

High-quality spin-on glass-based oxide as a matrix for embedding HfO₂ nanoparticles for metal-oxide-semiconductor capacitors

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Abstract By using a low cost, simple, and reproducible spin-coating method, thin films of SOG (spin-on-glass)-based oxides with electrical characteristics resembling those of a dry thermal oxide have been obtained. The superior electrical characteristics of Metal-Oxide-Semiconductor (MOS) capacitors based on SOG-oxides come from both (1) reducing the organic content of the SOG solutions after dilution with deionized water and (2) passivation of the silicon surface by a thin chemical oxide. Fourier transform infrared spectroscopy analysis shows that the organic content in H₂O-diluted SOG-oxides is reduced compared to undiluted SOG after N₂ annealing. In addition, by chemically embedding HfO₂ nanoparticles (np-HfO₂) to these SOG-based oxides, an effective increase in the accumulation capacitance of MOS capacitors is observed and this is related to the increase in the final dielectric constant of the resulting oxide after annealing so that potential use of SOG as a glass matrix for embedding HfO₂ nanoparticles and produce higher-k oxide materials is demonstrated.

Introduction

Initially, SOG-based oxides were commonly used by the semiconductor industry for relatively simple surface planarization and metal inter-level isolation processes [1]. Since then, the increasingly complex architecture and smaller dimensions of new electronic devices have required to use

SOG dielectrics in combination with other materials and/or deposition techniques in order to provide reliable planarization, isolation, and filling characteristics [2–5]. Nonetheless, even though the usually simple SOG-based deposition processing has become more complex, the chemical, physical, and electrical properties of these materials have been also enhanced with time in order to fulfill the most stringent technical requirements. Therefore, by using high quality SOG solutions along with one of the simplest thin-film deposition techniques (known as spin-coating), it could be possible to obtain specific electronic materials in a simple and economic fashion thus avoiding the need for complex deposition techniques and/or expensive deposition systems. On the other hand, using SOG as electric insulator has often required depositing a relatively thick layer (or multiple layers) of SOG on a substrate so that final SOG-based oxides thicker than 500 nm are usually obtained. By using high quality SOG solutions based on silicate polymers, it is possible to obtain thinner oxide films down to 200 nm after a 425 °C annealing [6], but the electrical characteristics of these oxides as well as the correlation to their chemical composition is not well understood specially for SOG-based oxides whose thickness are well below 100 nm. In addition, enhancement of the electrical characteristics of low-temperature SOG-based oxides are usually done after O₂/Ar plasma treatment in low vacuum conditions (~1 Torr) so that most of the remaining contaminants (mostly organic components) within the dielectric can be transformed into a gaseous phase and thus removed [7, 8]. Now, since high-quality dry thermal oxides are often grown on silicon at temperatures $T \geq 1000$ °C, using this relatively high temperature for SOG curing in N₂ ambient should provide SOG-based oxides with good physical properties after eliminating most of its organic components. For gate oxide applications, it is very important to understand the impact that a thinned

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SOG-based oxide has on the electrical performance of metal-oxide-semiconductor (MOS) devices and since these materials are obtained after a quite simple and low cost spin-coating technique, their possible technological applications should be explored.

In this study, we have used silicate-based SOG solutions (consisting of catalyzed alkoxysilanes which form a unique arrangement of silicate polymers of low molecular weight with an Si–O structure) diluted with organic/non-organic solvents in order to obtain oxides as thin as ~ 20 nm (after annealing in N_2 at $1000^\circ C$), and we compare and correlate their physical, chemical, and electrical characteristics. By introducing and hydrolyzing HfO_2 nanoparticles within the former SOG solutions, the resulting spin-on-dielectric (SOD) is used as the gate oxide of a different set of MOS capacitors. SOD-based oxides are able to increase the effective dielectric constant of the original SOG-oxide even though the current density in accumulation mode still remains excessively large. Nonetheless, the potential use of SOG as a matrix for embedding np- HfO_2 and thus developing new SOD materials is investigated.

Experimental

A silicate-type SOG material (from Filmtronics, 15A) was used for all oxide films. Silicon (100) wafers were 2" diameter, N/P type and with a resistivity range of 5–10 Ω cm. Since SOG-coating is adequate for planarizing or smoothing a wide range of substrate topographies, spin-coating large size wafers could be done as long as we are able to minimize some of the problems related to this deposition technique: spin flow, particle contamination, spin striations, thickness variations, pinholes, etc. Sometimes, spin-coating along with chemical–mechanical polishing and/or chemical–vapor deposition techniques could be needed in order to obtain uniform coating of very large size wafers. Standard RCA-cleaning procedures were applied to all wafers before they were dipped in HF solutions so that HF-last surfaces were obtained. For the silicon with a chemical oxide, the wafers were submerged in hot- H_2O_2 ($75^\circ C$, 15 min) after the final HF-last treatment so that a thin oxide (~ 2 nm) was developed atop the silicon surface. After substrates cleaning and preparation, the SOG solutions were directly applied on the wafers' surfaces and they were spin-coated at 7500 rpm by 20 s. After SOG application, all films were baked at $200^\circ C$ (10 min in N_2 ambient) in order to evaporate most of the organic solvents. A second thermal treatment of $1000^\circ C$ (30 min in N_2) was also performed within a quartz furnace so that better film densification and solvent removal could be obtained after this curing process. On the other hand, in order to reduce the resultant oxide thickness, several dilutions of SOG in

deionized water and 2-propanol (H_2O and C_3H_8O , respectively) were performed before applying these diluted SOG solutions on silicon and they were subjected to the thermal treatments mentioned previously. Both refractive indexes and thicknesses for all oxide films were measured with a Gaertner Ellipsometer L117 equipment. For electrical C–V and I–V characterization, all oxide films (diluted and non-diluted) were metalized with 1 μm of aluminum by evaporation and a gate capacitor area of $13.34e-4$ cm^2 was used for all MOS devices. A dry thermal oxide grown on silicon ($1100^\circ C$, 30 min in dry- O_2 , ~ 60 nm) was used as a monitor so that chemical and electrical characteristics of SOG-based oxides were compared against it. In order to increase the effective dielectric constant of SOG-based oxides, np- HfO_2 (99.9% purity, from American elements) were systematically introduced and hydrolyzed in the SOG solutions by weighing their total solute concentration to the final dissolution with an analytical balance AG285 from Mettler-Toledo. Also, in order to obtain as homogeneous solution mixtures as possible, the SOG + np- HfO_2 solutions were subjected to water bath (*bain-marie*, $80^\circ C$, 2 h) treatments before their application on silicon wafers. All films' chemical compositional analysis was obtained by FTIR spectrum measurements in absorbance mode with a Bruker Vector-22 system. Finally, C–V and I–V measurements were done by using a Keithley Model 82-DOS Simultaneous C–V system (at 100 kHz) and an HP 4156B semiconductor parameter analyzer, respectively. All electrical measurements were obtained at room temperature.

Results and discussion

By spin-coating non-diluted SOG solutions (100% SOG) directly on the silicon wafers and after baking these samples at $200^\circ C$ in N_2 , oxide films as thin as ~ 240 nm can be obtained. In order to get thinner films (less than 100 nm, for gate oxide applications), dilution of the SOG solutions with organic/non-organic solvents was performed so that the SOG viscosity could be decreased and thinner films were obtained as shown in Fig. 1. There, we notice that H_2O can be used as a better solvent for SOG in order to obtain thinner oxides as compared to C_3H_8O . For a solvent dilution percentage of 50%, H_2O -diluted SOG solutions produce oxides with a thickness of 44 nm, almost half the thickness from a C_3H_8O -diluted SOG solution (with an oxide thickness of 82 nm). The former tendency is explained in terms of the lower viscosity of H_2O compared to that of C_3H_8O (0.89 vs. 1.96 cP at $25^\circ C$) [9]. On the other hand, after measuring the absorbance spectrum of those samples by FTIR, the highly-organic content of a C_3H_8O -diluted SOG-based oxide is observed in Fig. 2, where higher absorbance peaks related to a high density of

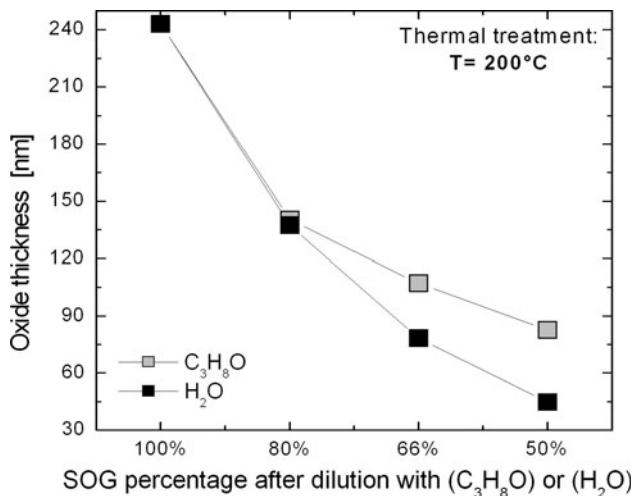


Fig. 1 Oxide thickness reduction after SOG dilution with C₃H₈O or H₂O solvents (thermal treatment of 200 °C in N₂). Because of its lower viscosity, H₂O can be used as a better solvent for SOG in order to obtain thinner oxide films

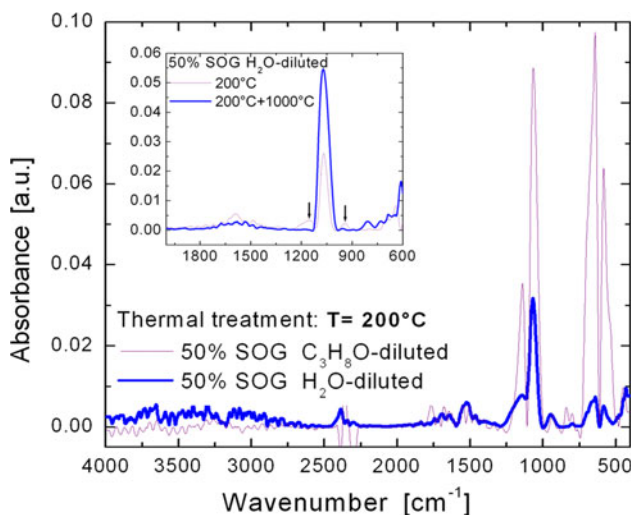


Fig. 2 FTIR absorbance spectrum for oxides whose SOG solutions were diluted 50% in C₃H₈O and H₂O. Because of the higher organic content of the C₃H₈O solvent, these oxides show higher peaks for alcohols (1139 cm⁻¹) and Si bonded to C-based molecules (band between 750 and 500 cm⁻¹). The inset shows that after applying a 1000 °C curing treatment to H₂O-diluted SOG-based oxides, better removing of any additional organic component is obtained for these oxides

Si atoms bonded to C-based molecules like CH₃ and C₆H₅ (band between 750 and 500 cm⁻¹) are found along with a higher absorption peak related to alcohols and ethers (peak at 1139 cm⁻¹) [10]. The inset shows that after applying a 1000 °C curing treatment to 50% H₂O-diluted SOG-based oxides, the original absorbance peaks related to alcohols and silicon atoms bonded to other organic components (band centered at 1139 cm⁻¹ and band between 980 and

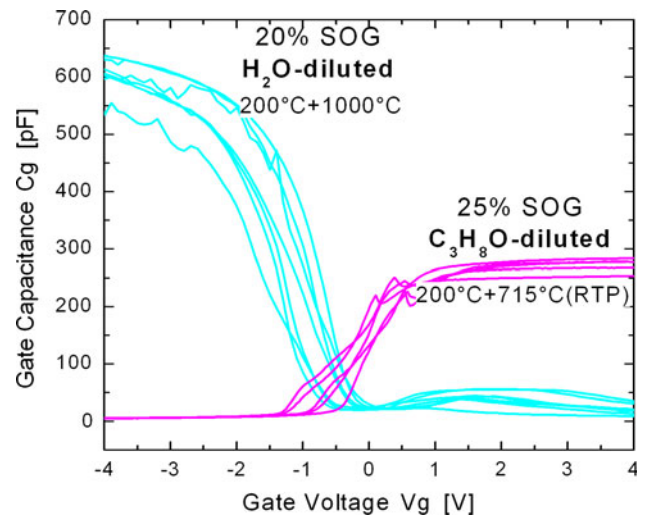


Fig. 3 C–V characteristics of SOG-based oxides diluted with C₃H₈O or H₂O. Even though both oxides present poor C–V characteristics, we notice that H₂O-diluted SOG solutions are able to produce the thinnest oxides as noticed by the higher C_{ox} when compared to organic dilutions

900 cm⁻¹, respectively) are further reduced thus indicating that a relatively high-temperature curing treatment in N₂ is helpful to better remove any additional organic component from this dielectric. In Fig. 3, we compare the C–V characteristics of SOG-based oxides whose solutions were previously diluted in H₂O and C₃H₈O. The C₃H₈O-diluted SOG-based oxide has been annealed at 715 °C, 5 min using a Rapid Thermal Processing (RTP) furnace in order to avoid organic contamination from these samples to our standard quartz furnaces during annealing. A highly-organic SOG-based oxide produce poor C–V characteristics like spreading of the oxide capacitance (C_{ox}), a high density of interface-states and spreading of the flatband voltage (V_{fb}). Nonetheless, the same applies for the H₂O-diluted SOG oxides where the same electrical defects are observed. For the H₂O-diluted SOG samples, it is important to mention that HF-last silicon surfaces were used before these MOS capacitors were fabricated so that a rough interface between the silicon surface and the SOG-oxide is thought to exist. Even though both oxides present poor C–V characteristics, we notice that H₂O-diluted SOG solutions are able to produce the thinnest oxides as noticed by the higher C_{ox} when compared to organic dilutions.

Figure 4 shows that by systematically increasing the dilution percentage of the SOG solutions with H₂O, a linear decreasing trend is observed regarding the final oxide thickness after a complete thermal treatment (baking at 200 °C and curing at 1000 °C, all in N₂). With both the increased dilution percentage and the final 1000 °C curing treatment, oxide films as thin as 20 nm can be obtained. On the other hand, for the same figure, we observe that the refractive indexes for all samples follow a more or less

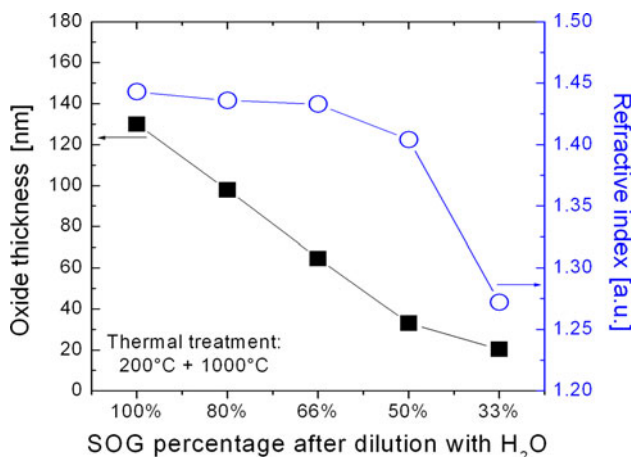


Fig. 4 Oxide thickness and refractive index reduction of SOG-based oxides after increasing dilution percentage of SOG in H₂O. Oxide films as thin as 20 nm can be obtained but with a low refractive index of 1.27

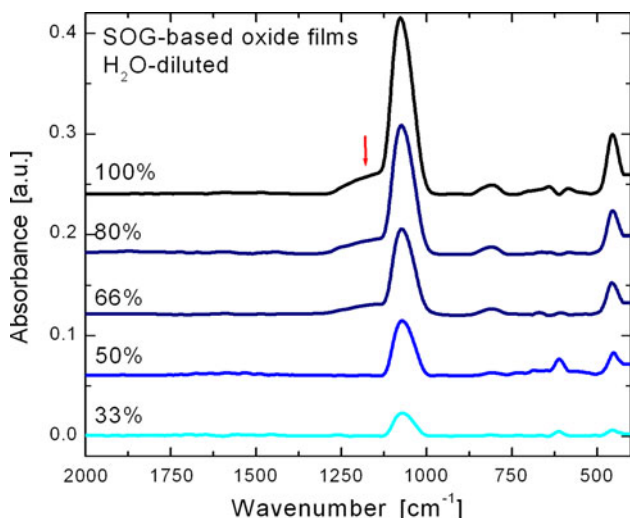


Fig. 5 FTIR absorbance spectrum for oxides based on H₂O-diluted SOG solutions. The *arrow* points at the peak related to chemical bonds present in alcohols and whose magnitude gets decreased as more solvent is used thus confirming that H₂O-dilutions are an effective way to reduce the organic content of SOG-based oxides

linear tendency down to a 50% dilution percentage, where this parameter is kept above 1.40. For a 33% dilution SOG-based oxide, the refractive index drops to about 1.27, which is close to the refractive index of water–ice 1.3, thus, indicating that highly-diluted SOG-based oxides will increase the porosity of the oxide film [11]. The absorbance spectrum of the former samples was taken after FTIR measurements in order to see the effect that H₂O dilutions have on the organic content within the oxide films. The results are shown in Fig. 5, where the small arrow points at the peak related to chemical bonds present in alcohols and whose magnitude gets decreased as more solvent is used

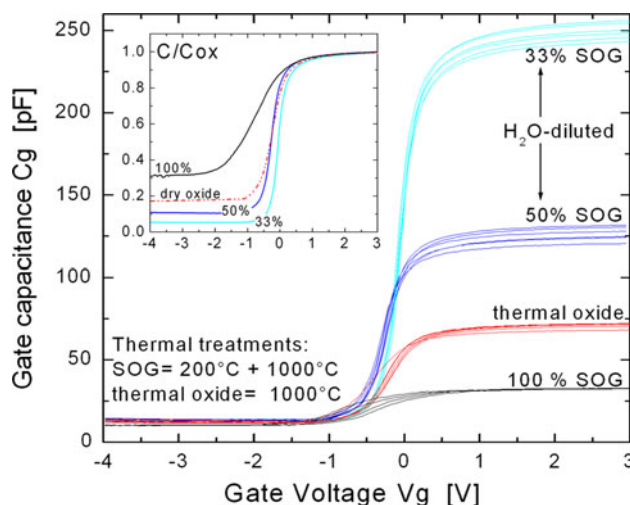


Fig. 6 C–V characteristics of SOG-based oxides (diluted with H₂O) deposited atop a thin chemical oxide. Compared to non-diluted 100% SOG-based oxides, H₂O-diluted SOG oxides are able to proportionally increase Cox as they get thinner after dilution. A dry thermal oxide is also shown for comparison purposes. The *inset* shows C/Cox for all oxides where H₂O-diluted SOG oxides are able to resemble the C–V characteristics of a dry thermal oxide grown on silicon

thus confirming that H₂O-dilutions are an effective way to reduce the organic content of SOG-based oxides. By introducing a thin chemical oxide (able to passivate the silicon surface) before deposition of the H₂O-diluted SOG-based oxides, enhanced C–V electrical characteristics are obtained for these MOS capacitors as shown in Fig. 6. Compared to a non-diluted SOG-based oxide, H₂O-diluted SOG oxides are able to proportionally increase Cox as they get thinner after dilution. A dry thermal oxide is also shown for comparison purposes. For all measured capacitors, we notice good uniformity in Cox (indicating good oxide uniformity throughout the silicon surface) as well as lower interface-states and fixed charge densities, and tight Vfb distribution. Since interface-states density Dit is a very important parameter regarding the quality of a Si–SiO₂ interface, charge-pumping measurements will be done on smaller area MOSFET devices (containing these H₂O-diluted SOG-based oxides atop a thin chemical oxide) so that better Dit quantization would be expected after reducing the density of extrinsic defects that are normally found on large area capacitor devices, these results will be published elsewhere. As of now, we think that initial passivation of dangling-bonds at the silicon surface by the thin chemical oxide is responsible of Dit reduction and this is visualized by the steeper slope of the normalized C–V curves (from accumulation to inversion). The inset in Fig. 6 shows the same C–V data after taking the average curve of several measurements for the correspondent oxides and normalizing all curves to their correspondent Cox. There, we notice that H₂O-diluted SOG oxides are able to resemble

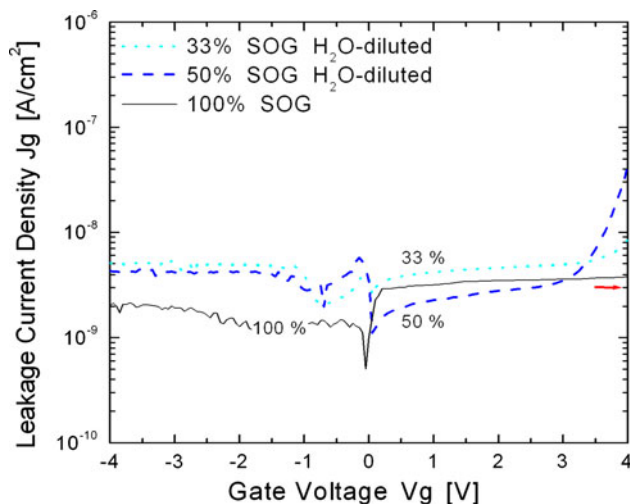


Fig. 7 Gate leakage current density versus gate voltage (J_g - V_g) characteristics of SOG-based oxides (diluted with H_2O) deposited atop a thin chemical oxide. Low J_g is found for both inversion and accumulation regimes, where a reference $J_g \sim 3 \times 10^{-9} \text{ A cm}^{-2}$ (shown by the arrow) belongs to a dry thermal oxide with thickness of $\sim 60 \text{ nm}$

the C-V characteristics of a dry thermal oxide grown on silicon. The dielectric constant k value for these SOG-based oxides is around 3.5 which is close to that of a dry thermal oxide. For the same samples, we notice in Fig. 7 that the gate leakage current density is kept slightly above the current density obtained from the thermal oxide (as shown by the arrow in accumulation regime or positive V_g) so that high-quality SOG-based oxides after H_2O -dilution have been confirmed just by introducing a thin chemical oxide at the silicon/SOG-oxide interface.

Up to this point, obtaining high-quality and very thin layers (down to $\sim 20 \text{ nm}$) of SOG-based oxides has been demonstrated. Now, by carefully introducing np-HfO₂ into the SOG solutions, we are able to embed some of these nanoparticles into the final SOG-based oxide so that regular increase in the dielectric constant of the final dielectric and therefore Cox of the MOS capacitor should be obtained. First, we use silicon wafers whose surfaces have been passivated by a thin chemical oxide as discussed before. This way, we ensure that good C-V characteristics could be obtained. Initially, we have introduced 50, 100, and 150 mg of np-HfO₂ into 1 ml of non-diluted SOG solution so that up to three different mixtures were prepared. Since a non-diluted 100% SOG solution produce oxides as thin as 130 nm (see Fig. 4), this solution is ideal to properly encapsulate the np-HfO₂ whose size is reported to be about 100 nm by the fabricant. After introducing np-HfO₂ into SOG and mixing this solution within an ultrasonic vibrator for 10 min, the final solution was directly applied to silicon and spin-coated. After the spin-coating process, the samples were subjected to complete thermal treatments including

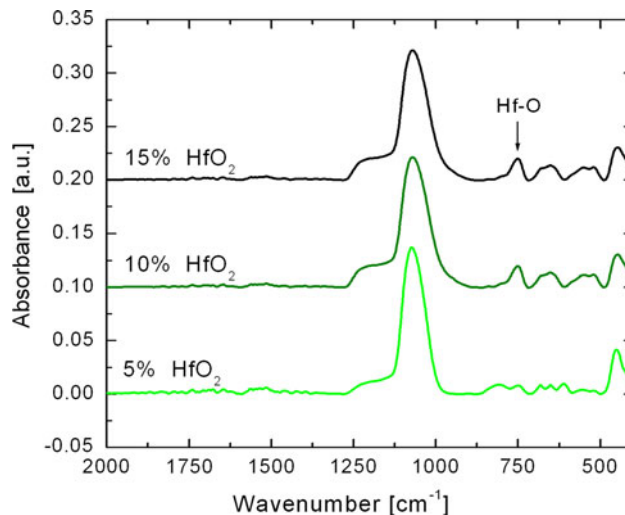


Fig. 8 FTIR absorbance spectrum for oxides based on SOG solutions with HfO₂ nanoparticles directly embedded to the glass matrix (SOG + np-HfO₂). By increasing the Hf content, there is a corresponding increase in the magnitude of the peak related to the Hf-O chemical bond, found at 752 cm^{-1}

both baking and curing at 200 and 1000 °C in N₂. The absorbance spectrum for these samples is shown in Fig. 8 after FTIR measurements. There, we notice that compared to H_2O -diluted SOG-based oxides, the band whose peak is centered at 1139 cm^{-1} (related to alcohols) is broaden for all cases as expected since we have used non-diluted SOG solutions. On the other hand, we also notice that by increasing the Hf content in the prepared solutions, there is a corresponding increase in the magnitude of the peak related to the Hf-O chemical bond (as shown by the arrow) and which is found at 752 cm^{-1} [12]. In order to visualize the surface roughness and spatial distribution of the np-HfO₂ embedded in SOG, interferometric and optical microscopy images were respectively taken. Figure 9a shows that by adding 15% of np-HfO₂ directly to SOG, no uniform distribution of these nanoparticles could be achieved after observing a high average roughness of the oxide ($R_{\text{rms}} \sim 80 \text{ nm}$), quite close to the size of the np-HfO₂ themselves. Figure 9b shows a picture of the same oxide surface in which random conglomeration of np-HfO₂ is clearly observed and whose final grains can reach several microns in size so that unstable and/or poor electrical characteristics from these oxides could be expected. Figure 9c-d shows surface roughness and surface's nanoparticles distribution, respectively, for an oxide whose composition was taken from a precipitated solution of SOG + np-HfO₂ mixture. There, we can see that surface roughness has been dramatically reduced ($R_{\text{rms}} \sim 5 \text{ nm}$) although there is no indication that np-HfO₂ were properly distributed in the oxide.

In order to promote a uniform distribution of the np-HfO₂ within the SOG, a small amount of glacial acetic

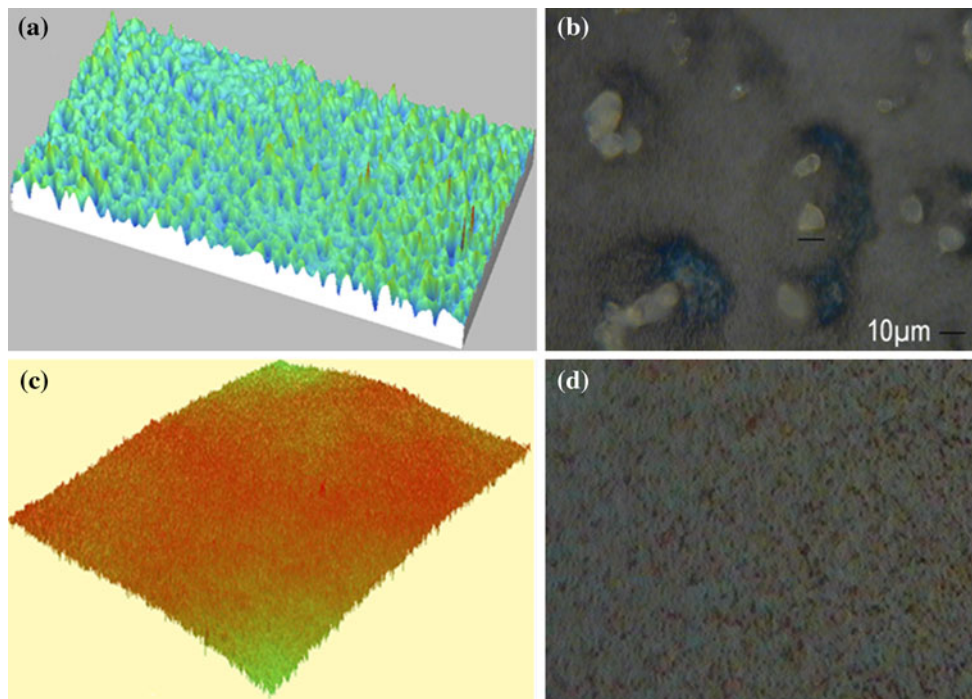


Fig. 9 **a, b** Interferometric and optical microscopy, respectively, of SOG-based oxides with 15% of np-HfO₂. High surface roughness and conglomeration of large grain-size HfO₂ nanoparticles is observed. **c, d** Interferometric and optical microscopy, respectively, of SOG-based

oxides with a precipitated solution of np-HfO₂. Low surface roughness is obtained although no indication that np-HfO₂ were properly distributed in the oxide is observed

acid (0.3 ml) has been intentionally added to a single SOG + np-HfO₂ mixture (6.6% of np-HfO₂, used as a reference) in order to increase its reactivity so that better dissolution of major polar and inorganic compounds could be expected. Along with other mixtures of SOG + np-HfO₂ (with the exception of a 15% np-HfO₂), all prepared solutions were also subjected to water baths (*bain-marie*) before they were applied to silicon surfaces. All these procedures were done under carefully controlled conditions in order to enhance the distribution of np-HfO₂ in SOG and also, get reproducible electrical characteristics. Figure 10 shows the absorbance spectrum for the above discussed samples. We still notice a high-intensity peak for the Hf–O bond in the oxide whose SOG solution was added with 15% of np-HfO₂. For 5 and 6.6% of np-HfO₂ dissolved into SOG, those oxides show rather low peak intensity for the Si–O bond found at 1070 cm⁻¹ (stretching mode) although the Hf–O bond is still detected. For the precipitated solution, there is no Hf–O bond detection and the quality of this oxide is rather compromised as seen by the irregular and low intensity of even the Si–O bond feature. Figure 11 shows C–V characteristics for these SOG + np-HfO₂-based oxides in which uniformity promotion was sought and above discussed. It is interesting to note that adding more np-HfO₂ to SOG does not necessarily produce oxides with the highest C_{ox} when comparing nanoparticles concentrations of 5, 6.6, and 15%. The

highest C_{ox} is obtained after increasing the chemical reactivity of the SOG matrix by adding a relatively small amount of CH₃COOH to the SOG + np-HfO₂ solution so that the solubility of np-HfO₂ in the final solvent is increased. This way, even with a concentration of 6.6% of np-HfO₂, the final oxide capacitance of these MOS capacitors is greatly increased as shown in the inset, where the C–V characteristics of a SOG + np-HfO₂(6.6%)-based oxide is compared against a non-diluted SOG-based oxide. Since a non-diluted or 100% SOG solution was used as the basis for all subsequent mixtures with np-HfO₂, the effective increase of C_{ox} is due to the addition and embedding of these nanoparticles within the oxide so that an effective increase of ~8.6× for C_{ox} is observed. The dielectric constant *k* value is then increased to around 30 for this particular sample. Figure 12 shows that SOG + np-HfO₂-based oxides have highly leaky characteristics for the accumulation regime, where an increase of up to nine orders of magnitude is observed when compared to the leakage current density of a thermal oxide (shown by the arrow). Since there is no indication that the np-HfO₂ are uniformly distributed all throughout the oxide, this high electronic conduction in accumulation regime should be due to localized conduction mechanisms taking place at specified paths formed by the conglomeration and consequent connection of Hf nanoparticles themselves; nonetheless, the exact conduction mechanisms for these samples

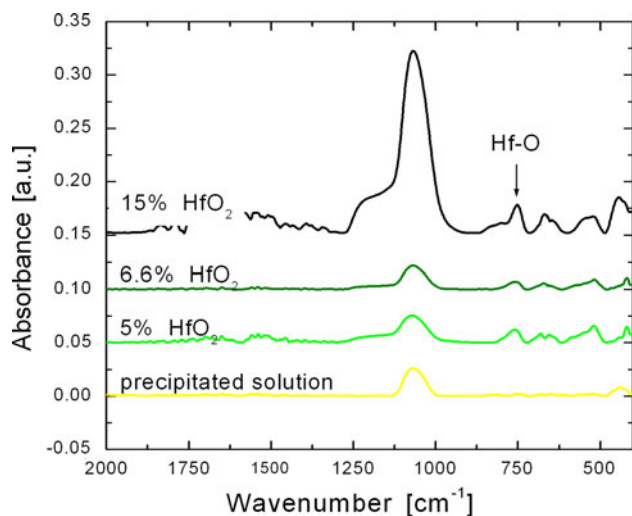


Fig. 10 FTIR absorbance spectrum for oxides based on SOG solutions with embedded HfO₂ nanoparticles. For these oxides, uniform distribution of np-HfO₂ within the glass was promoted by chemical methods like water bath treatments and small additions of acetic acid

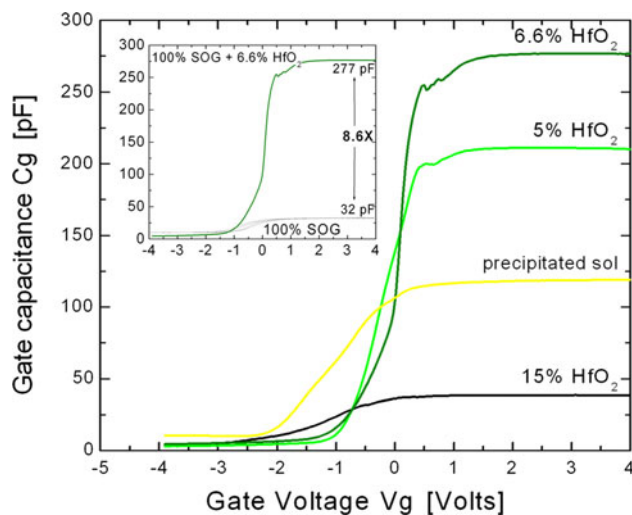


Fig. 11 C–V characteristics of SOG + np-HfO₂-based oxides with promotion of uniform distribution of nanoparticles throughout the glass. The highest Cox is obtained after increasing the chemical reactivity of the SOG matrix by CH₃COOH addition to the SOG + np-HfO₂ solution. The inset shows about ×8.6 increase in Cox for SOG-based oxides to which 6.6% of np-HfO₂ were introduced

would require further investigation. In inversion regime or negative gate voltage, the gate leakage current characteristics for the SOG + np-HfO₂ (6.6%)-based oxide is kept at the relatively low value of $\sim 1 \mu\text{A cm}^{-2}$ thus providing a slight chance to introduce these materials in some specific electronic applications like thin-film transistors based on these oxide films.

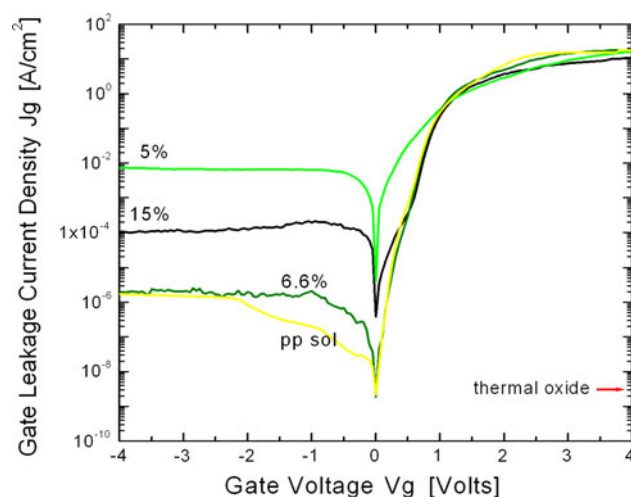


Fig. 12 J_g–V_g characteristics of SOG + np-HfO₂-based oxides (with promotion of uniform distribution). High leakage current characteristics are observed for the accumulation regime whereas the inversion regime presents moderate leakage current densities

Conclusions

Direct evidence of the high physical, chemical, and electrical quality of H₂O-diluted SOG-based materials and their potential use as a matrix for embedding HfO₂ nanoparticles in order to increase their dielectric constant have been demonstrated. Using C₃H₈O as a solvent for SOG will produce relatively thicker oxide films with highly organic content and poor C–V characteristics compared to H₂O-diluted oxides, where thinner oxides with better C–V characteristics and low gate leakage current densities were obtained along with reduced organic contents. By introducing HfO₂ nanoparticles to non-diluted SOG solutions, the correspondent Hf–O bond (shown at 752 cm⁻¹) was detected after FTIR measurements and its intensity was increased in proportion to an added volume of np-HfO₂. In order to promote uniform distribution of these nanoparticles within a SOG-based oxide, different chemical procedures were performed and the addition of a small amount of CH₃COOH to the SOG + np-HfO₂ solution proved to be an effective way to increase the gate oxide capacitance Cox of the MOS capacitors being tested. Even though highly leaky characteristics were found in accumulation regime for SOG + np-HfO₂-based oxides (resembling those of a metal-semiconductor rectifying junction), their correspondent C–V curves have shown a promising increase of the oxide capacitance so that potential use of SOG as a glass matrix for embedding HfO₂ nanoparticles and produce higher-k materials has been demonstrated by using a very simple, low cost, and yet reproducible spin-coating technique.

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