Electrical, optical, and structural characteristics of Al_2O_3 thin films prepared by pulsed ultrasonic sprayed pyrolysis

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The optical, structural, and electrical characteristics of aluminum oxide thin films deposited by pulsed ultrasonic sprayed pyrolysis are reported. The films are deposited on crystalline silicon at temperatures from 400 to 550 °C using a chemical solution of aluminum acetylacetonate, as source of aluminum, and *N*, *N*-dimethylformamide, as solvent. A H_2O-NH_4OH mist is supplied simultaneously during deposition to improve the films' properties. The results showed that the properties of the as deposited films depended strongly on the number of pulses used and on the substrate temperature. The thickness of the films is under 300 Å and the best films' properties showed an index of refraction close to 1.6 and a root mean square surface roughness of about 7.5 Å in average. Infrared spectroscopy shows that SiO_2 is observed at the interface with silicon of the Al_2O_3 films and seemed to play, as expected, a dramatic role in the electrical characteristics of the interface. Films with a dielectric constant higher than 8 and an interface trap density at midgap in the 10^{10} eV^{-1} cm^{-2} range are obtained. Films deposited with three pulses and at 550 °C are able to stand an electric field up to 4 MV/cm. © *2008 American Institute of Physics*.
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I. INTRODUCTION

Aluminum oxide (Al_2O_3) thin films deposited by physical or chemical methods actually find several applications in different areas of technological research. This is mainly because of their properties such as high mechanical resistance, chemical and thermal stability, and high electrical resistivity. In the microelectronics industry, Al_2O_3 is considered a good candidate to replace silicon oxide (SiO_2) as complementary metal-oxide-semiconductor transistor gate dielectric. Actually, for further advance in silicon microelectronics, alternative thin films are strongly demanded. In this application, the main goal is to get an alternative high-*k* gate dielectric with a low "equivalent oxide thickness" (EOT). In addition, for these alternative oxides, good thermodynamic stability and high interface quality on silicon are necessary.[1,2] Besides Al_2O_3, some of the most studied oxides are HfO_2,[3] La_2O_3,[4] Y_2O_3,[5] ZrO_2,[6] etc. These oxides have dielectric constants higher than 10 (SiO_2 has a dielectric constant of 3.9). Al_2O_3 shows high dielectric strength (a dielectric constant close to 10), good thermodynamic stability up to high temperatures, and high interface quality when deposited on silicon. Furthermore, Al_2O_3 films are amorphous when the deposition temperature is of the order of or lower than 700 °C. All these properties are considered favorable when synthesizing alumina thin films.[2,7–14] Al_2O_3 films with good characteristics have been obtained when sophisticated techniques such as atomic layer deposition[7] (ALD), metal organic chemical vapor deposition,[8] (MOCVD), or pulsed laser deposition[9] (PLD) are used. For example, low leakage currents of about 1 nA/cm^2 at an applied electric field of 2 MV/cm are observed in alumina films with 120 Å thickness using ALD. Under the best experimental conditions, Al_2O_3 films deposited by MOCVD show a leakage current of 10 nA/cm^2 at an equivalent oxide thickness of 3.6 nm. The films of Al_2O_3 deposited by PLD on silicon have shown an interface trap density in the range of 1.2×10^{10} eV^{-1} cm^{-2} when deposited at room temperature. Al_2O_3 films grown by rf magnetron sputtering[10] at room temperature and 200 and 300 °C showed that the best dielectric breakdown strength was of 2.1 MV/cm. In this latter case, the thickness of the films ranged from 45 to 130 nm. However, the spray pyrolysis technique is the only inexpensive technique that has been used to obtain high quality alumina thin films with properties similar or superior to the ones obtained with some of the most elaborated methods.[15–17] In previously reported work, 100 nm thick alumina films deposited with the spray pyrolysis technique were obtained with dielectric strengths of about 5 MV/cm, interface trap densities in the range of 10^{11} eV cm^2, and a dielectric constant in the range of 7.9. It was shown that the addition of a mist of H_2O improved the electrical, structural, and optical properties of the films.[16–18] More recent reports have shown that the addition of a source of nitrogen during deposition of the films can improve their properties.[2,5] In the present work, we used the pulsed sprayed ultrasonic method to obtain alumina thin films as thick as 300 Å. From our best information, no previous attempts had been performed to obtain alumina films with excellent properties in this range of thickness with any low cost deposition technique such as spray pyrolysis. The films were pulsed

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deposited under short aerosol shots in order to control their final thickness. H_2O-NH_4OH mist was also supplied simultaneously during deposition. The result was that we obtained films with high dielectric constant as well as good optical and structural properties.

II. EXPERIMENTAL PROCEDURE

The Al_2O_3 thin films were prepared from aluminum acetylacetonate [Al(acac)$_3$] dissolved in *N*,*N*-dimethylformamide (*N*,*N*-DMF). These raw materials were supplied by Alfa AESAR and J.T. Baker, respectively. The films were deposited in the temperature range from 400 to 550 °C on crystalline Si(100) and Si(111). For this work, a modified ultrasonic spray pyrolysis technique was used,[19,20] in which the aerosol droplets, consisting of Al(acac)$_3$ in *N*,*N*-DMF, were supplied during fixed periods of time. This method was adopted in order to achieve a smaller film thickness than those previously achieved with the regular spray deposition method. The modified method is named in this work as pulsed ultrasonic sprayed pyrolysis (PUSP). The films were obtained supplying one, two, or up to three short pulses (shots) that elapsed 3 or 5 s each. For the films that were deposited at 400 °C, each deposition pulse (shot) elapsed 5 s, and the interruption time between one pulse and the next was of 3 s. For the films deposited from 450 to 550 °C, each deposition pulse elapsed 3 s, and the interruption time between one pulse and the next was of 3 s, too. So, when supplying two or three pulses, a period of 3 s separated each one. The spray pyrolysis technique has been used widely to obtain films or coatings of different materials, mainly metallic oxides, since the technique is used under atmospheric pressure conditions. The spray pyrolysis technique is considered an inexpensive and scalable technique to obtain films and coatings with excellent properties. The technique consists in supplying an aerosol from a chemical solution which undergoes a pyrolytic decomposition on a hot substrate, leading to a solid film or coating on top of the surface used as substrate. However, the technique has not been attempted to obtain films thinner than 1000 Å. Most of the films that are obtained with this technique usually range from 0.1 up to a few microns.[21] For this work, the films of Al_2O_3 were prepared with a 0.09 mol/l chemical solution formed with Al(acac)$_3$ in *N*,*N*-DMF. A molten tin bath was used as thermal energy source for the substrate to achieve the pyrolytic reaction. A mist of $1H_2O-1NH_4OH$ was supplied simultaneously during deposition. Furthermore, nitrogen (N_2) at a flow rate of 5 l/min was used as carrier gas. The as deposited films were characterized in a Gaertner LSE Stokes ellipsometer (632.8 nm), a Nicolet infrared spectrometer, model 519P, and an atomic force microscope, the latter one from Digital Instruments. In addition, the Al_2O_3 films were incorporated into metal-oxide-semiconductor structures in order to carry out the electrical characterization. For this task, aluminum dots (with a surface area of 0.001 cm^2) were evaporated on top of the Al_2O_3 films. Ramp *I*-*V* (ramp rate of 0.5 V/s) and capacitance-voltage measurements were obtained with a Keithley commercial system using the model 82-DOS simultaneous *C*-*V* program.

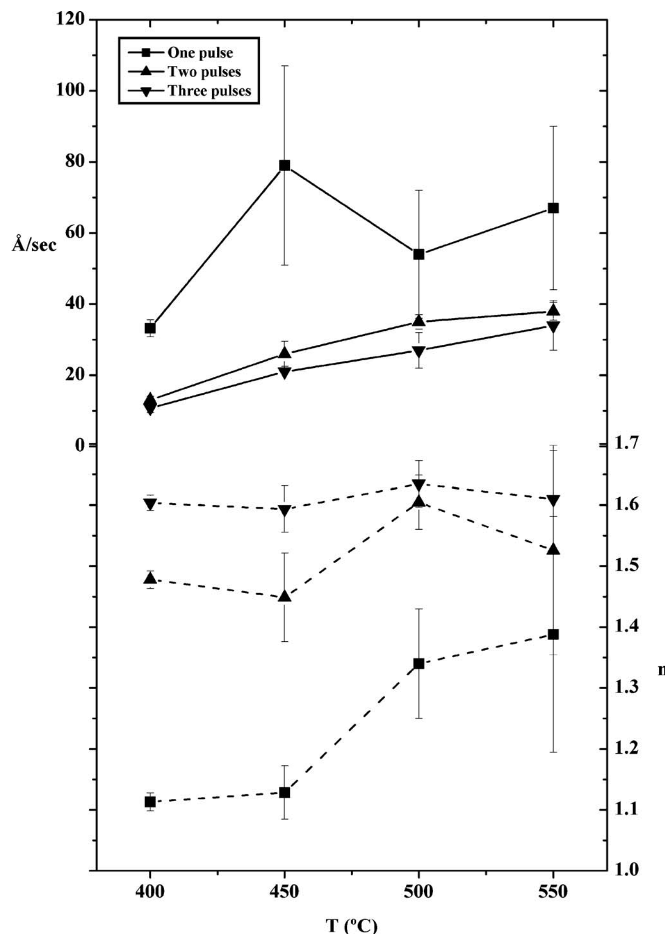

FIG. 1. Deposition rate (continuous line) and index of refraction (dashed line) of the Al_2O_3 deposited as a function of the substrate temperature.

III. RESULTS AND DISCUSSION

Figure 1 shows the deposition rate and the index of refraction as a function of the substrate temperature of the Al_2O_3 films deposited from one and up to three pulses. It is observed that the index of refraction increases in general with the number of pulses applied during deposition. This indicates that the density of the films is improved with the number of pulses supplied. The high index of refraction in the films (close to 1.66) is indicative of a high quality amorphous alumina thin film.[22] However, the films that are deposited with a single pulse result with a very low index of refraction (lower than 1.4). This low value indicates that the films deposited under this condition are porous and have as a consequence a very poor overall quality.[23] The films that are deposited with two pulses show an intermediate index of refraction between the ones obtained with one and three pulses. The deposition rate of the films is also shown in Fig. 1. It is observed that the films that are deposited with two or three pulses show a deposition rate that increases almost linearly with temperature. The deposition rate is found in the range of about 10 up to ~30 Å/s when the substrate temperature varies from 400 to 550 °C. This deposition rate compares favorably with the ones obtained previously in thicker alumina films that were deposited with the same molar concentration but using air as carrier gas.[15–17] In this work the range of deposition rates are higher compared with the

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TABLE I. Main properties of the best Al_2O_3 films deposited at different substrate temperatures and with the different aerosol pulses.

T (°C)	κ	d (Å)	d_1 (Å)	d_2 (Å)	D_{it} (eV^{-1} cm^{-2})	d_2/d_1	E (MV/cm)
				Three pulses			
450	8.2 ± 0.5	202	28	174	$(3.3 \pm 0.1) \times 10^{10}$	6.2	≤ 0.9
500	8.2 ± 0.6	289	41	248	$(2.7 \pm 0.5) \times 10^{10}$	6.0	≤ 2
550	8.9 ± 0.8	307	24	283	$(2.6 \pm 0.3) \times 10^{10}$	11.8	≤ 4
				Two pulses			
550	8.9 ± 0.8	201	16	185	$(4.4 \pm 0.2) \times 10^{10}$	11.6	⋯
				One pulse			
500	9.1	146	10	136	1×10^{11}	13.6	⋯

one obtained by other authors using a CVD technique.[8,13] A high deposition rate could be a good advantage for some applications. The films that are deposited with a single pulse show in average depositions rates larger than 30 Å/s. However, in this latter case, it seems that the films are not homogeneous throughout the entire substrate surface, since large variations in thickness were found. In all cases, at least three different measurements of the thickness were performed on random sites of the surface of the film. The lack of homogeneity in the films deposited with a single pulse seems to be confirmed with the low values of the index of refraction measured. From these results, it seems that the time elapsed during deposition pulses is of great importance for obtaining Al_2O_3 films with good homogeneity and high index of refraction using the pulsed sprayed pyrolysis technique. From these results, it can be inferred that, under the experimental conditions mentioned, a good decomposition of the aerosol to yield an alumina film requires about 13 s when deposition is carried out at 400 °C and about 9 s when deposition is done in the range of 450–550 ° C. It seems that the latter elapsed time values are necessary to obtain homogeneous and high index of refraction alumina films. The thickest film was about 300 Å, as shown in Table I, which lists a selected group of films deposited under different conditions, with the best characteristics.

Figure 2 shows the infrared spectra of the films that were deposited at temperatures ranging from 400 to 550 °C and with three pulses. Previous infrared results from thick and amorphous Al_2O_3 films present a wide band centered around 700 cm^{-1}. This band could correspond to the overlapping of the Al–O stretching mode (750–850 cm^{-1}) and the O–Al–O bending mode (650–700 cm^{-1}), as reported previously.[22] In this work, all the alumina films deposited with the different experimental conditions show the characteristic Al–O band centered close to 700 cm^{-1}. The whole infrared characteristics of the films deposited are depicted in the spectra shown in Fig. 2. The intensity of the Al–O band is small for the films deposited at 400 or 450 °C. This is probably due to the existence of a small number of Al–O bonds formed because of a very thin film. On the other hand, when the films are deposited at 500 or 550 °C, the intensity of this band is larger, probably because thicker films are obtained at these temperatures. An absorption peak at approximately 1100–1050 cm^{-1} is clearly noticed in the films deposited at 500 and 550 °C. This one is associated with the Si–O bond vibration in the stretching mode.[24] The SiO_2 film grown immediately on top of the silicon substrate is due to the oxidation of the silicon wafer. The SiO_2 is very noticeable when the films are deposited at 500 or 550 °C even when a single pulse is used. It has been shown in previous studies that it is in general very difficult to get rid of the growth of SiO_2 mainly because most systems work under nonequilibrium conditions. The presence of SiO_2 on top of the crystalline silicon is even observed in thin films that are fabricated under high vacuum conditionings. In addition, the existence of

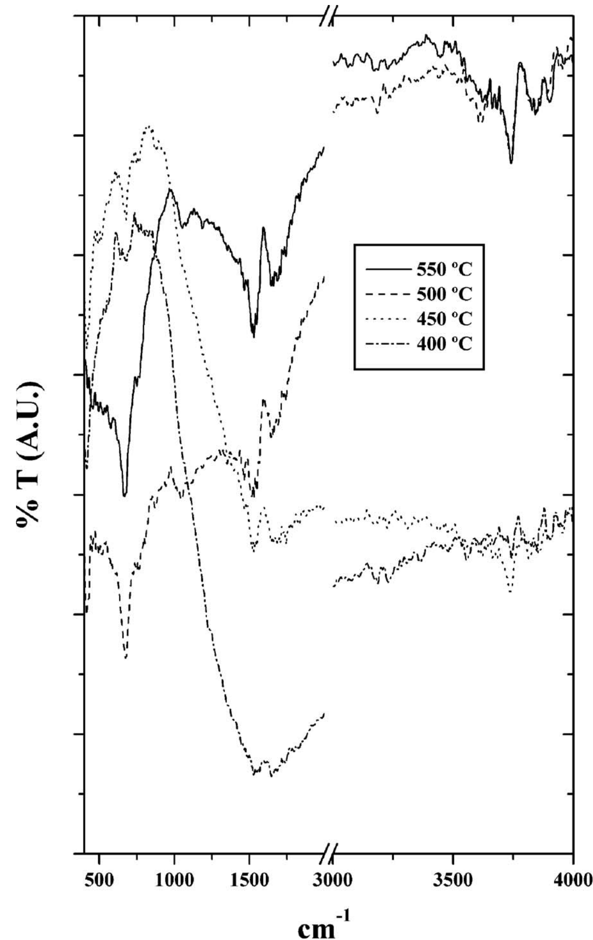

FIG. 2. Infrared spectra of the Al_2O_3 films deposited with three pulses and at 550 °C.

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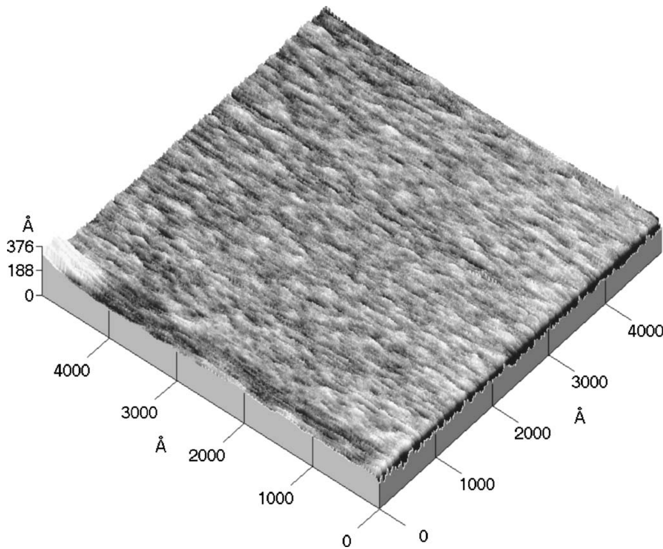

FIG. 3. AFM image of the surface of an Al_2O_3 film deposited with three pulses and at 550 °C.

SiO_2 increases the EOT of the films and reduces the effective dielectric constant of the alternative high-k dielectric. A lot of work is being done about this issue.[1,2] A further discussion on this SiO_2 layer will be presented later when the electrical characterization of the films is discussed. The infrared spectra show in addition a band around 1500 cm^{-1} due to carbon related bonds.[25] This one might be due to an incomplete decomposition of the Al(acac)$_3$ used as aluminum source. It has been observed that most films that are deposited by means of an organic source are usually not free from carbon related bonds. In our case, further measurements need to be done in order to estimate the amount of carbon that might remain in the films, although it seems that it does not have a dramatic influence in them.[16]

Figure 3 shows an atomic force microscope (AFM) image of the surface of an Al_2O_3 film deposited at 500 °C with three pulses. Our films show in average a surface roughness of 15 Å. In general, a low surface roughness of alternative high-k dielectrics is required for the application in the microelectronics industry.[2,17] Films deposited at 500 °C show in general the lowest root mean square surface roughness (∼7.5 Å). Figure 4 shows the behavior of the rms surface roughness as a function of the substrate temperature for the films deposited under the different numbers of pulses. On the other hand, films deposited at 450 and 550 °C present average rms roughnesses that are much higher, ∼22 and ∼16 Å, respectively. It is still not clear why there is a minimum in the surface roughness in the films deposited at 500 °C, but it might be related to the surface kinetics during the growth of the films.[21]

The electrical characterization of the alumina thin films is shown in Figs. 5–8. Figure 5 shows a typical capacitance versus voltage graph (C-V) in low and high frequencies for a film deposited at 500 °C under three pulses. In general, the electrical results are the average of at least three different capacitors randomly chosen on each alumina thin film deposited for this purpose. The inset in Fig. 5 shows the interface

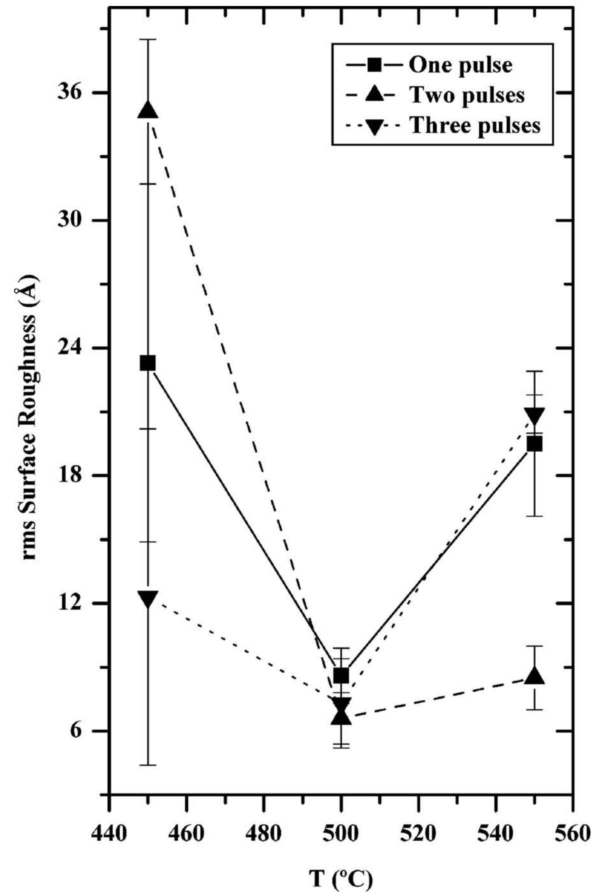

FIG. 4. Root mean square surface roughness of the Al_2O_3 deposited films as a function of the substrate temperature.

trap density (D_{it}) of the films. The D_{it} of the films was experimentally determined from the simultaneous low and high frequency C-V curves.[26] The D_{it} is shown in Fig. 6 and is listed in Table I for the best films obtained. In almost all cases, the interface trap density was in the 10^{10} range at midgap. This interface trap density is one order of magnitude lower than the one that was reported in previous works.[16,17] These results suggest that a high quality interface was achieved probably due to the influence of the ammonia during deposition of the alumina films. The good participation of the source of nitrogen during deposition of thin films has been highlighted in some high-k dielectrics such as yttrium oxide and hafnium oxide.[5,27] The effective dielectric constant of our films was also determined from the high frequency C-V curves in accumulation. The second column in Table I also lists the effective dielectric constant. The effective dielectric constant was found between 8 and 9. This is shown in Fig. 6, too. Again, these values are higher than those reported in previous work[16,17] (∼7.9). These results suggest that the role of the SiO_2 layer in our films is very dramatic. In order to appraise its important influence, the following analysis and discussion were performed.

The thicknesses of the SiO_2 and Al_2O_3 layers, denoted, respectively, by d_1 and d_2 are calculated according to a relation that results from the series capacitance between the SiO_2 and Al_2O_3 layers,

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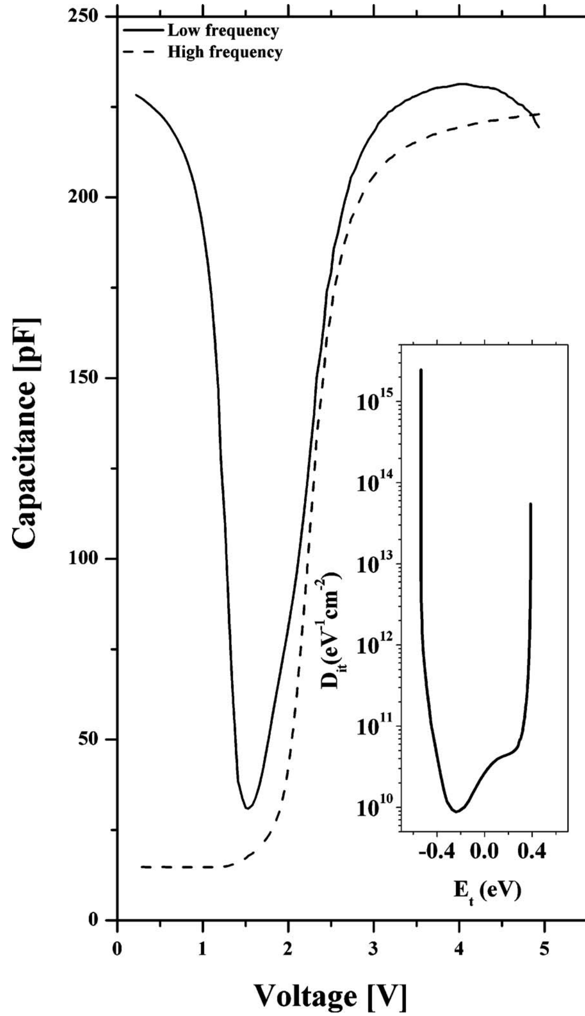

FIG. 5. Typical capacitance vs voltage graph (C-V) in low and high frequencies for a film deposited with three pulses at 550 °C. The inset shows the interface trap density as a function of the silicon midgap.

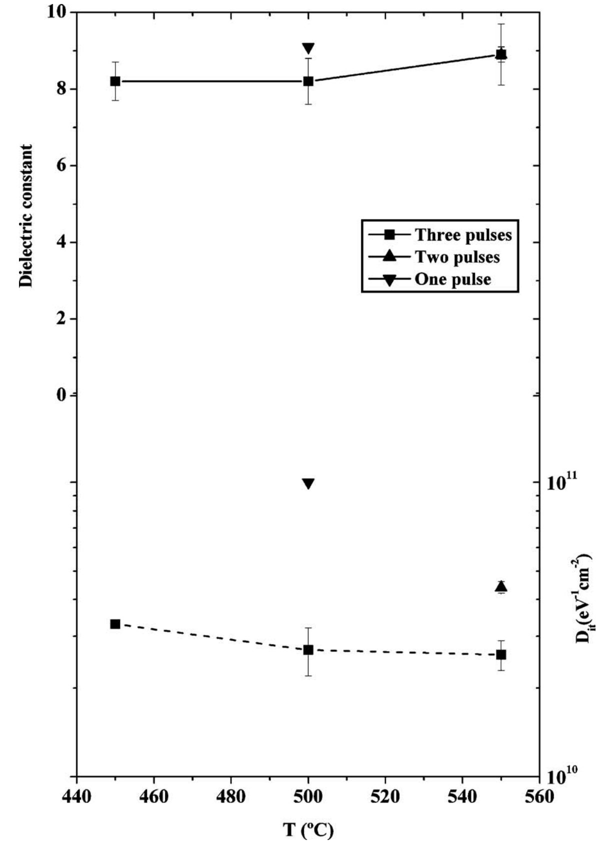

FIG. 6. Dielectric constant (continuous line) and interface trap density (dashed line) as a function of the substrate temperature of the best Al_2O_3 films deposited.

$$\frac{d}{\kappa}=\frac{d_1}{\kappa_1}+\frac{d_2}{\kappa_2}, \tag{1}$$

where

$$d=d_1+d_2 \tag{2}$$

is the total thickness of the films. The thickness d was experimentally measured by ellipsometry. In Eq. (1), κ is the effective dielectric constant, which was also determined experimentally. For this task, we had to assume that the SiO_2 layer has a dielectric constant of 3.9 ($=\kappa_1$). The dielectric constant of the Al_2O_3 layer varies with the deposition technique,[2,10,11] but recent results have shown that it can reach values up to ≈ 10 ($=\kappa_2$). Taking into account the above information, the following relation should hold for d_2:

$$d\left(\frac{1}{\kappa}-\frac{1}{3.9}\right)=d_2\left(\frac{1}{10}-\frac{1}{3.9}\right). \tag{3}$$

From Eq. (3), d_2 can be calculated, and taking into account Eq. (1), d_1 can also be determined. Table I also lists these calculated thicknesses of the Al_2O_3 and SiO_2 layers, respectively. From the results of Table I, it is observed that the thinner the SiO_2 layer, the larger the interface trap density. This result might suggest that a very thin SiO_2 film might start losing its excellent passivation properties on silicon. It has been stated that the properties of SiO_2 are usually lost when its thickness is of only a few atomic layers.[1] This result is shown in Fig. 7. In addition, the effective dielectric constant increases as the SiO_2 layer thickness is being reduced. This result is shown by plotting the d_2/d_1 ratio as a function of the effective dielectric constant (inset in Fig. 7). However, this latter result should saturate at higher values of the ratio. An equivalent result is shown by Groner *et al.*[7] From this analysis, it seems that the interface SiO_2 layer plays an important role in the interface properties of the alumina films deposited by the PUSP technique. It can be concluded that the combination of a SiO_2 layer under an Al_2O_3 layer can provide a high effective dielectric constant with an interface trap density close to the one shown by a high quality[26] SiO_2 when a nitrogen source is added during the deposition process. Thus, the electrical properties of the films can be tuned in order to obtain films with a desired value of interface traps and with a high dielectric constant.

Finally, ramped I-V measurements are shown in Fig. 8 for alumina films deposited with three pulses, which presented the best I-V characteristics when deposited at 550 °C. It was observed that at low fields, only the constant displacement current associated with the voltage ramp applied is present. However, at higher fields (after 2 MV), an injection current across the oxide is observed up to electric field close to 4.0 MV/cm at which a destructive breakdown is ob-

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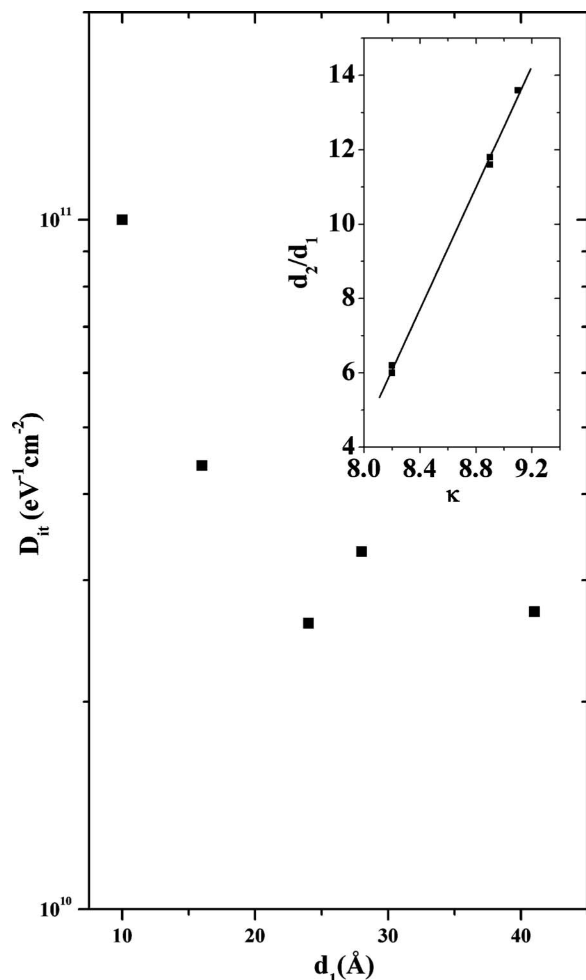

FIG. 7. Interface trap density as a function of the SiO_2 layer thickness. The inset shows the variation of the dielectric constant as a function of the ratio of the Al_2O_3 to the SiO_2 layer thicknesses.

served. Films deposited at 550 °C with only two pulses can stand an electric field lower than 2.0 MV/cm. Films deposited at lower temperatures (450 or 400 °C) with three pulses can stand at most 0.9 MV/cm (not shown). In general, films deposited with two and one pulses at any temperature do not lead to high quality *I-V* characteristics.

IV. CONCLUSIONS

High quality aluminum oxide thin films were deposited on silicon using the pulsed ultrasonic spray pyrolysis technique from $Al(acac)_3$ in *N*,*N*-DMF. The results showed that the optical, structural, and electrical properties of the as deposited films depended strongly on the number of pulses and on the substrate temperature used during deposition. The addition of a H_2O-NH_4OH mist supplied simultaneously during deposition seemed to improve the interface quality of the as deposited films. Films as thick as ~300 Å were obtained with an index of refraction close to 1.6 and a root mean square surface roughness of about 7.5 Å. SiO_2 found in the films at the interface with silicon seemed to play an important role in the electrical characteristics of the interface. Films with a dielectric constant higher than 8 and an interface trap density at midgap in the 10^{10} eV^{-1} cm^{-2} range were

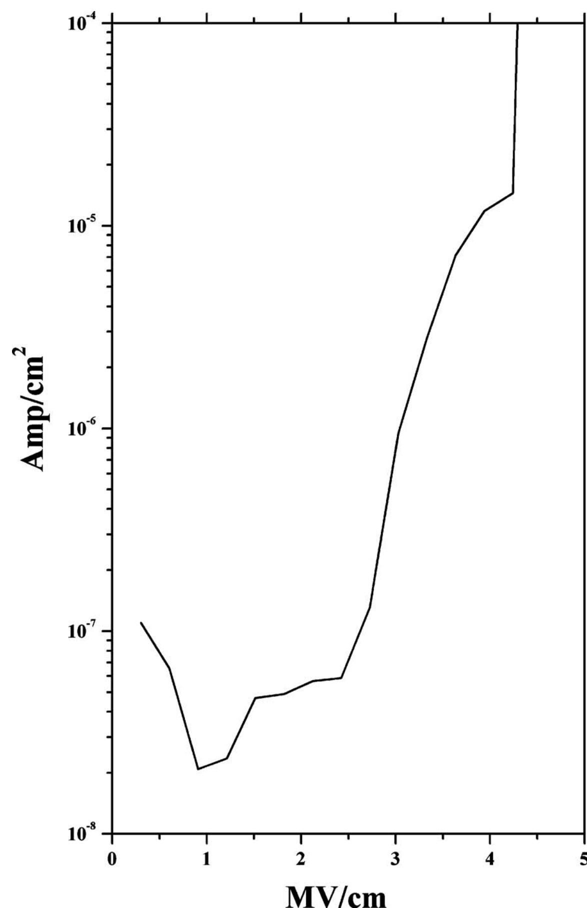

FIG. 8. Ramp *I-V* characteristic for an Al_2O_3 film deposited with three pulses and at 550 °C.

obtained. Films deposited with three pulses at 550 °C were able to stand an electric field up to 4 MV/cm.

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