Thickness evaluation of mesoporous silicon layer using ultrasonic method

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Abstract
The manufacturing processes of porous silicon (PoSi) now allow samples with variable depths and porosities to be obtained. Thickness can be critical to ensure reliability in microelectronic applications. However, thickness measurement methods are generally destructive or strongly limited by layer thickness or pore diameter. Therefore in this study a non-destructive ultrasonic method based on an immersion insertion-substitution technique is investigated. Wavelength in ultrasonic method is much larger than optical ones, allowing to observe porous silicon layers having larger pore diameter or higher thickness. Total thickness of the wafer (675 microns) and high sound speed in pure silicon (8450 m.s⁻¹) require high centre frequencies transducers. The acoustic parameters of these samples, such as velocity and attenuation, are measured using time domain analysis. These measurements are compared with those obtained through a frequency based method, using a homogenization approach for the porous layer based on Biot’s Theory.

Keywords: Biot’s theory, porous silicon, ultrasonic testing

1. Introduction

Electrochemical etching of silicon (Si) in HydroFluoric acid (HF) based solutions is nowadays a very well-known process and numerous reports have been made on the porous silicon (PoSi) growth mechanisms [1], [2]. In the wide range of morphologies that can be produced, one can distinguish microporous Si, a sponge-like structure of 1 to 5 nm crystallites, macroporous Si, mostly tubular pores with diameters up to 50 nm and, an intermediate category, the mesoporous Si with sizes between 5 and 50 nm [3]. This material has found many applications in microelectronics. One can point out for example, the use of mesoporous Si as an isolating substrate for RF applications [4], [5] or the application of the high specific surface of PoSi in sensors [6]. Mesoporous silicon is mainly produced from highly doped silicon. When such a material is immersed into an HF solution, the interface reacts as a Schottky contact. Then, a very thin space charge region is localized at the electrolyte/ Si interface and acts as a passivation region against silicon dissolution on the pore walls. The hole diffusion, at the origin of the reaction, is then mainly ensured by a tunneling mechanism located at the pore tip [7]. The observed morphologies are consequently most of the time very anisotropic (Figure 1). Moreover, in high current density regimes, the pore growth direction is governed by the crystallography. In many applications, one needs to obtain thick layers with a constant porosity or constant pore diameters in short times.

The measurement of the PoSi parameters, such as thickness or porosity, cannot be used to monitor the fabrication process. Using the strong relationship between the mechanical properties of a medium and the properties of an elastic wave travelling through it, ultrasonic non-destructive testing can be a good way to measure these parameters [8]. Moreover this method is contactless, which is also an essential feature for microelectronics due to the contamination risk.

2. Materials
2.1 Fabrication of the PoSi layers

The PoSi layers were formed by the anodization in HF based solutions of highly doped p-type Si (10-50 mΩ.cm)<100> samples with thicknesses varying between 650 and 700 µm. This material is known to produce mesoporous silicon with pore diameters between 10 and 100 nm [7]. The electrochemical etching was performed in a double tank electrochemical cell developed by AMMT. The HF concentration is 30% and the surfactant used is acetic acid with volume ratios HF (50%): Acetic acid: H₂O of 4.6 : 2.1 : 1.5. The anodization was performed in a galvanostatic mode. A current density of 28 mA/cm² was fixed to obtain an average porosity of 50%. Then, the duration determined the total thickness of the porous layers. In our case, a duration of 87, 174 and 270 minutes lead to 100, 200 and 300 µm respectively. The average technological dispersion is in the range of 5 to 10%.

2.2 Material specification

The samples used for this study are square-shaped crystalline silicon wafers on which a circular shaped PoSi layer with a one inch (2.54 cm) diameter is etched. This diameter is higher than that of the active surface of the piezoelectric transducer to ensure that all the ultrasonic signal passes through the porous medium.

In order to guarantee the precision of the final results, the wafer thickness is checked using a digital micrometer and values are summarised in Table 1. The physical parameters of the crystalline silicon and water used for this study are noted in Table 2 [9]. The speed of sound in water is strongly dependant on temperature [10]. In our study, the temperature of the water is kept constant at 20°C.

Table 1: Sample geometrical characteristics.
Table 2: Material properties at 20°C.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Wafer thickness</th>
<th>Expected PoSi thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>674±1 µm</td>
<td>0 µm</td>
</tr>
<tr>
<td>2</td>
<td>675±1 µm</td>
<td>250±25 µm</td>
</tr>
<tr>
<td>3</td>
<td>683±1 µm</td>
<td>200±20 µm</td>
</tr>
<tr>
<td>4</td>
<td>680±1 µm</td>
<td>100±10 µm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Silicon</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density ρ</td>
<td>2329 kg/m³</td>
<td>1000 kg/m³</td>
</tr>
<tr>
<td>Speed of sound C</td>
<td>8433 m/s</td>
<td>1480 m/s</td>
</tr>
</tbody>
</table>

3. **Time domain measurement**

3.1 **Signal reconstruction**

Figure 2 presents the insertion/substitution principle used to determine the velocity and the attenuation of the acoustic waves propagating in the samples based on broad-band transmission [11]. First, a measurement of the signal transmitted through the reference medium (water) is performed (top of Figure 2). The sample is then inserted between the two transducers (bottom of Figure 2).

The reference signal ref (t) is recalled at Eq. 1. This equation can be divided into a impulse response h(t) of the emission/reception system, an attenuation effect depending on the distance d and αw which is the attenuation coefficient of water in neper per meter and a diffraction effect diff(d,f), depending on the center frequency f.

\[
ref(t) = h(t, f) \exp(-\alpha_w d) \text{diff}(d,f)
\]  \hspace{1cm} (1)
The distance $d$ and the frequency range of transducer are unchanged during the measurement process, so we can consider the attenuation and the diffraction of the signal in water as a constant. Given that, the reference signal can be interpreted as the impulse response of the whole system $h'(t)$:

$$h'(t) = ref(t)$$  \hspace{1cm} (2)

Taking the hypothesis that porous silicon and bulk silicon are not dispersive media, signal recorded after propagation through the sample can be defined as a summation of reference signal:

$$s(t) = \beta_0 h'(t - \tau_0) + \sum_{i=1}^{\infty} \beta_i h'(t - \tau_i)$$  \hspace{1cm} (3)

where the parameters $\beta_i$ are the amplitude of the $i^{th}$ incoming signal. This parameter depends on the reflection/transmission coefficient, and so on the acoustical impedance ratio, the attenuation in each medium and the diffraction in each medium. The diffraction in the sample can be considered as negligible because of the thickness of the sample compared to the tank size and the distance between the transducer and the sample. $\tau_i$ is the delay of the $i^{th}$ signal through the sample compared to the reference. Parameters $\beta_0$ and $\tau_0$ are usually used to calculate respectively the effective speed of sound and the effective attenuation in the sample. These informations can be used to calculate the acoustical parameters of the system.

To simulate the signal through the sample, an iterative algorithm is used where the delay and the amplitude of each echoes are calculated using the material properties, such as attenuation, speed of sound and the density. Amplitude are calculated using transmission and reflection coefficient on each interface (respectively $T_{ij}$ and $R_{ij}$):

$$T_{ij} = \frac{2 \cdot Z_i}{Z_i + Z_j}$$  \hspace{1cm} (4)

$$R_{ij} = \frac{Z_j - Z_i}{Z_i + Z_j}$$  \hspace{1cm} (5)

where $Z_n = \rho_n \cdot c_n$ is the acoustic impedance of medium $n$. The density $\rho_{posi}$ of the porous silicon can be defined as shown in Eq. 6 where $p$ is the porosity rate, $\rho_w$ is the density of water and $\rho_{Si}$ is the density of bulk silicon.

$$\rho_{posi} = p \cdot \rho_w + (1 - p) \cdot \rho_{Si}$$  \hspace{1cm} (6)

### 3.2 Inverse problem solving

The optimization method chosen for this study is based on a Genetic Algorithm (GA), which was developed by Holland [12]. This method mimics a natural selection process in order to ensure convergence towards a global solution even for complex problems. In this representation, each variable (or gene) $g_i$ used for the optimization is comprised between 2 boundaries, respectively $g_i^{min}$ and $g_i^{max}$. These boundaries can be very large and only determined by physical limits. An individual is composed by one or several genes, which can be grouped into chromosomes.

Solutions of GA have a low dependence on initial population so convergence is possible even if several local solutions exist [13].

Figure 3 describes the GA loop during the optimization. First, each individual is created by randomly choosing its genes between their boundaries. Then, GA loop begins and population is evaluated using a fitness function according to the experimental data. Fitness of each chromosome allows the selection process to be done and reproduction processes...
(mutation and crossover) are performed in order to obtain a new generation of chromosomes. This new generation is again evaluated and this loop continues until the end condition is reached.

Historically, most of the GA have been implemented in binary representation because of its low cardinality [14]. However, this is far from real-based physical systems where GA can be used for optimization so some authors have proposed to use a representation better adapted to physical systems [15]. Then, Goldberg developed a floating-point based genetic algorithms. This optimization method is currently used in several research fields [16], [17].

Figure 3: typical Genetic Algorithm loop.

In this study, we have chosen a fitness function based on the least-square distance $d$ between theoretical and experimental signals. For simplicity, the inverse of distance $d$ is used. In order to limit premature convergence risk, a parameter $\beta$ is added to the distance $d$ to limit the maximal value of fitness function (Eq. 7) [16]. This parameter is set at 1, enabling a good convergence of this optimization problem [16].

$$f = \frac{1}{d + \beta} \quad (7)$$

The selection of parents is performed using a universal stochastic method in order to optimize convergence [18]. Elitism, which consists in keeping the best individuals, is used during this study in order to enhance the convergence of the GA [13].

The crossing over procedure used in this study is arithmetical [19], based on the averaging of two randomly chosen parents $g^k_1$ and $g^k_2$ with a random weighting factor $\alpha$, comprised between 0 and 1, to create a new chromosome $g^{k+1}$:

$$g^{k+1} = \alpha g^k_1 + (1 - \alpha)g^k_2 \quad (8)$$

One of drawbacks of GA is the fact that retrieved parameters are always an approximation of the global optimum (precision depends on the number of generations). In order to converge at a closer value, Michalewicz proposed a non monotonous mutation operator which allows the input parameter range to be refined during optimization, using an ageing effect (Eq. 9) [20]. The advantage of this operator is that it limits the mutation rate, which allows a good convergence, and improves the optimization at the end by using a smaller search space.

$$g^{k+1} = \begin{cases} g^k + \Delta(t, g_{\text{max}} - g^k) & \text{if } s < \frac{1}{2}, \\ g^k - \Delta(t, g^k - g_{\text{min}}) & \text{otherwise}, \end{cases} \quad (9)$$

where $s$ and $r$ are random numbers between 0 and 1. $g^k$ represents the gene value at the $n^{th}$ generation and parameter $b$ allows nonlinear behavior of mutation to occur. The value of $b$ is set at 1.2, enabling a good final tuning without increasing premature convergence risk [20].

GA population size and mutation rate choice is very important to guarantee a good convergence behavior during optimization. In this study, population size is set at 20
chromosomes and the mutation rate at 20%, as proposed in literature for optimization procedures [16], [21] to ensure a good convergence of real-based genetic algorithms. End condition is fixed at 500 generations in order to achieve a good precision on retrieved parameters.

4. Results

Measurements are made with transducers having a center frequency of 15 MHz. The speed of sound in the porous silicon with a porosity of 50% is close to 4500 m/s, giving a wavelength of about 300 µm in porous layer. Therefore, a huge overlapping is observed on recorded signal through samples and direct measurement of acoustical parameters is not yet possible. Limits taken for the values of celerity and thickness for the genetic algorithm are given Table 3. These ranges are large enough to characterize most of the possible porous silicon layers which can be obtained using this manufacturing method.

<table>
<thead>
<tr>
<th>variable</th>
<th>min</th>
<th>max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Speed of sound (m/s)</td>
<td>2000</td>
<td>6000</td>
</tr>
<tr>
<td>Thickness (µm)</td>
<td>0</td>
<td>500</td>
</tr>
</tbody>
</table>

Thickness retrieval using this method are very close to values obtained using the frequency based method using biot’s theory, as shown Table 4. It can be observed that the maximal discrepancy is 21 µm, which is as low as 7% of wavelength. Moreover, retrieved speed of sound is similar for all the etched sample, with a value of 4750 m/s.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Time domain method</th>
<th>Frequency method [16]</th>
<th>Discrepancies between methods</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>12 µm</td>
<td>8 µm</td>
<td>4 µm 1.3 %</td>
</tr>
<tr>
<td>2</td>
<td>249 µm</td>
<td>270 µm</td>
<td>21 µm 7 %</td>
</tr>
<tr>
<td>3</td>
<td>160 µm</td>
<td>178 µm</td>
<td>18 µm 6 %</td>
</tr>
<tr>
<td>4</td>
<td>117 µm</td>
<td>110</td>
<td>7 µm 2.3 %</td>
</tr>
</tbody>
</table>

5. Conclusion

This method has shown that acoustical parameters of a two layers structure can be retrieved using inverse problem solving even if overlapping is observed on recorded signals. Comparison with frequency based method has shown a good agreement with a maximal discrepancy of 7% of the wavelength. In further works, modelling of slow wave will be implemented for the porous layer to improve the characterisation to geometrical aspect of pores.
References


