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Influence of the synthesis conditions of silicon nanodots in an industrial low pressure chemical vapor deposition reactor

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Abstract

Experiments conducted in an industrial tubular low pressure chemical vapor deposition (LPCVD) reactor have demonstrated the reproducibility and spatial uniformity of silicon nanodots (NDs) area density and mean radius. The wafer to wafer uniformity was satisfactory (density and radius standard deviations <10%) for the whole conditions tested except for low silane flow rates, high silane partial pressures and short run durations (<20 s). Original synthesis conditions have then been searched to reach both excellent wafer to wafer uniformities along the industrial load of wafers and high NDs densities. From previous results, it was deduced that the key was to markedly increase run duration in decreasing temperature and in increasing silane pressure. At 773 K, run durations as long as 180 and 240 s have thus allowed to reach NDs densities respectively equal to 9×10^{11} and 6.5×10^{11} NDs/cm^2 for the two highest silane pressures tested in the range 60–150 Pa.

Keywords: Silicon nanodots; Chemical vapor deposition; Area density; Ellipsometry; SEM

1. Introduction

Since several years, silicon nanodots (NDs) are increasingly attracting attention because their unique physical properties, especially quantum mechanical effects, allow developing new silicon based functional devices, such as resonant tunnel components, one-dimensional transport devices, silicon non volatile memories and single electron transistors [1–3].

Various methods have been proposed for Si NDs synthesis such as low pressure chemical vapor deposition (LPCVD) [4–5], ion implantation [6] and aerosol [7]. The CVD method appears as a convenient technique because of its dominant position in ultra large scale integration (ULSI) processing, due to the excellent control of the deposition parameters and the possibility to obtain, immersed in a silicon dioxide matrix, well isolated storage nodes [8]. According to Nicotra et al. [9], one of the major issues of the field is a strict control of the NDs size distribution since fundamental parameters ruling the electronic transport, such as the Coulomb blockade and the energy quantization due to the carrier confinement are strong functions of the size and shape of NDs. The control of their area density is also of main importance. For Makihara et al. [10], densities as high as 10^{12} cm^{-2} on an ultrathin SiO_2 layer are a crucial factor for developing floating gate MOS devices.

Such structures are conventionally formed using hot wall LPCVD reactors and involve very short deposition times comprised between 5 and 100 s [2,8,10–12]. NDs density and size are piloted by the synthesis conditions as by the nature of the substrate [13].

Miyazaki et al. [4] and Mazen et al. [5] have shown that a convenient hydro fluoric acid (HF) pre-treatment of thermally grown SiO_2 substrates sharply increases the area density of silicon NDs and decreases their radius; the reason is that HF pre-treatments increase the silanol Si–OH bonds number to the detriment of that of siloxane Si–O–Si bridges which are naturally present on the SiO_2 substrates and which are much less reactive.

Many authors [2,4,9,11–15] have studied the influence of the deposition time on NDs features. They all found that the coverage of SiO_2 substrates by Si deposition increases with time. For Kajikawa and Noda [16], the initial stage of a CVD film is characterized by the existence of an incubation time, i.e.

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a time delay for deposition. Within the incubation period, film deposition is slower than during continuous film growth but grows exponentially with time. Nicotra et al. [9] found that nucleation is a continuous process occurring all along the deposition and that NDs size increases with time. For Puglisi et al. [1,8], a capture zone of about 4 nm exist around each nucleus within which new deposited Si monomers preferentially contribute to the growth of a previously nucleated seed rather than aggregate to form a new nucleus. Some authors [9,13,15] have observed an increase in NDs density with deposition time till a saturation value close to 10^{12} NDs/cm^2. For longer run durations, the growing NDs merge with adjacent ones by coalescence and the NDs density decreases.

For Mazen et al. [15] and Nicotra et al. [12], the nucleation process has a low free energy barrier which explains the weak influence of temperature on NDs density.

Makihara et al. [10] have shown that there is an optimal silane pressure in terms of NDs density close to 50 Pa (0.4 Torr) for a deposition temperature of 540 °C. Miyazaki et al. [4] have found that at 560 °C the NDs density increases with P_{SiH_4} between 0.02 and 0.1 Torr and remains constant between 0.1 and 0.2 Torr. For Lombardo et al. [13], the NDs density increases with silane pressure between 0.05 and 0.2 Torr.

To the best of our knowledge, no study has been published concerning the reproducibility of NDs formation and also the uniformity of NDs features on a wafer and from wafer to wafer along an industrial load. These parameters are obviously of main importance in the technological framework presented above. From the brief bibliographic survey above, it is clear that the application of LPCVD to NDs synthesis is under development and the optimization of the deposition parameters is still under progress. These are the reasons why the present work deals with the experimental study of the influence of the main synthesis conditions on the NDs area density and radius and on their reproducibility and spatial uniformity on wafers in an industrial LPCVD reactor.

2. Experimental

Silicon NDs were deposited in an industrial tubular hot wall LPCVD reactor from Tokyo Electronic Limited (TEL), as schematically presented in Fig. 1. The reactor is composed of a

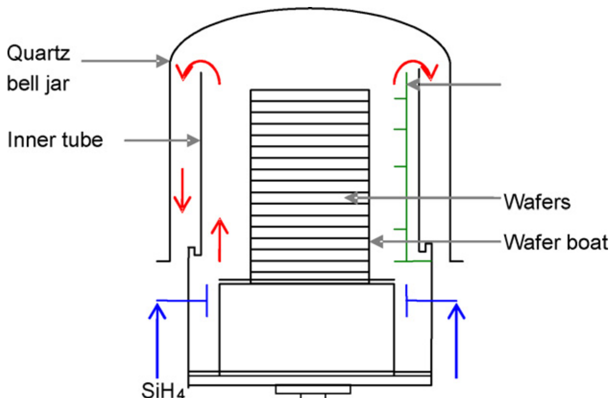

Fig. 1. Schematic representation of the industrial LPCVD reactor.

vertical quartz bell jar of 35 cm of internal diameter and 96.9 cm high, closed in its upper part by a bell of 6 cm high. In the bottom of the bell jar, a 36 cm high and 26.8 cm in diameter quartz pedestal supports the wafer boat. 170 ⟨1 0 0⟩ 8 inch silicon wafers per run can be treated. They are placed horizontally on the quartz boat, a few millimetres far from each other. Gases are fed at the bottom part of the reactor and flow upward perpendicular to the wafers in an annular region of 3.4 cm width around the boat. They are exhausted downward through the outer 4 cm wide gap between the bell jar and the inner tube.

The Si wafers were all thermally oxidized in dry mode at 1123 K. Two kinds of SiO_2 substrate are studied: a ''non-treated'' and a ''treated'' one. The SiO_2 thickness for the ''non-treated'' substrate was 5 nm. Prior to deposition, standard ozone cleaning was performed. According to Mazen et al. [5], dry oxidation mainly provides siloxane Si–O–Si surface bonds. For the ''treated'' substrates, a 7 nm thick dry silicon dioxide layer was grown and just before deposition, a thickness of 2 nm was etched off in a dilute (0.2%) aqueous HF solution.

Before field effect gun scanning electron microcopy (FEG SEM) analyses, an oxygen plasma was applied in order to clean wafers. This treatment was made to obtain a good visualization and to take off possible organic contamination occurred during manipulation or the storage of wafers. An in-lens equipment, Hitachi S5000, was used for FEG SEM NDs observation. NDs density was measured by direct counting on FEG SEM images. A high accelerate tension, 30 keV, and a tilt angle, 40°, permit to have a three-dimensional view and more easily discern Si NDs for the density counting. Si NDs have been observed on 5 mm × 3 mm cross section samples. For each wafer, otherwise precised, nine zones have been studied and an average value has been retained (one point at the wafer center, four points at 23 mm from the center and four points at 47 mm from the center on two perpendicular diameters).

Spectroscopic ellipsometry (KLA-TENCOR UV180 with wavelengths, between 250 and 750 nm) allowed the determination of the equivalent thickness and the silicon fraction of a continuous layer including vacuum and silicon (Bruggemann Effective Medium Approximation model). The diameter D of the as-deposited NDs is calculated in assuming hemispherical NDs and using the following equation:

$$\pi \times \frac{D^3}{12} = \frac{e_{Si}}{d_{dot}} \tag{1}$$

where e_{Si} is the equivalent thickness of silicon and d_{dot} is the NDs density (in number of NDs/cm^2); the intrinsic error on NDs density and radius measurements is estimated to 7%.

In Table 1 the operating conditions tested are given. Pure silane was used. Both treated and untreated substrates have been studied for temperatures ranging between 773 and 883 K and total pressures between 5 and 150 Pa. For confidentiality reasons, the pressures are not given. In Table 1 only the relative values of the various pressures tested (P1 < P2 < P3 ...) are mentioned.

Table 1
Experimental conditions tested

Run	Nature of the substrate	Temperature (K)	Inlet silane flow rate (sccm[*])	Pressure	Run duration (s)
T03	Untreated	883	100	P1	50
T04	Untreated	853	300	P3	25
T06	Treated	863	300	P3	15
T07	Treated	873	300	P3	7
T09	Untreated	853	300	P4	25
T10	Untreated	853	300	P2	25
T11	Untreated	853	100	P3	25
T12	Untreated	853	180	P3	25
T13	Untreated	853	300	P3	15
T14	Untreated	853	300	P3	35
T16	Treated	773	1500	P5	120
T17	Treated	773	1500	P5	180
T18	Treated	773	1500	P6	60
T19	Treated	773	1500	P6	120
T20	Treated	773	1500	P6	180
T21	Treated	823	1500	P5	60
T22	Treated	823	1500	P5	120
T23	Treated	823	1500	P5	180
T24	Treated	773	1500	P5	240
T25	Treated	773	1500	P5	300

[*] sccm: standard cubic centimeters per minute.

3. Results and discussion

3.1. Reproducibility and spatial uniformity

The dispersion in density and radius of NDs has been evaluated by calculating the standard deviation σ as follows:

$$\sigma = \sqrt{\frac{n\Sigma x^2 - (\Sigma x)^2}{n^2}} \quad (2)$$

where n is the number of density or radius values considered and x the values of the density or radius.

First, reproducibility experiments have been performed consisting in synthesizing NDs in the conditions of run T12 twice at several months of interval. Measurements have been conducted in the center of three wafers along the load, at its bottom, middle and top parts.

The standard deviations for densities are respectively equal to 2.9, 0.7 and 2.9% when rising along the load. The reproducibility for wafers placed in the middle part of the load is then excellent, whereas those of the two extremities are acceptable.

The standard deviations for radii are respectively equal to 1.9, 1.8 and 2.9%, when rising along the load. They are smaller than those of the densities and the smallest one is also that of the middle part of the boat. This could be due to a better reproducibility in temperature in the middle part of the load.

As a first conclusion, the reproducibility in the TEL reactor is satisfactory both in density and in radius, since the maximum standard deviation is equal to 2.9%.

Table 2
NDs densities and standard deviations (σ) measured for runs T03, T06 and T07

	Point 1	Point 2	Point 3	Point 4	Point 5	σ on a wafer (%)
Run T03						
Bottom	2.6×10^{11}	2.7×10^{11}	2.6×10^{11}	2.5×10^{11}	2.7×10^{11}	2.9
Middle	2.6×10^{11}	2.8×10^{11}	2.7×10^{11}	2.7×10^{11}	2.5×10^{11}	3.8
Top	2.7×10^{11}	2.8×10^{11}	3.0×10^{11}	2.7×10^{11}	2.9×10^{11}	4.1
σ from wafer to wafer (%)	–	1.7	6.1	3.6	11.3	
Run T06						
Bottom	7.5×10^{11}	8.2×10^{11}	8.57×10^{11}	8.5×10^{11}	8.2×10^{11}	4.6
Middle	7.9×10^{11}	8.2×10^{11}	8.5×10^{11}	8.4×10^{11}	8.5×10^{11}	2.7
Top	8.1×10^{11}	8.2×10^{11}	8.2×10^{11}	8.4×10^{11}	8.6×10^{11}	2.2
σ from wafer to wafer (%)	–	0	2.9	0.6	2	
Run T07						
Bottom	6.3×10^{11}	7.4×10^{11}	6.9×10^{11}	7.2×10^{11}	7.2×10^{11}	5.5
Middle	8.6×10^{11}	7.8×10^{11}	8.9×10^{11}	9.2×10^{11}	8.6×10^{11}	5.4
Top	8.5×10^{11}	9.5×10^{11}	9.1×10^{11}	9.7×10^{11}	1.0×10^{12}	5.6
σ from wafer to wafer (%)	–	11.1	12	12.4	13.3	

To safely use NDs in an industrial production of NVM, the dispersion in density and radius must remain lower than 10% both on a wafer and from wafer to wafer [17].

In the present study, NDs densities and sizes have been measured for runs T03, T06 and T07 for three wafers along the load, in the top, middle and bottom parts, in five points per wafer (one point at the wafer center, four points at 47 mm from the center on two perpendicular diameters, numbered points 1–5). Results are detailed in Table 2.

For run T03, the density in NDs is low and radii are high whereas an opposite trend is observed for runs T06 and T07. The low density of run T03 is due to the fact that the substrate was untreated. The positive effect of a HF pre-treatment mentioned in the literature [5] is then confirmed. The standard deviations are convenient since for density, they are lower than 5.6% and for radii, lower than 3.3%. No influence of the position into the load has been evidenced. The highest standard deviations have been obtained for run T07, probably because of its very short duration (7 s) which could involve incomplete diffusive transport phenomena in the inter wafer zones. But the whole results demonstrate that NDs depositions in the TEL LPCVD reactor are quite uniform on each wafer for the tested conditions.

For these three runs, a first analysis of the uniformity in NDs density and size from wafer to wafer along the load has been performed, considering the three positions along the load previously mentioned. Standard deviations for densities in each of the five points analysed per wafer are also detailed in Table 2. This uniformity is convenient for run T06 (σ lower than 3% for densities and than 1% for radii). It is acceptable for run T03 (σ lower than 6.2% for densities except for point five, and lower than 2.2% for radii). As detailed in Table 2, σ exceed 10% for densities of run T07 whereas they are lower than 6% for radii. The bad results of run T07 could be due to the fact that the shorter the run duration, the worse will be the wafer to wafer uniformity probably because steady state conditions are not reached especially in the top part of the load. The wafer to wafer uniformity is studied more deeply as a function of the operating conditions in the Section 3.2.

3.2. *Influence of operating conditions*

In order to better understand the phenomena involved in the nucleation and growth of silicon NDs, the influence of the main operating conditions on their density and radius has been studied in the central position of three wafers along the load (in its bottom, middle and top parts), for the nominal run T04 and for experiments T09 to T14 of Table 1. These runs correspond to untreated substrates. The influence of the inlet silane flow rate, total pressure and deposition time on the Si NDs density and size has been analysed. The operating conditions have *a priori* been chosen so as NDs were far from coalescence.

The temperature of synthesis has not been studied because Mazen et al. [5] and Nicotra et al. [12] have shown that this parameter does not significantly act on NDs densities and sizes on non-treated substrates.

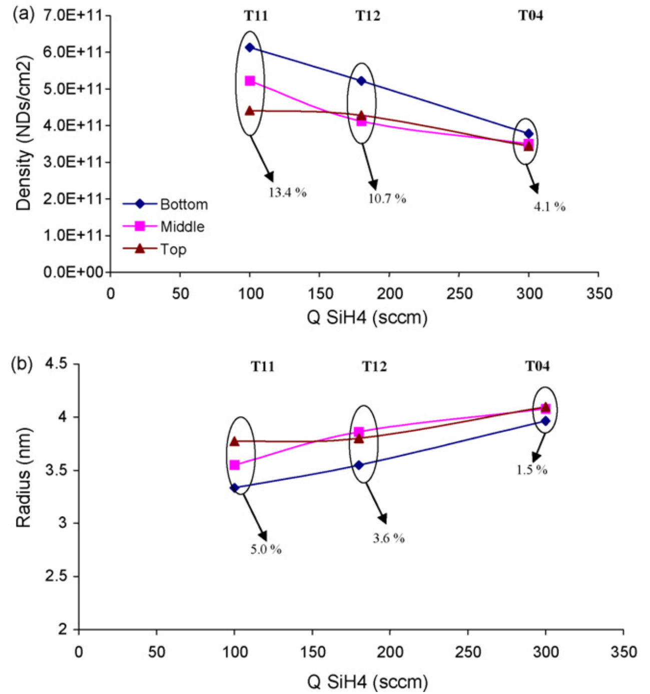

Fig. 2. Experimental evolution of silicon NDs (a) density and (b) radius versus inlet silane flow rate (runs T11, T12 and T04).

For these runs, an analysis of the uniformity in NDs density and size from wafer to wafer along the load has also been performed, considering the three positions along the load previously mentioned. The corresponding standard deviations are given on the figures below.

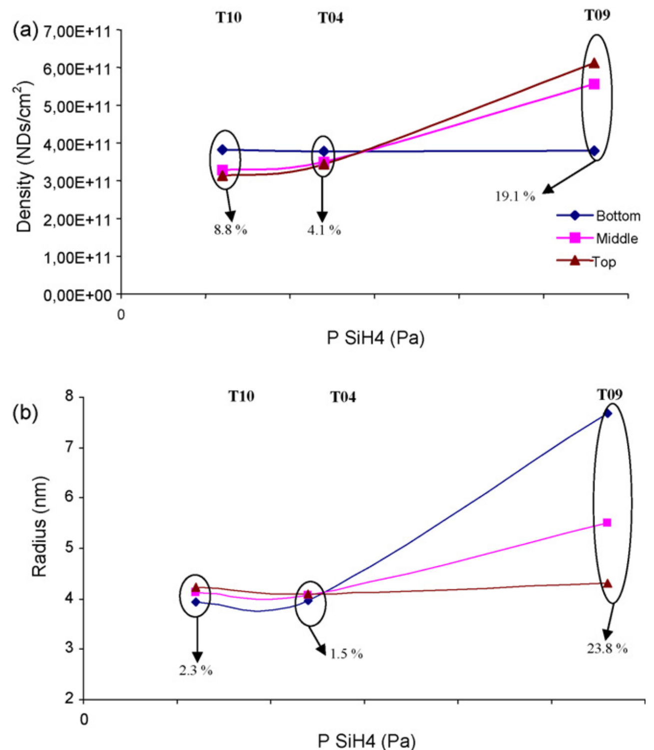

Fig. 3. Experimental evolution of silicon NDs (a) density and (b) radius versus silane pressure (runs T10, T04 and T09).

First, for the nominal run T04, NDs densities were close to 3.8×10^{11} NDs/cm^2 and radii around 4 nm. The wafer to wafer uniformity along the load is good since the standard deviations are respectively equal to 4.1 and 1.5% for NDs densities and radii. The mean density is low because substrates are non-treated wafers. The fact that the wafer to wafer uniformity is convenient could indicate that run duration of 25 s for the conditions tested (in particular untreated wafers), is sufficient to allow steady state conditions to be reached along the load.

The influence of the inlet silane flow rate Q_{SiH_4} on NDs density and radius is illustrated in Fig. 2 for runs T11, T12 and T04. If the inlet silane flow rate decreases, the NDs density increases whereas their mean radius decreases. These trends are sharper in the bottom part of the load than in its top part. As a consequence, the heterogeneities in density and size of NDs along the load increase when the inlet silane flow rate decreases. This result seems logical since it is well known that the depletion in silane along the load increases if the inlet silane flow rate decreases. So, an increase in silane flow rate allows improving the uniformity in density and size along the load but in decreasing NDs density and in increasing their size. The increase of the silane flow rate then favours growth to the detriment of nucleation. This could be explained by the fact that if Q_{SiH_4} increases, the amount of silane molecules near wafers and the deposition rate are increased, which would favour the deposition of continuous layers via more intense growth phenomena.

Fig. 3 shows the evolution of NDs density and size versus the silane partial pressure for runs T10, T04 and T09. When P increases, the NDs density increases but only for the middle and top parts of the load whereas the NDs radius also increases but only in the first half of the load. The fact that NDs density increases with pressure has already been observed [4,13]. This means that in the bottom part of the load, near the inlet zone, the arrival of matter is sufficient to ensure a maximum nucleation for the tested conditions. The existence of a saturation density corresponds to a well-known phenomenon [2,13,15]. The additional arrival of matter due to the pressure increase is then used to ensure NDs growth. In the top part of the load, near the reactor exit, the silane depletion is higher, nucleation is not at its maximum; an increase in pressure allows increasing the density

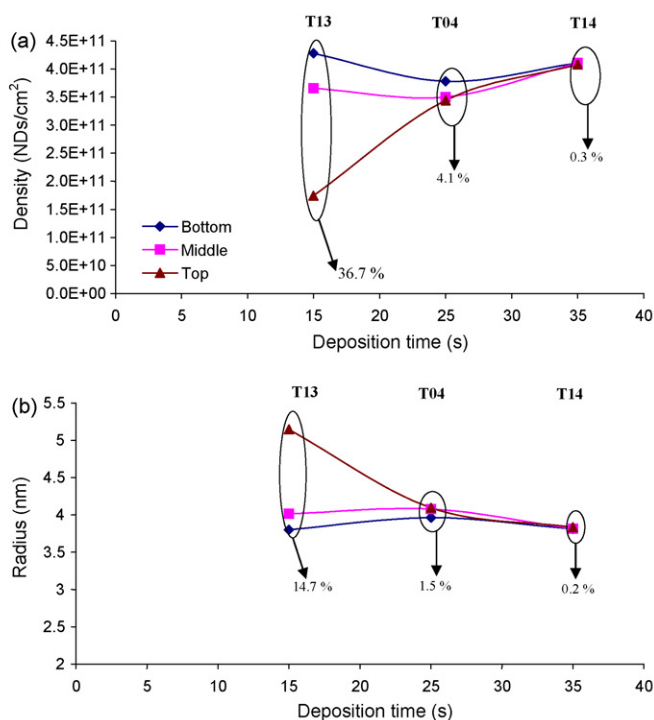

Fig. 4. Experimental evolution of silicon NDs (a) density and (b) radius versus deposition duration (runs T13, T04 and T14).

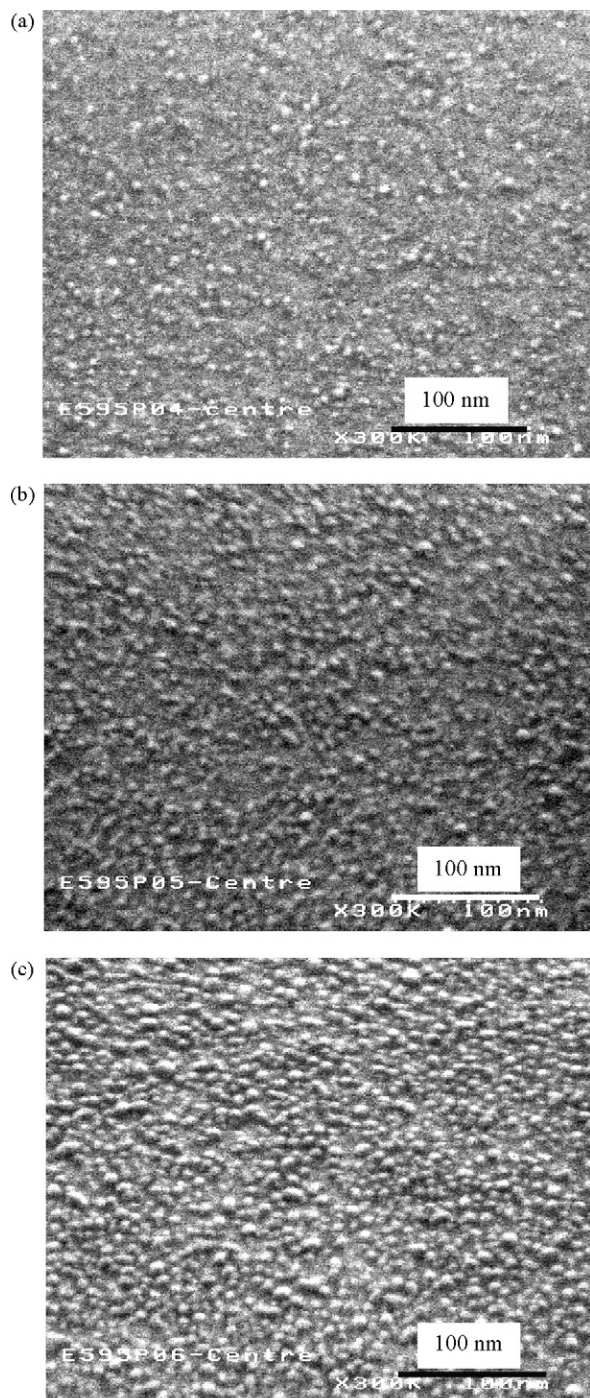

Fig. 5. FEG SEM views of silicon NDs deposited at 773 K after (a) 60 s, (b) 120 s and (c) 180 s (runs T18, T19 and T20).

and then nucleation without modifying growth. The nucleation phenomenon is here limited by the matter arrival.

It can be observed in Fig. 3 that an optimum of wafer to wafer uniformity is obtained for the intermediate pressure (run T04). For this run, the density and radius standard deviations are of 4.1 and 1.5%, respectively. At lower and higher pressures, this standard deviation is worse especially for the highest pressure. At this pressure, the kinetics of nucleation and growth phenomena are probably identical for all positions in the wafers boat.

Fig. 4 presents the evolution of NDs density and size as a function of run duration for runs T13, T04 and T14. It appears that in the top part of the boat, the density tends to increase with time whereas it is the opposite for the radius. For the two other positions, NDs densities and radii little change with the deposition time. The temporal evolution of density is similar to that of Nicotra et al. [9], Lombardo et al. [13] and Mazen et al. [15].

The slopes of evolution are much more marked for densities and radii in the top part of the load, far from the gas entrance. As observed for the influence of silane pressure, nucleation in the top part of the load is limited by the matter arrival and then favoured by long deposition durations.

To increase the run durations allows then to reach a better uniformity along the load, in favouring nucleation in the top part of the boat, where phenomena are the longest to establish. At 15 s, there is a bad standard deviation, 36% in densities and 14% in radii. At 35 s, standard deviations lower than 1% were obtained for NDs densities and radii but densities did not exceed 4.5×10^{11} NDs/cm^2 for the tested conditions.

3.3. Towards new ranges of operating conditions

The previous results indicate that to go towards an industrialization of the LPCVD process, it is necessary to improve the wafer to wafer uniformity in conditions for which high NDs densities are obtained, in particular by using treated substrates.

The objective of this part of the study was to improve the uniformity of NDs along the load and also, if possible, their density, in increasing the run duration. For the operating conditions studied in the two previous paragraphs, if run duration is increased, coalescence phenomena will probably rapidly occur. The only way of progress is then to modify the temperature and the total pressure to ensure conditions far from coalescence for run durations higher than 60 s. In Section 3.2, as in the literature [4,13], it has been observed that an increase in pressure exalts both the kinetics of deposition and of the nucleation. To obtain high NDs densities in increasing the silane pressure for long run durations, it is then necessary to decrease the temperature. According to Nicotra et al. [12], to modify the temperature around 873 K lowly influences the NDs density and size. As a consequence, temperature must be significantly decreased to work at higher pressure for longer run durations.

Temperatures comprised between 773 and 823 K and pressures between 60 and 150 Pa have then been tested. NDs have been synthesized on treated substrates in order to favour nucleation. Only one wafer in the first half of the load has been studied. The exact conditions tested correspond to runs T16 to T25 of Table 1.

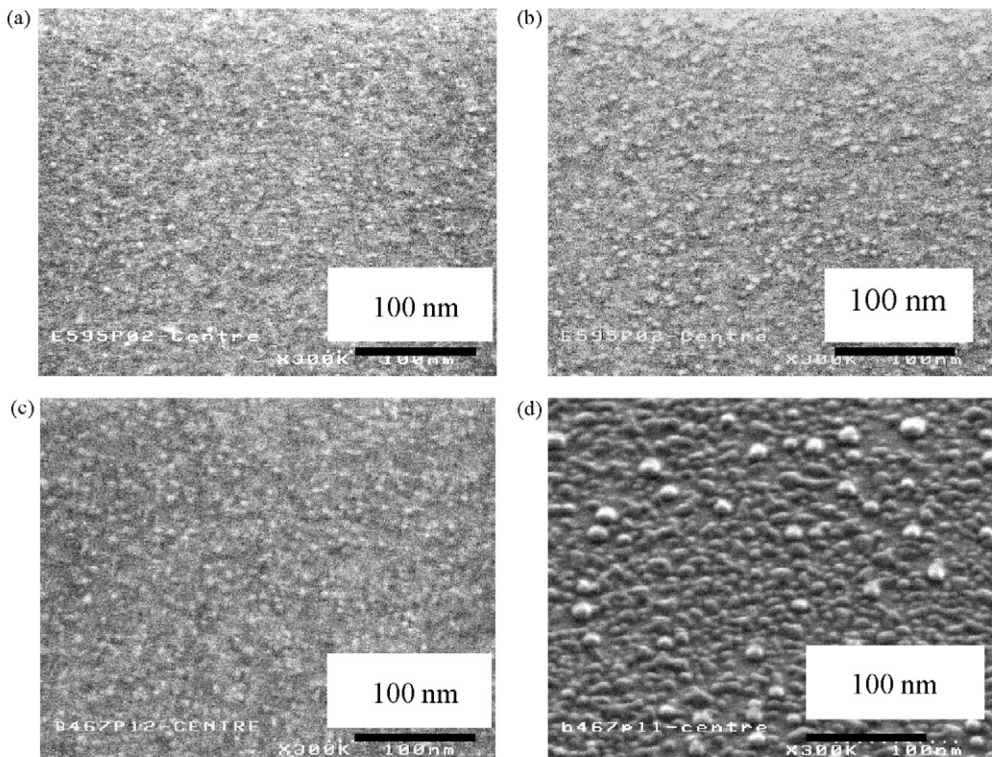

Fig. 6. FEG SEM views of silicon NDs deposited at 773 K after (a) 120 s, (b) 180 s, (c) 240 s and (d) 300 s (runs T16, T17, T24 and T25).

The trends of Section 3.2 concerning the influences of pressure and run duration have been found also for these operating conditions.

Coalescence occurred from 60 s of deposition at 823 K (runs T21, T22 and T23). NDs were no more visible at 120 and 180 s because from 120 s, a continuous silicon layer was formed. The deposition rate was then too high at 823 K. In these conditions, to obtain high NDs densities without coalescence, the run duration must be lower than 60 s, which is not the objective.

Fig. 5 shows the FEG SEM images of Si NDs formed at 773 K and the pressure P6 (runs T18, T19 and T20), for run durations between 60 and 180 s. Densities of 5.9×10^{11}, 7.9×10^{11} and 9×10^{11} NDs/cm^2 have been obtained for durations of 60, 120 and 180 s, respectively. Radii of 3 nm have been measured for durations of 60 and 120 s; this size increased to 4 nm for 180 s of deposition. For longer deposition duration, coalescence occurred.

For a lower pressure, P5, the kinetics of deposition is lower and thus the deposition duration must be longer. Fig. 6 shows the FEG SEM images of Si NDs formed at 773 K and the pressure P5 (runs T16, T17, T24 and T25). The run durations varied between 120 and 300 s.

For 120 s, the NDs were too small (< 1 nm) to be observed by FEG SEM. It is likely that for these conditions 120 s were close to the incubation period. The density of NDs was of 5.4×10^{11}, 6.5×10^{11} and 6.1×10^{11} NDs/cm^2 for 180, 240 and 300 s, respectively. The radius of NDs was equal to 3 nm for 180 s and 240 s and increased to 4 nm at 300 s. The decrease of density and the increase of size observed at 300 s probably indicate the beginning of coalescence.

So, with these new operating conditions, the deposition duration can be increased till 180 s at the pressure P6 and 240 s at P5 for high NDs densities (respectively 9×10^{11} and 6.5×10^{11} NDs/cm^2). With such run durations, the uniformity on the load should be improved but this point remains to be ascertained.

4. Conclusions

The reproducibility and spatial uniformity of silicon nanodots (NDs) area density and mean radius have been studied in an industrial tubular LPCVD reactor treating 170 wafers 8′ in diameter, per run. The influence of the main operating conditions on NDs features and wafer to wafer uniformity has also been analyzed.

The reproducibility of NDs synthesis and the uniformity of NDs density and size on wafer have been demonstrated for the conditions tested. The wafer to wafer uniformity was satisfactory (density and radius standard deviations <10%) for the whole LPCVD conditions tested except for low silane flow rates, high silane partial pressures and short run durations (<20 s). Indeed for the conditions tested, an increase in silane flow rate leads to an increase in NDs radius and to a decrease in density especially near the silane entrance zone, thus favouring the wafer to wafer uniformity. A rising of the silane flow rate thus exalts not only the overall silicon deposition rate but also the growth phenomenon to the detriment of the nucleation one. An increase of silane pressure enhances NDs density only for the second half of the load and increases NDs mean radius only for its first half. This non trivial evolution means that near the entrance zone, NDs density has reached its maximum saturation value and then the additional arrival of matter is used for NDs growth whereas the situation is drastically different near the exit zone where silane depletion is higher: nucleation is not at its maximum and the additional arrival of silicon precursor allows to increase nucleation. A similar trend has been observed for NDs density in the top part of the load when increasing run duration.

Original synthesis conditions have then been searched to reach both excellent wafer to wafer uniformities along the industrial load of wafers and high NDs densities. From previous results, it was deduced that the key was to markedly increase run duration in decreasing temperature and in increasing silane pressure. At 773 K, run durations as long as 180 and 240 s have thus allowed to reach NDs densities respectively equal to 9×10^{11} and 6.5×10^{11} NDs/cm^2 for the two highest silane pressures tested P5 and P6.

These results show that the way is today opened to ensure a rapid industrialization of quantum dots based silicon devices.

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