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Analytical Modelling of the Boron Diffusion and Etching Process for the Accurate Design and Fabrication by Bulk Micromachining Technology of the Silicon Capacitive Pressure Sensors for Biomedical Applications

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ABSTRACT

The boron silicon layers doped by diffusion from non-oxidizing (boron nitride – BN) sources and oxidizing (boron tribromide – BBr_3 or boron doped oxides B_2O_3) diffusion sources can be used as efficient etching-stop layers within the frame of the bulk micromachining technology to obtain silicon capacitive pressure sensors for biomedical applications. However, as the boron concentration during and after diffusion is not uniform, decreasing from the silicon surface to the silicon bulk according to the diffusion laws, a careful analysis should be applied to control the etching process, determining finally the thickness of the membrane of the capacitive pressure sensors for biomedical applications. Such an analysis, modelling and simulation of the boron diffusion and etching process is presented in this paper, showing that the etching rate and the etching time can be simulated by means of explicit relations deduced considering the distinct boron diffusion profiles obtained from non-oxidizing (BN) diffusion source and oxidizing sources, expressed by analytical solution of the boron diffusion equation. These results, useful both for the design and the control of the fabrication technological processes, show that the diffusion profile after diffusion from BN sources is more effective for the preparation of the silicon capacitive pressure sensors for biomedical applications, due to the specific diffusion profile.

Keywords: Silicon Capacitive Pressure Sensors for Biomedical Applications; Boron Diffusion Profile in Silicon; Boron Non-Oxidizing And Oxidizing Sources; Modelling and Simulation of the Boron High Concentration Profile; Calculation/Simulation of the Etching Rate and Etching Time

Introduction

The silicon capacitive sensors are used in a large range of biomedical applications, especially due their compatibility to the human body and due the experience accumulated in the fabrication of the microelectronic intelligent chips of microprocessors and microelectromechanical systems (MEMS) in the silicon planar technology, allowing the miniaturization, low cost production and versatile integration in measurement telematics line together

with silicon microprocessors in long-time monitoring medical procedures [1,2]. It is therefore not surprising the intensive development in an accelerated rhythm of these devices and their diversification, comprising the silicon MEMS-type microphone for the pulse measurement, silicon-based CMOS (complementary metal-oxide-semiconductor) and BiCMOS (combined bipolar junction transistor and the CMOS gate) for the detection of the heartbeat, of respiration and of the peripheral and cranial nerve activities, and even for refined small-scale molecular processes to detect the proteins and photo lipids, or the molecules of the deoxyribonucleic acid (DNA) [2,3]. The main problem concerning the achievement of silicon membranes with a certain well-defined thickness according to the design requirements and the sensitivity range of the silicon capacitive pressure sensors for biomedical applications [4], is strictly related to the control of the technological process within the bulk micromachining technology [5,6] referring to the boron diffusion profile at high concentrations used as a self-limiting etching process [7,8], and the etching process of the silicon [9,10].

Such a control requires a practical method to simulate the boron diffusion profile in silicon, according to the technological required parameters, time and temperature, and also to the type of the diffusion source [11]. As it was shown in a recent article [3], the boron diffusion process from oxidizing (BBr_3 and B_2O_3) sources in the silicon bulk (along the Ox axis) within the planar technology, can be well described and controlled if it is taken into account that the boron diffusion coefficient D is proportional with the square root of boron concentration C, i.e. $D \sim C^{1/2}$ on the high concentration range. In this paper it is modelled and simulated the boron diffusion in silicon from non-oxidizing boron nitride (BN) in comparison with the oxidizing (BBr_3, B_2O_3) sources, which show distinct characteristics, and consequently each of them needs a different system of relations for the accurate design and control of the fabrication of the silicon capacitive pressure sensors for biomedical applications by bulk micromachining technology. The modelling and simulation of the boron diffusion allows a subsequent simulation of the silicon etching, which is the key process for the achievement of the geometrical and material properties of the silicon capacitive pressure sensors for biomedical applications.

Modelling/Simulation of the Boron Diffusion from Non-Oxidizing/Oxidizing Sources

Although a remarkable effort was made to express the boron diffusion experimental data under the form of a unique "universal" boron diffusion profile in silicon, represented as a normalized function, i. e. C/C_j vs. x/x_j, where C is the boron concentration, C_j a concentration corresponding to a depth x_j of a lower-doped region assimilated with the depth junction, where the diffusion concentration would be non-concentration dependent (particularly $C_j = 10^{18}cm^{-3}$), it was demonstrated that the boron diffusion profile determined after diffusion from a BN source at the temperature T=1100°C does not obeys such a universal curve [12]. In Figure 1 there are represented comparative graphs, one of them with experimental data after diffusion from BN sources and another after diffusion from BBr_3 source and B_2O_3 deposited source. The solid lines represent the theoretical profiles, one taking into account a dependence of the boron diffusion coefficient D on C as D~C, and another one as $D \sim C^{1/2}$ respectively. In a previous analysis of the boron diffusion profile in silicon [13], a vacancy-assisted diffusion mechanism was supposed to operate during the diffusion, enhancing on the high concentration range (C>ni, ni – the intrinsic carrier concentration at the diffusion temperature) the diffusion process due to the contribution of the mono-charged vacancies.

However, the analyzed diffusion profile data do not obey the corresponding shape, as it can be seen also from Figure 1. The participation to the diffusion process of the self-interstitial atoms [14] induced by silicon oxidation of the silicon surface [15], determines a "diffusion tail" [16], with observable consequence on the boron diffusion profile during the diffusion from oxidizing sources. In contrast to this case, the diffusion from the BN source seems to be really carried out by mono-charged vacancy only. We explain this behavior by a different chemistry of the silicon surface, where the oxygen atoms cannot participate to the surface chemical reactions, due to the barrier role of a thin silicon nitride (Si_3N_4) layer against the oxygen diffusion, and therefore the vacancies based mechanism becomes really evidential: within the high concentration range C>ni, the positively charged vacancies carry the negatively ionized boron atoms, so on this range D~C, whereas within the intrinsic region (C<ni) the diffusion is performed with a diffusion coefficient non-depending on concentration, i.e. D~const. Such a behavior can be observed in Figure 1, where experimental results on boron diffusion at high concentration from non-oxidizing [17,18] and oxidizing diffusion sources [19,20] are compared with theoretical profiles obtained by simulation, as it is explained below.

According to the above discussion, it is properly to distinguish therefore between the diffusion from oxygen and non-oxygen related boron diffusion in silicon and to approach adequately the modelling and simulation of the boron diffusion in silicon at high concentration, in each of these two cases. Taking into account these observations, analytical solutions are presented, applicable to the diffusion from the BN as non-oxidizing source and from the chemical oxidative diffusion sources like BBr_3 or B_2O_3, taking into consideration the suitable dependence of the diffusion coefficient on concentration, i.e. D~C and $D \sim C^{1/2}$ respectively in the range C>ni. For this, it is convenient to express the diffusion coefficient of boron in silicon as D=Do(C/Co) during the diffusion from BN source, and as $D=Do(C/Co)^{1/2}$ during the diffusion from oxidizing sources, where Do=D(x=0) and Co=C(x=0).

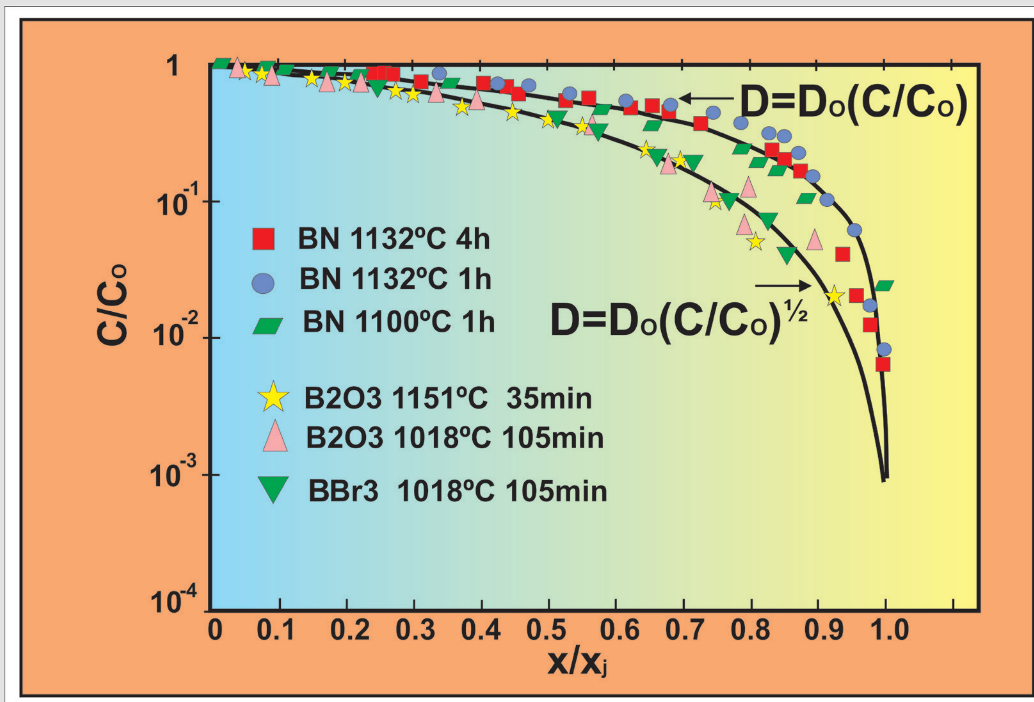

Figure 1: Normalized boron diffusion profiles C/Co vs. x/xj experimentally determined after diffusion from BN sources at 1100°C for 60 min. by SIMS technique (ref. [17]) and at 1132°C for 1h and 4h (ref. [18]) by sheet resistivity, and from B_2O_3 source [ref. [19]) and BBr_3 source (ref. [20]), determined by electrical sheet resistivity method, compared with the normalized profiles theoretically deduced in this paper for D=Do(C/Co) and D=Do(C/Co)$^{1/2}$ respectively.

The defined value Co is the surface concentration, corresponding actually with the limit solubility of boron in silicon. On the other hand, because the diffusion source is in permanent contact with the silicon surface, this case is equivalent with the diffusion from an infinite source to a semi-infinite solid. The diffusion depth can be therefore expressed in a normalized form as X=x/2(Dot)$^{1/2}$, where t is the diffusion time, and the concentration normalized to the surface value as c=C/Co. With these notations, the diffusion equation with partial derivatives in x and t:

$$\frac{\partial}{\partial x}\left(\left(\left(\frac{DoC}{Co}\right)\frac{\partial C}{\partial X}\right)\right)=\frac{\partial C}{\partial t} \quad (1)$$

is reduced to a unidimensional problem, expressible only in X, as following:

$$d/dX\left(cdc/dX\right)=-2X\left(dc/dX\right) \quad (2)$$

Although reduced to only one variable, this non-linear equation is still difficult to be solved. However, we have to observe that the equation (2) shows a fundamental particularity: because the diffusion flux of impurities, expressed in the normalized form dc/dX, decreases as c decreases [21], a finite characteristic value of X could be find, when both the flux and the concentration become null. Such a behavior is different from the diffusion described by a constant diffusion coefficient, case in which it is obtained a solution represented by the well-known erfc-type function, where $c \to 0$ only if $x \to \infty$. We will focus now to find a solution for the boron diffusion from non-oxidizing (BN) source in the high (C>ni) and low (C<ni) concentration range, for which D=Do(C/Co). As the diffusion flux on the high concentration range becomes zero at a certain value of X, the diffusion will continue only by means of a constant diffusion coefficient, naturally supported by the neutral vacancies for C<ni. We can therefore approximate the solutions on the two diffusion regions by the following expressions:

$$C=Co\times\left[1-\left(2/3\right)^{1/2}X-\left(1/2\right)X^{2}\right] \quad (3)$$

on the high concentration range (C>ni), and

$$Ci=ni\times erfc\left(rX\right)/erfc\left(rXi\right) \quad (4)$$

on the intrinsic range (C<ni).

These solutions are coupled by the condition:

$$Xi=\left(2/3\right)^{1/2}\left\{-1+\left[1+3\left(1-ni/Co\right)\right]^{1/2}\right\} \quad (5)$$

where Xi corresponds to C(Xi)=ni, r = (Do/Di)$^{1/2}$ and Di is the intrinsic boron diffusion coefficient, not concentration dependent.

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The solution on the high concentration range (C>ni) was deduced as a series of the variable X, showing a very rapid convergence. The finite value Xo=√(2/3) is obtained for C=0. At the diffusion temperatures T (°K), the corresponding values Xi are very close to Xo, where the concentration gradient strongly decreases to the limiting null value. Relation (1) is simple, but sufficiently accurate to simulate the boron diffusion profile with an approximation better than 1%. As it can be seen from the Figure 2, the agreement between the theoretical profile and the experimental data is very good, well supporting this analytical model. In this figure is shown the boron diffusion profile after diffusion of 120 min. at 1100°C from BN source, determined by two methods: by electrical sheet resistivity technique to measure the concentration of the free carriers, and by emission spectrography to determine the total amount of boron in each removed layer [22-25]. As it can be seen from this figure, the entire boron amount is electrically active, participating to the electrical conduction.

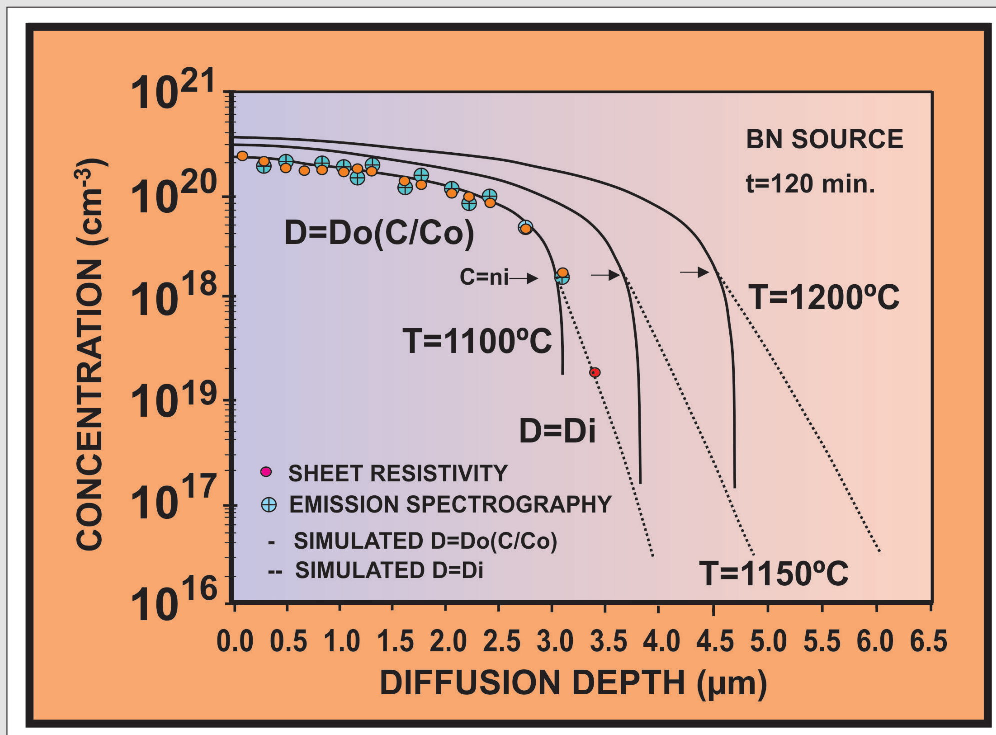

Figure 2: Boron diffusion profile in silicon experimentally determined by electrical (sheet resistivity) method and by chemical (emission spectrograph) technique [22] and simulated (the solid line continued by the dashed line) after the diffusion from BN source at 1100°C, 1150°C and 1200°C for 120min.

In the same figure are represented some simulated boron diffusion profiles after diffusion of 120min. at 1150°C and 1200°C, allowing a further simulation of the etching process to obtain silicon membranes. For simulation, there were used the data reported earlier by Vick and Whittle [19], because within a study on the solid solubility recently reported [26], it was shown that these data are well supported by more than one documental source. To maintain the coherence of the simulation, the values of Di and Co (the solid solubility of boron in silicon) were obtained from the experimental data earlier reported [19]. An accurate expression for ni seems to be still under debate [27], but for the diffusion purposes, the used data for the intrinsic carrier concentration were calculated from a formula reported relatively recently, claiming accurate results [28], as follows:

$$ni = 5.29\times10^{19}\left(T^0K/300\right)^{2.54}\exp\left(-6726/T^0K\right) \quad (6)$$

From the experimental data on the intrinsic diffusion coefficient Di and solid solubility [19], the following expressions were used for calculations:

$$Di = 6\times10^{-7}\exp\left(-1.7eV/kT^0K\right) \quad (7)$$

$$Co = 1.1\times10^{23}\exp\left(-0.713eV/kT^0K\right) \quad (8)$$

where k is the Planck constant, the concentrations ni and Co are expressed in cm^{-3} and the diffusion coefficient Di in cm^2/sec. The diffusion from BBr_3 source is assisted actually by an oxidation process to form a B_2O_3 glass on the silicon surface, which becomes

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an infinite diffusion source for the boron atoms, but also a source of self-interstitial atoms, contributing to the boron diffusion process and an additional formation of the "tail" region, as discussed above The diffusion profile in this case is expressible on the high concentration range (C>ni) taking into account a form $D=Do(C/Co)^{1/2}$ as a consequence of a combined vacancy-interstitial assisted mechanism of atomic diffusion. With this form of the diffusion coefficient and the same signification for Do, Xo and ni, the profile simulation of the boron diffusion from oxidizing chemical sources on the high concentration range (C>ni) could be expressed by the following solution of the diffusion equation, as it was recently shown [3]:

$$C = Co\left[\left(1-\left(\sqrt{1/6}\right)\right)X-\left(1/3\right)X^{2}\right]^{2} \quad (9)$$

which shows that the concentration becomes null for $Xo=\sqrt{3/2}$.

Modelling and Simulation of the Etching Rate and Etching Time for the Design and Fabrication of the Micromechanical Structures

During the chemical etching process, the etching front advances to the silicon bulk with a constant etching rate, which starts to decrease as the concentration of the boron atoms increases within the boron-doped layer [9,10]. Therefore, the etching rate depends gradually on the boron doping concentration of the doped silicon layer and for the simulation of this process, the analytical expressions of the boron diffusion profile presented above are corroborated with the results earlier reported [29], showing the dependence of the etching rate on doping level of silicon, if an uniform bulk concentration is considered. So, it was previously shown [29], the etching rate R of the uniformly boron-doped silicon layers can be expressed as a function of the boron concentration by the relation:

$$R/Ri = 1/\left[1+\left(1+C/Cc\right)^{a}\right]^{4/a} \quad (10)$$

where R_i represents the etching rate of the low-doped silicon substrate, Cc is a critical value in the range (2-4)×1019 cm^{-3}, which depends on the etching temperature Te and on the concentration of the etching solution of the alkaline type (KOH, NaOH, LiOH) or of EDP, and "a" is a shape parameter. The reported results indicate that a=4 for 10% KOH (NaOH, LiOH) and ethylene-diamine-based (EDP) solution type S, and a=2 for 24% alkaline-type solutions [29]. Considering that the etching front advances to the silicon doped surface along the diffusion direction, but in the contrary sense, with a rate R=dx/dt [30], the chemical etching time t necessary to etch a boron diffusion layer can be deduced as following:

$$t = -\int_{xj}^{x}\left(R/Ri\right)^{-1}\left(dx/Ri\right) = \int_{xj}^{x}\left(R/Ri\right)^{-1}\left(dx/Ri\right) \quad (11)$$

and can be therefore finally expressed as:

$$\Delta t/\tau = \left(1/xj\right)\left[\int_{0}^{x}\left(1+\left(C\left(x\right)/Cc\right)^{a}\right)^{4/a}dX/_{X\in[xi,xj]} + \int_{xi}^{x}\left(\left(1+\left(Ci\left(x\right)/Cc\right)^{a}\right)^{4/a}dX/_{X\in[xi,xj]}\right) \quad (12)$$

where x_j is the junction depth used in rel. (12) as a normalizing parameter, Δt = t-to (to – the initial moment at x_j boundary), xi the depth where C=ni, and $\tau = x_j/Ri$. As Ce defines a critical value of reaction, where this becomes significantly diminished by doping, the contribution of the second term in rel. (12) on the intrinsic variation range x∈[xi,xj] is actually negligible, because Cc and ni vary in approximatively the same range of values in the discussed temperature interval. Therefore, for the calculation xj can be substituted actually by xi, which is the depth where Ci=ni. In Figure 3 it is shown the variation of the ratio R/Ri with the depth x into the silicon boron-doped layer after diffusions at 1100°C, 1150°C and 1200°C by using a chemical etching solution for which a = 4 and a=2, and the corresponding variation of the etching necessary time. The value of the etching temperature of solution for the etching process simulated in Figure 3 is 110°C, for which Cc=3.0×1019 cm^{-3}. By fitting the experimental data of Cc previously reported [29] with an Arrhenius law, the following relation is obtained:

$$Cc = 5.5\times10^{19}\ \exp(-0.025eV/kT^{0}K)cm^{-3} \quad (13)$$

which allows to calculate Cc for any temperature of etching solution.

From Figure 3 it can be observed a slower variation of the etching process in a solution corresponding to a=2, but both solutions (a=2 and a=4) are efficient for the fabrication of the silicon membranes, showing values of about 103 times higher near silicon surface than the values corresponding to Ri in the silicon low-doped bulk. Moreover, it can be also seen that the increase of the diffusion temperature allows to obtain larger values of the thickness of the micro-mechanical elements if the same etching duration is considered, and an increasing of the etching duration if the same value of the thickness of the micro-mechanical elements is considered. However, after diffusion at higher temperatures, larger widths of the silicon membranes could be obtained for the same time of the etching process, predictable from rel. (12). Similar conclusions could be drawn from the simulation graphs showing the variation of the etching rate and etching time during an etching process of boron-doped silicon layers after diffusion from oxidizing BBr_3 diffusion earlier reported [3].

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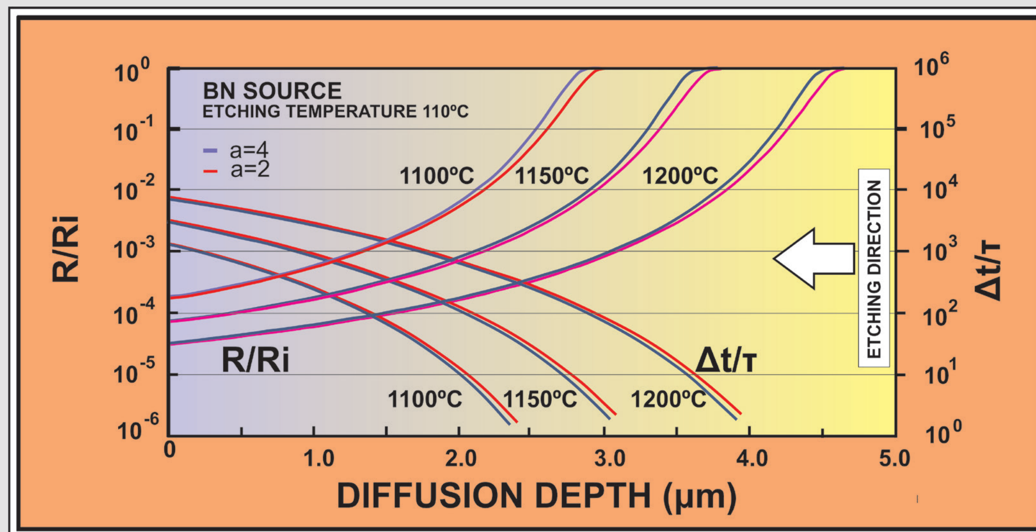

Figure 3: The simulated R/Ri ratio and Δt/τ during the chemical etching process of the boron silicon layers diffused from BN source at 1100°C, 1150°C and 1200°C for 120 min, in an etching solution at 110°C.

Although the variation of the etching rate as a function o the etching solution type is not too large in both cases, the control of fine differences is very important not only to sharply define the thickness of the micro-mechanical elements, but also to eliminate the diffused regions where the concentration gradient is sufficiently large to induce mechanical stress into the silicon membrane. It is to be note also that the doping gradient of the boron diffusion profile after diffusion BN sources are lower near the silicon surface in the highly doped region than after diffusion in oxidizing conditions, as it can be observed from Figure 1 Such a slower variation is beneficial, because of lower stress is induced in the achieved membranes due to the gradient of concentration. The high diffusion time, the lower diffusion gradient is obtained in the highly doped region, so the simulation permits an accurate and correct managing of the etching process depending on the required properties of the silicon membranes. It is also important to observe that the behavior of the boron-doped layers by the diffusion from BN sources is better than that obtained by oxidizing source, because the gradient of concentration is higher near the depth corresponding to xi, as it can be seen from Figure 1 In other words, the boron profile obtained by diffusion from BN sources is close to a step-type profile, ideal for a self-stop etching process. The suitable control both of the thickness of the silicon membrane and of the residual stress inside of the membrane depend therefore not only on the diffusion temperature, time and the characteristics of the etching process, but also on the boron diffusion source, and the simulation results presented above allow a suitable management of the technological parameters, serving also as predictable data for the design of the micro-mechanical elements and device structures obtained by bulk micromachining technology, in particular for the design and fabrication of the silicon capacitive pressure sensors for biomedical applications.

Conclusion

The distinction between the boron diffusion processes using non-oxidizing (BN) and oxidizing (BBr_3) sources is analyzed, showing that in the first case the boron diffusion profile can be calculated taking into account a dependence of the boron diffusion coefficient D on the boron concentration C of the form D=Do(C/Co), as predicted by the vacancies-induced diffusion mechanisms, and of the form $C=Co(D/Do)^{1/2}$, as a results of combined self-interstitial and vacancy mechanisms of diffusion in the second case. The agreement between the experimental and analytical results is very good, supporting the theoretical models. The obtained solutions of the diffusion equations on the high concentration range show simple and accurate expressions for the determination of the diffusion profiles in silicon and can be used for further evaluation of the chemical etching rate and etching time for the design and fabrication of the micro-mechanical elements, in particular of the silicon membranes for the achievement of the silicon capacitive pressure sensors for biomedical applications. The derived expressions to calculate the chemical etching rate and the etching time allow an appropriate and fine-tuning control of the etching process as a function of the variation of the boron diffusion concentration in silicon after diffusion at various temperature, time duration and diffusion chemical source, demonstrating the high applicability of the analytical results in a large range of variation of the technological parameters. It was shown that, besides the accurate design and technological control of the membrane thickness, the presented results allow an effective control of the

residual stress in the silicon membranes responsible for micro-mechanical deformations, and the corresponding elimination/reduction of the stressed silicon doped regions. The diffusion profiles from BN sources are more efficient stop-layers than that obtained from oxidizing source, because of a more pronounced dropping of concentration near C=ni. Moreover, the slower gradient near the silicon surface in this case, induces lower residual stress, so assures a better quality of the silicon membranes.

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