolarization after 30V application that was recorded for 1 hour.

All of these features demonstrate that transient processes are controlled by charge accumulation within the material. The discharge process depends both on the field-induced space charge and the nature of trapping centers that may be dependent on the position in the sample. Further understanding would require identification of the nature of carriers and localization of trapped charges which are actually not available.

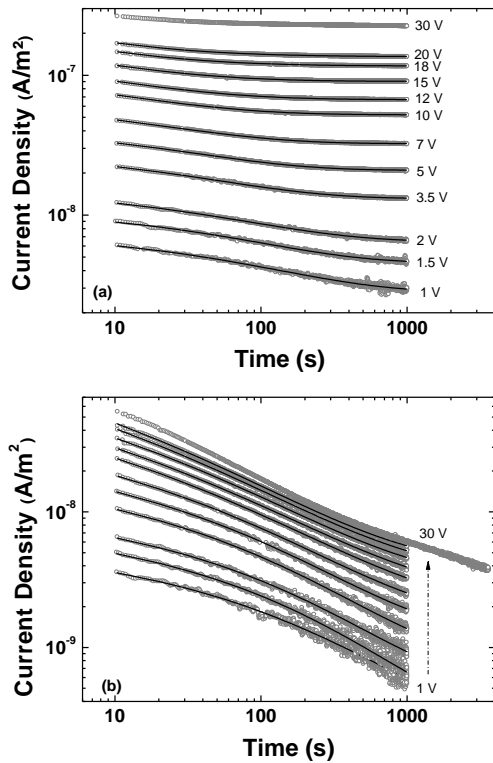


Figure 8. Current transients obtained on SiON with $\gamma=25$ during (a) charging and (b) discharging for applied fields in the range 5 to 150 kV/mm.

Fig. 9 shows the current vs. field characteristics obtained on the monolayer samples for $\gamma=2$ to 100. As commented earlier, the conductivity significantly increases as γ is decreased. The response appears linear up to fields of the order of 40kV/mm, without clear effect of the composition.

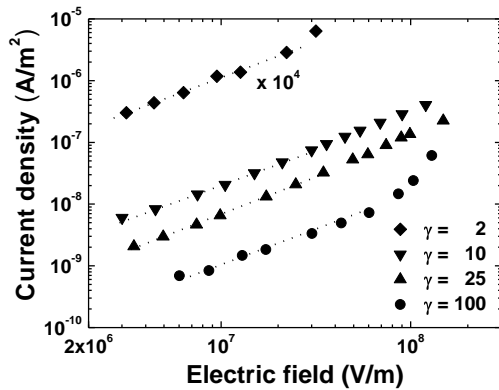


Figure 9. Current density versus electric field for different γ -values. For $\gamma=2$, the measured current density has been divided by 10^4 for clarity.

CONCLUSION

The structural, physico-chemical and electrical characterizations of silicon nitrite-oxide layers deposited singly or in a multi-layer structure show the possibility to assembly a structure with gradual properties and to be in

command of the conductive properties of as-deposited multi-layer system. Results from different diagnostic techniques are consistent with each other. The results from XPS measurements not only confirm those from FTIR diagnostic presented earlier but go further in the description of the deposited materials. The electrical measurements demonstrate that transient processes are controlled by charge accumulation within the material.

ACKNOWLEDGMENT

The authors would like to acknowledge the invaluable help of Dr. Caroline Bonafos (CEMES-CNRS, Toulouse, France) and S. Perret-Tran-Van (LAPLACE, Toulouse, France) for the observations and interpretation of the results obtained by TEM. The work was supported by the RTRA STAE in France under project SYMIAE.

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