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INVESTIGATION OF ULTRASONIC ABSORPTION IN THE MHz FREQUENCY RANGE BY SILICON SUBSTRATES WITH A BUILT-IN POROUS SILICON LAYER

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Abstract

The present study is focused on the development and the characterization of an innovative substrate to optimize the axial resolution of ultrasonic transducers on a silicon substrate that are dedicated to ultrasound imaging. The substrate must efficiently dampen the wave propagation to avoid the degradation of the axial resolution of ultrasound images. In this study, the proposed approach implements a silicon substrate with a built-in acoustic damping layer made of porous silicon. Porous silicon layers with thicknesses of less than 100 μm and porosities varying from 27 % to 62 % were fabricated and their substrate resonances were characterized. The experimental results obtained in the frequency range from 6 MHz to 10 MHz show that the substrate acoustic damping is controlled by adjusting the characteristics of the porous silicon layer; a significant damping of 70 % is demonstrated with only 70 μm of porous silicon.

Keywords: Porous silicon, Ultrasound transducers, Substrate, Attenuation

I. INTRODUCTION

The present study focused on the development and the characterization of an innovative substrate to optimize the axial resolution of ultrasonic transducers on a silicon (Si) substrate that are dedicated to ultrasound imaging. Significant progress has been achieved in the field of ultrasound imaging since its first use for medical application in the middle of the 20th century. The concomitant advances in the ultrasound transducer technologies and microelectronic industry have paved the way for new imaging modalities, such as real-time 3D imaging [1], intravascular imaging [2] and, more recently, wearable ultrasound patches [3,4]. These recent modalities have required the development of more sophisticated systems which include electronic and ultrasonic transducer integration on the same chip. These chips are supported by the development of new transducer technologies that are fabricated on a Si substrate, such as micromachined ultrasonic transducers, which are based on micrometric vibrating membranes [5,6]. These technologies are based on the same manufacturing processes as those used for microelectronics. Consequently, they make possible the 3D integration of the ultrasonic transducer and the front-end electronic required for the imaging system on the same chip. For this purpose, the integrated electronic circuits are usually bonded onto the rear side of the ultrasonic transducer substrate and the electrical connections are transferred from the transducer side to the substrate rear side through the substrate [7].

The acoustic resonance of the substrate (the so-called thickness-mode resonances) is one of the drawbacks inherent to the technologies of ultrasonic transducers on Si substrates. Indeed, a part of the ultrasonic wave produced by the transducer is transmitted to the supporting substrate. These substrate waves interfere with the transducer acoustic response and result in a distortion of the impulse response [8]. Especially for imaging purposes, the thickness-mode

resonance is responsible for an elongation of the impulse response which leads to a degradation of the axial resolution. Furthermore, parts of the substrate wave are radiated into the propagation medium leading to imaging artefacts [9]. It is then fundamentally necessary to dampen the substrate resonance to preserve the image quality. To date and to our knowledge, no method compatible with a 3D integration of ultrasonic transducers attenuates these substrate waves. Some authors have proposed to glue a millimetre-thick film on the transducer on the opposite side [10]. This film is composed of an attenuating material such as tungsten powder in an epoxy matrix. Nevertheless, the attenuating film avoids the 3D integration that is required for gathering the ultrasound sensing function and maintaining the front-end electronics on the same chip. Another strategy to reduce the influence of the wave propagation is reduce the thickness of the substrate [10]. The substrate thickness-mode resonance frequency is then increased and shifted outside the frequency range of interest. This second method allows for 3D electronic integration but does not avoid the substrate waves propagation, which remain a potential artefact source for imaging.

Hereby, the substrate is based on a Si wafer with a built-in acoustic damping layer in porous silicon (PS) to reduce the wave propagation in the substrate. PS is usually obtained by the electrochemical etching of a Si wafer in a hydrofluoric acid-based electrolyte [11]. This process is fully compatible with microelectronic processes and the joint integration of a localized PS area; further, the electronic components have already been proposed [12]. Then, the use of a PS layer to dampen the substrate waves is compatible with the 3D integration of the ultrasonic transducer and the electronic components. The pore dimensions of the PS ranges from a few nanometres to tens of micrometres, and several layer structures can be achieved as summarized in [13] (e.g., periodic, branched or random). The layer characteristics (i.e., thickness and porosity) are controlled by adjusting the etching conditions

[14]. The porosity, defined as the ratio between the void volume and the Si volume, mainly depends on the hydrofluoric acid concentration of the electrolyte and the current density applied during the electrochemical etching [15], while the layer thickness depends on the etching duration. It should be noticed that PS formation by electrochemical etching is a top-down technique; therefore, the Si layer thickness decreases, but the total thickness of the Si/PS bilayer remains constant and equal to the initial thickness of the Si wafer. The opportunity to locally change the properties of the Si substrate has already found an interest in numerous applications such as optical systems (e.g., photonic crystals for sensing [16]) or acoustics (e.g., phononic crystals for filtering [17]). The role of the porosity on the PS bulk acoustic properties (e.g., the characteristic acoustic impedance and attenuation) has already been discussed in the literature [18–20]. For air-filled PS layers, the attenuation is proportional to the porosity, whereas the acoustic impedance is inversely proportional to the porosity, and is comprised between the characteristic acoustic impedance of the Si and those of the air. In other words, high porosity ($> 60\%$) is favourable to a significant attenuation but its acoustic characteristic impedance ($< 9 \times 10^5 \text{ kg} \cdot \text{s} \cdot \text{m}^{-2}$) differs strongly from that of the Si ($19.7 \times 10^5 \text{ kg} \cdot \text{s} \cdot \text{m}^{-2}$), leading to an important acoustic mismatching at the Si/PS interface (transmission coefficient < 0.6). Furthermore, from a technological point of view, the synthesis of a stable highly porous and/or thick PS layer ($> 100 \mu\text{m}$) is non-trivial. Indeed, the mechanical stress in this type of PS may result in cracking and peeling of the porous layer or a millimetre curvature of the wafer [21]. Hence, the main challenge is to find a suitable trade-off between the thickness and the porosity of the PS layer to obtain significant acoustic damping (i.e., superior to the measurement uncertainties, which were estimated to be 16 % in this work).

In this study, a series of Si substrate samples with built-in PS layers of different morphological characteristics were fabricated and characterized. A description of the fabrication method and the corresponding samples characteristics is proposed in the first part of this article. The quality factor of the thickness-mode resonance was monitored to evaluate the changes to the acoustic damping of the substrate. Both the experimental protocol developed for this purpose and the theoretical model, which is based on transmission line theory used to interpret the results, are described in the first part of the article. In the second part of the article, the experimental and theoretical results are discussed and analysed. The phenomena responsible for the substrate resonance damping are discussed and generalized rules for the design of thin damping layers are proposed.

II. METHODOLOGY

2.1. Sample fabrication

The PS layers were obtained by electrochemical etching in an electrolyte composed of 30 wt. % of hydrofluoric acid and 25 wt. % of acetic acid. This type of electrolyte was chosen to obtain porosity values between 30 % and 60 %. In this study, only mesoporous silicon obtained from highly doped Si and formed of nanometre-scale voids were investigated (see Fig. S1 in the supporting information) since highly doped Si is favourable to electronic integration (i.e., it is used as the bottom electrode for the ultrasonic transducers and to transfer the electrical contacts through the substrate). The PS layers were formed on 133 cm² of a microelectronic standard and commercially Si wafer that are available from MEMC; 6-inch (100)-oriented p-type Si wafers have a thickness of $508 \pm 15 \mu\text{m}$ and a resistivity of 20 m Ω .cm. The porosity was controlled by adjusting the current density from 10 mA/cm² to 50 mA/cm² and 80 mA/cm². The thicknesses of the PS layers were kept under 80 μm to preserve the mechanical stability of the PS layers. The etching durations were adjusted to get layer thicknesses of 80 μm (i.e., the maximum value) for each current density, and the intermediate

thicknesses of approximately 20 μm and 40 μm were arbitrarily chosen. For each sample, SEM cross-sectional observations were used to estimate the PS layer thicknesses (h_{PS}). The average porosity (Φ) was evaluated by gravimetry [22,23]. For this purpose, the silicon wafer mass (m_1) was measured before PS etching. Freshly formed PS samples were dried on a hot plate at 120°C for 10 min before the second weighting (m_2). Finally, the porosity was calculated from the weight variation, the etched area (A), the layer thickness and the silicon density ($d_{Si} = 2330 \text{ kg} \cdot \text{m}^{-3}$) as described in Eq. (1).

$$\Phi = \frac{m_1 - m_2}{A \cdot h_{PS} \cdot d_{Si}} \quad (1)$$

The sample etching conditions and the PS characteristics are summarized in Table 1. The low porosity samples were obtained at 10 mA/cm² and had average porosity values between $27 \pm 1 \%$ and $34 \pm 1 \%$, depending on the layer thickness. The average porosity values of the moderate porosity samples (50 mA/cm²) were measured between $40 \pm 3 \%$ and $49 \pm 3 \%$ and average porosity values of the high porosity samples (80 mA/cm²) were between $47 \pm 3 \%$ and $62 \pm 4 \%$. It should be noticed that the etching of the thickest PS layers at 50 mA/cm² and 80 mA/cm² led to local cracks in the PS layers at the edges of the PS region. Finally, PS layers with a progressively amount of porosity were investigated with an aim of improving the energy transfer between the Si and the PS by reducing the acoustic impedance mismatch at the interface between the two layers. Three PS layers with controlled porosity were synthesized as described in a previous work [24] with layer thicknesses similar to those of the other samples. The average porosity values of the PS samples with controlled porosity were in the range of $50 \pm 2 \%$ to $44 \pm 2 \%$ and were the result of a gradual decrease of the local porosity with depth (from 55 % at 25 μm -in-depth to 35 % at 80 μm -in-depth). The Si/PS wafers were cut into 3x3 cm² square samples. For homogeneity issues, only the centre part of each wafer was used for the acoustic characterizations. A Si sample with the same

characteristics as the material used to fabricate the PS layer was also characterized as a reference to monitor the acoustic damping effect.

2.2. Experimental protocol of the acoustic characterization

The technique used for the acoustic characterization is based on the electrical impedance measurement of an ultrasonic transducer in contact with the characterized sample (i.e., the Si substrate or the Si/PS substrate). The front side of the ultrasonic transducer is then acoustically loaded by the plate under test. The experimental setup used for in-contact measurement is represented in Fig. 1. The Si side of the plate under test was placed in contact with the transducer while the PS side was free, in contact with air, and avoiding any risk of fluid infiltration into the pores. This configuration was chosen because, in the final application, the ultrasonic transducers are layered on the Si side whereas the PS side is maintained in contact with the air. The ultrasonic transducer and the electrical cables were kept in a fixed position. The sample was placed on a mobile support to be able to contact the transducer with a micrometric accuracy. A water-based acoustic gel (Aquasonic 100, Parker) was used to ensure the acoustic coupling between the transducer and the sample. A few millimetre-thick hollow plastic holder was used to hold the sample, to keep the centre of the sample rear side in contact with the air and to avoid any acoustic coupling between the holder and the sample. The sample and its holder were put onto a force sensor to control the force applied between the transducer and the sample (for additional information see Fig. S2 in the supporting information). The use of a force sensor was mandatory to ensure the repeatability and the reproducibility of the characterization method. For each measurement, the contact force was fixed at 13 N. In these conditions, the thickness of the gel layer is sufficiently thin to neglect the wave propagation in it without any risk of damage to the sample. The ultrasonic transducer was chosen in such way that the resonance frequencies of the samples

(between 8.06 MHz and 8.55 MHz for a Si thickness of $508 \pm 15 \mu\text{m}$) were in agreement within the transducer bandwidth. The measurements were carried out using a flat unfocused single element transducer (U8436023, Olympus) with a 6 mm diameter that was centred at 10 MHz with a frequency bandwidth of 60 % at -6 dB. The ultrasonic transducer was connected to an impedance analyser (HP E5100A, Agilent). The electrical impedance measurements were performed at frequencies around the resonance frequency of the substrate thickness-mode in a frequency range sufficiently broad to correctly measure the shift in the resonance frequency caused by the PS layer; the frequency range was also kept narrow enough to preserve the accuracy for the quality factor measurement. A frequency range of [6 MHz-10 MHz] was found to be the best trade-off for all the samples with a pitch of 4 kHz (the smallest pitch available with our equipment).

A procedure was specifically implemented to determine the input acoustic impedance (Z_a) of the sample under test from the experimental electrical impedance curves. An ultrasonic transducer reciprocally transforms a voltage V and current I into a mechanical wave defined by a velocity v in the surrounding medium and a pressure P . For all linear systems, the ultrasonic transducer can be modelled as a two-port electrical network defined by a 2x2 matrix, as described in [25]. The relationship between V , I , P and v is given by Eq. (2).

$$\begin{bmatrix} V \\ I \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \times \begin{bmatrix} P \\ v \end{bmatrix} \quad (2)$$

A and C represent two transfer functions (V/P and I/P respectively) for the clamped acoustic boundary conditions, meaning that the incident velocity field v is null. B and D represents two transfer functions (V/v and I/v , respectively) for the free acoustic boundary conditions, meaning that the incident pressure P is null. When the transducer is loaded by a semi-infinite medium of known characteristic acoustic impedance Z_a or a plate with input acoustic impedance $Z_a(f)$, its electrical impedance $Z_{el}(f)$, at a given frequency of analysis f is defined by Eq. (3).

$$Z_{el}(f) = \frac{V(f)}{I(f)} = \frac{A' \cdot Z_a(f) + B'}{C' \cdot Z_a(f) + 1} \quad (3)$$

where $A' = A/D$, $B' = B/D$ and $C' = C/D$.

If A' , B' and C' are known, $Z_a(f)$ is determined from the measured electrical impedance $Z_{el}(f)$. A' , B' and C' are determined from a calibration step based on the measurement of $Z_{el}(f)$ for three reference media. In this work, air ($Z_{air} \approx 0$), water ($Z_{water} = 1.5 \times 10^5 \text{ kg.s.m}^{-2}$) and isopropyl alcohol ($Z_{IPA} = 0.92 \times 10^5 \text{ kg.s.m}^{-2}$) [26] were chosen. For a given sample, the input acoustic impedances are thus obtained from Eq. (4):

$$Z_a(f) = \frac{Z_{el}(f) - B'}{A' - C' \cdot Z_{el}(f)} \quad (4)$$

The resonance of the plate leads to a peak (i.e., a local maximum) on the real part of the input acoustic admittance $Y_a(f) = 1/Z_a(f)$ (i.e., the acoustic conductance) at the fundamental frequency of the substrate thickness mode resonance f_r . The resonance peak quality factor (Q) is defined from the acoustic conductance by using Eq. (5), where f_1 and f_2 are the frequencies for which the peak amplitude is divided by 2 and f_r is the resonance frequency.

$$Q = \frac{f_r}{f_2 - f_1} \quad \text{with } f_2 > f_1 \quad (5)$$

The quality factor value of the resonance peak reflects the global acoustic damping of the Si/PS substrate; therefore, this value gives information on the acoustic attenuation in the substrate. The experimental protocol was set in such a way that the thickness of the acoustic gel layer was thin enough to be negligible and to ensure the repeatability of the contact between the transducer and the characterized sample. Using these conditions, a comparison of the quality factors of the resonance peak of the Si sample and those the Si /PS samples can give information on the attenuation in the PS layer. For each measurement, the quality factors were determined from the acoustic conductance at the resonance frequency. The acoustic calibration procedure was carried out before each sample characterization to avoid any drift

over time in the measurements. The acoustic admittance of the Si reference sample was systematically controlled before each measurement campaign on Si/PS samples to check the data consistency (see Fig. S3 in the supporting information).

2.3. Modelling of the input acoustic admittance of the Si/PS bilayer

The input acoustic admittance of the Si/PS plates ($Y_{Si/PS}$) was modelled by means of the transmission line theory as defined in [27]. Based on this method, the input acoustic admittance of the bilayer ($Y_{Si/PS}$) is given by Eq. (6).

$$Y_{Si/PS} = \frac{1}{Z_{Si}} \frac{Z_{Si} Y_{PS} + j \cdot \tan(k_{Si} \cdot h_{Si})}{j Y_{PS} Z_{Si} \cdot \tan(k_{Si} \cdot h_{Si}) + 1} \quad (6)$$

where Y_{PS} is the input acoustic admittance of a stress-free PS layer defined by:

$$Y_{PS} = \frac{1}{j \cdot Z_{PS} \cdot \tan(k_{PS} \cdot h_{PS})} \quad (7)$$

Z_{Si} is the characteristic acoustic impedance of Si ($Z_{Si} = 19.7 \times 10^5 \text{ kg} \cdot \text{s} \cdot \text{m}^{-2}$ along the (100) axis) [28]. k_{Si} is the longitudinal wavenumber in the Si layer with thickness h_{Si} and k_{PS} is the longitudinal wavenumber in the PS layer with thickness h_{PS} . Acoustic losses in both materials can be considered by introducing a loss factor δ under the complex form of the wavenumber [29], as described in Eq. (8).

$$k = \frac{2 \cdot \pi \cdot f}{v \cdot (1 + j\delta)} \quad \text{with } (\delta \ll 1) \quad (8)$$

where v is the longitudinal wave velocity.

The acoustic properties of the PS layer were chosen to agree with the literature. The porous frame was maintained in air; in these conditions, the longitudinal velocity in the PS layer (v_{PS}) was considered to only change with the layer porosity, as defined in Eq. (9).

$$v_{PS} = v_{Si} \cdot (1 - \Phi)^K \quad \text{with } 0 < \Phi < 1 \quad (9)$$

where v_{Si} is the longitudinal velocity in Si ($v_{Si} = 8430 \text{ m. s}^{-1}$ along (100) axis [28]) and K is a constant that depends on the layer morphology and pore size. From [30], K was approximated according to the substrate resistivity. In our case, K was fixed at 0.8, which corresponds to a 20 m Ω .cm resistivity. Finally, the PS layer density (d_{PS}) was deduced from the layer porosity and the Si density from Eq. (10).

$$d_{PS} = d_{Si} \cdot (1 - \Phi) \quad \text{with } 0 < \Phi < 1 \quad (10)$$

Two series of simulations, in agreement with the fabricated samples, were undertaken for PS layers with fixed porosities of 30 % and 60 %. The total thickness of the plate was maintained at 500 μm . The PS layer thickness was varied from 20 μm to 80 μm ; therefore, the underlying Si layer thickness was, reduced from 480 μm to 420 μm . Finally, the substrate resonance frequencies and peak quality factors were extracted from the simulated acoustic conductance.

III. RESULTS AND DISCUSSION

3.1. Influence of the PS layer porosity and thickness

In Fig. 2, the acoustic conductance of the moderate porosity Si/PS samples is compared to the acoustic conductance measured on the Si reference sample (there are additional measurements provided in Fig. S4 of the supporting information). The substrate resonance peaks of the Si/PS samples are clearly distinct from those of the Si reference sample. Indeed, for all the PS layer thicknesses, the substrate resonance of the Si/PS sample are observed at higher frequencies than that of the Si reference sample. In addition, the resonance peak of Si/PS substrates is damped compared with that of the Si substrate, even with thin layers. The characteristics of the resonance peaks were extracted for all of the samples to properly evaluate the influence of the PS layer features on the substrate resonance in comparison with the Si substrate. The resonance frequency of the Si substrate was found to be equal to 8.09

MHz, which is in good agreement with the expected value and the quality factor of the substrate resonance peak of the Si was equal to 190. This value is kept as a reference for the next part of the study.

The evolution of the resonance frequencies and quality factors of the Si/PS bilayer resonance peaks as a function of PS layer thickness are, represented in Fig. 3 and Fig. 4, respectively, for all the samples etched under a constant current density. In all of these cases, the Si/PS substrate resonance frequency increases with the PS layer thickness. The resonance frequency shifts from 8.31 MHz to 8.47 MHz for the low porosity samples with thicknesses values equal to 25.6 μm and 77.6 μm , respectively. For the highest porosity samples, the substrate resonance ranges from 8.41 MHz for the 25.6 μm thick PS to 8.63 MHz for the sample with a PS layer thickness of 73.1 μm . The quality factor is also modified by the presence of the PS layer on the rear face of the samples. The quality factor is systematically lower for the Si/PS substrates than for the Si reference plate (i.e., from 150 down to 60 for the Si/PS samples compared to 190 for the Si reference plate). These results reveal for the first time that a PS layer with a thickness much smaller than the initial wavelength in the Si has a noticeable impact on the substrate thickness-mode resonance in air. A significant decrease of 70 % of the quality factor peak for the thickest and the highest porosity PS layer is obtained. As expected, the diminution of the resonance peak quality factor depends on the layer porosity. More surprisingly, these results suggest that low porosities ($\approx 30\%$) are more favourable to attenuate the substrate resonance for a PS layer thickness lower than 40 μm . Indeed, a 40 % peak damping is observed for the 26- μm thick low porosity sample in air.

These experimental investigations were compared to the set of simulations of the Si/PS bilayer to get a better understanding of the phenomena responsible for the peak damping and

the frequency shift. Losses in Si and PS were thus arbitrary fixed at, respectively, $\delta_{Si} = 2.5 \cdot 10^{-3}$ and $\delta_{PS} = 3 \cdot 10^{-2}$ to be consistent with the experimental results. The resonance frequencies and the quality factors as a function of the PS layer thickness are plotted in Fig. 5 and Fig. 6, respectively, for 30 % and 60 % porosity values and PS layer thicknesses ranging from 20 μm to 80 μm . An increase of the PS layer thickness leads to a shift of the resonance frequency towards higher values (cf. Fig. 5), which is consistent with the experimental results. For a given PS layer thickness, the frequency shift is the highest for the highest porosity. Therefore, the quality factor decreases with an increase of the PS layer thickness; tendencies similar to those found previously are observed here. For PS layer thicknesses less than 50 μm , the most efficient way to dampen the peak is to use the lowest porosities (cf. Fig. 6). This tendency is reversed for PS layer thickness values greater than 50 μm , where the quality factor of the substrate resonance peak is smaller for the highest porosity layers.

Considering the PS layer thickness range, the observed phenomena can be interpreted by making an analogy with the behaviour of a quartz crystal plate acoustically loaded by a thin polymer film [31–33], which dampens and decreases the resonance frequency of the Si resonator (i.e., the Si layer). The resonance frequency diminution is caused by a mass effect and the frequency shift is proportional to the mass deposited. The resonance frequency of the Si layer is then shifted towards lower frequencies as much as the mass of the PS layer is high. As a consequence, for a given PS thickness, the resonance frequency of the substrate is proportional to the porosity (cf. Fig. 3 and Fig. 5). Furthermore, the Si layer thickness decreases with an increase of the PS layer thickness when the Si sample is converted into PS. Hence, the resonance frequency of the Si layer increases with the thickness of the PS layer (i.e., the resonance frequency of the Si resonator is inversely proportional to its thickness). The evolution of the resonance frequency is thus a combination of two phenomena. First,

reducing the Si layer thickness down increases the resonance frequency. Second, the PS layer acts as a mass and decreases the resonance frequency of the Si resonator.

A similar analysis can be done to explain the evolution of the quality factor. As described in [33], three different regimes are observed depending on the ratio between the thin film thickness and the acoustic wavelength in the film at the resonance frequency of the substrate (h/λ). For the thinnest films ($h/\lambda < 0.05$), the quality factor is inversely proportional to the mass. Thus, for a given PS layer thickness, the damping is higher for the lowest porosity as suggested by the experimental and theoretical results. For thicker layers ($h/\lambda < 0.1$), the damping still depends on the reverse of the mass but also highly depends on h/λ (a third order relationship). As a result, for the same thickness, a smaller wavelength is favourable to acoustic damping. Finally, the last regime corresponds to the thin film resonance (i.e., $h = \lambda/4$), for which the maximum damping is reached. The acoustic wavelength in the PS layer is inversely proportional to the porosity; thus, when the PS layer increases, the highest porosities reach the optimal $\lambda/4$ -value faster than the lowest porosities. The crossing point observed on the evolution of the quality factors shown in Fig. 3 and Fig. 6 is a consequence of these phenomena. It should be noted that a thickness of $\lambda/4$ corresponds to the optimal damping, however the acoustic damping for two PS layers with an equivalent h/λ but different porosities is not the same since the mass must be considered. Indeed, for an equivalent h/λ , the lowest porosities are favourable for achieving a higher acoustic damping (as illustrated in Fig. S5 of the supporting data). In conclusion, for PS etched at a constant current density, thick PS layers of low porosity are more suitable to efficiently dampen the wave propagation in the substrate, and optimal damping is achieved when the layer thickness reaches the $\lambda/4$ -value.

3.2. Influence of the in-depth variation of the porosity

Under electrochemical etching conditions with constant current density, the PS layer porosity monotonically increases with depth [34] owing to HF molecule diffusion issues in the nanoscale size pores [35]. From a technological point of view, this behaviour can induce a mechanical stress or even film lift-off in the case of thick and/or high porosity PS layers [21]. Thus, from a technological point of view, the etching of sufficiently thick layers ($> 200 \mu\text{m}$) with a low average porosity ($< 30 \%$) cannot be considered to enhance the performance of the damping layer. As a consequence, it was interesting to evaluate other ways to improve the mass-loading effect. One possibility was to take advantage of the PS electrochemical etching technique to control the porosity in-depth and thus its in-depth acoustic properties. Hereby, we investigated PS layers with an acoustic impedance gradient (i.e., decrease of the porosity) from the layer surface towards the interface with the Si. The aim was to reduce the acoustic impedance mismatch at the interface between the two layers to improve the energy transfer between the two materials as well as to investigate the impact of the PS layer micro-structure on the acoustic damping. The resonance frequencies and quality factors extracted from the measurements of the controlled porosity samples are presented on Fig. 7 and Fig. 8, respectively, and are compared to the moderate porosity samples (i.e., those with a similar average porosity values). If only the mass-loading effect is considered, the tendencies observed for controlled porosity samples are expected to be similar to the those observed from the moderate porosity samples, since they have comparable average porosity values. Nevertheless, the frequency shift is lower for the controlled porosity samples than for the moderate porosity samples. Moreover, the resonance frequencies of the two thinnest controlled porosity samples are lower than those of the low porosity samples of equivalent thicknesses (cf. Figs. 3 and 4). This first evidence indicating that the influence of the in-depth layer properties is also observed on the evolution of the quality factor. Indeed, in the

investigated range of thicknesses, the quality factor of the controlled porosity samples is almost constant; the quality factor of the moderate porosity samples decreases as the layer thickness increases. It is interesting to note that, for the thinnest layers (20 μm and 40 μm), the lowest quality factor is obtained from the controlled porosity samples; thus, the thickness of the damping layer can be reduced by adapting the acoustic impedance of the PS to that of the Si layer. Further investigations are required to understand the phenomena involved in such a structure. However, these results show that better performance in terms of attenuation can be obtained by a proper control of the in-depth porosity.

IV. CONCLUSIONS

Several sets of silicon/porous silicon substrates were fabricated. Their resonance frequencies and the attenuation of their thickness-mode resonance values were measured in a relevant ultrasound imaging range of frequencies (from 6 to 10 MHz). The results indicate that porous silicon layers are suitable to be used to optimize the axial resolution of ultrasonic transducers integrated on a silicon substrate. More specifically, it was shown that this optimization can be achieved by a proper combination of layer porosity and thickness. In the samples tested, the most efficient combination was a porosity of 62 % with a thickness of 73 μm which allowed to achieve a damping of 70 % of the substrate resonance. A descriptive model based on the transmission line theory was used to define the general rules for the design of optimal built-in porous silicon acoustic dampers. It was shown that the most effective way to dampen the substrate resonance is to reduce the porosity and increase the layer thickness (i.e., close to a quarter of the wavelength in the porous silicon). Finally, it was shown that by progressively decreasing the porosity of the layer to adapt the acoustic impedance from the silicon towards the porous silicon, the thickness of the porous silicon layer can be reduced to 27 μm . These results are, to the best of our knowledge, the first evidence of the efficiency of silicon/porous

silicon substrates dampening the substrate resonance to the axial resolution of ultrasonic transducers dedicated to ultrasound imaging. The design rules reported in this article are transposable to other thin attenuating layers suitable for integration with ultrasound-based micro-systems currently developed to overcome the next challenges of ultrasound imaging (e.g., high-frequency transducers, flexible devices, *in vivo* imaging, etc.). Further investigations must be carried out to clarify how the porous silicon morphology (i.e., the pore diameter and shape) and/or the evolution of the in-depth porosity influence the substrate damping. The finality of this work is to integrate an ultrasonic transducer on a silicon substrate with a built-in porous silicon acoustic damping layer to evaluate its impulse response (i.e., the pulse duration and bandwidth) and the potential benefit on the axial resolution of ultrasound images.

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FIGURE CAPTIONS

Figure 1: Schematic representation of the experimental setup (additional information and relevant dimensions are shown in Fig. S2. in the supporting information).

Figure 2: Real part of the acoustic admittance of the moderate porosity samples (blue lines) with various thickness PS layers compared to the acoustic admittance of the reference Si plate (black line). The data are normalized in comparison with the maximum value of the real part of the acoustic admittance of the Si plate.

Figure 3: Resonance frequencies extracted from the real part of the acoustic admittance of the Si/PS samples etched at constant current densities (low porosity: black circles, moderate porosity: blue squares and high porosity: red triangles) as a function of the PS layer thickness. The error bars correspond here to the measurement uncertainties of 4 kHz previously estimated samples. The resonance frequency measured for the Si plate was equal to 8.09 MHz.

Figure 4: Quality factors extracted from the real part of the acoustic admittance of the Si/PS samples etched at constant current densities (low porosity: black circles, moderate porosity: blue squares and high porosity: red triangles) as a function of the PS layer thickness. The error bars correspond here to the measurement uncertainties of 11 previously estimated samples. The quality factor measured for the Si plate was equal to 190.

Figure 5: Theoretical resonance frequencies extracted from the real part of the acoustic admittance simulated for the Si/PS samples (30 %-porosity PS: black circles and 60 %-porosity: red squares) as a function of the PS thickness.

Figure 6: Theoretical quality factor extracted from the real part of the acoustic admittance simulated for the Si/PS samples (30 %-porosity PS: black circles and 60 %-porosity: red squares) as a function of the PS thickness.

Figure 7: Resonance frequencies extracted from the real part of the acoustic admittance of the Si/PS samples with controlled porosity (controlled porosity: orange circles) compared to the Si/PS samples of moderate porosity etched at constant current density (moderate porosity: blue squares) as a function of the PS layer thickness. The error bars correspond here to the measurement uncertainties of 4 kHz previously estimated samples. The resonance frequency measured for the Si plate was equal to 8.09 MHz.

Figure 8: Quality factors extracted from the real part of the acoustic admittance of the Si/PS samples with a controlled porosity (controlled porosity: orange circles) compared to the Si/PS samples of moderate porosity etched at constant current density (moderate porosity: blue squares) as a function of the PS layer thickness. The error bars correspond here to the measurement uncertainties of 11 previously estimated samples. The quality factor measured for the Si plate was equal to 190.

V. Tables

Table 1: Anodization parameters and resulting features of the characterized PS samples.

	Current density [mA/cm²]	Duration [min]	Average porosity [%]	Thickness [μm]
Low porosity	10	22	27 ± 1	25.6 ± 0.2
	10	44	31 ± 1	42.1 ± 0.2
	10	88	34 ± 1	77.6 ± 0.6
Moderate porosity	50	5,75	40 ± 3	22.8 ± 0.5
	50	11.5	43 ± 2	39.4 ± 0.7
	50	23	49 ± 3	68.7 ± 1.7
High porosity	80	5	47 ± 3	25.6 ± 0.6
	80	10	54 ± 3	43.3 ± 0.8
	80	20	62 ± 4	73.1 ± 1.7
Controlled porosity	Variable	7	50 ± 2	26.6 ± 0.4
	Variable	12.5	49 ± 2	43.3 ± 0.4
	Variable	44	44 ± 2	82.1 ± 0.4

Supporting information

INVESTIGATION OF ULTRASONIC ABSORPTION IN THE MHz FREQUENCY RANGE BY SILICON SUBSTRATES WITH A BUILT-IN POROUS SILICON LAYER

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I. SEM observations of the porous silicon layers

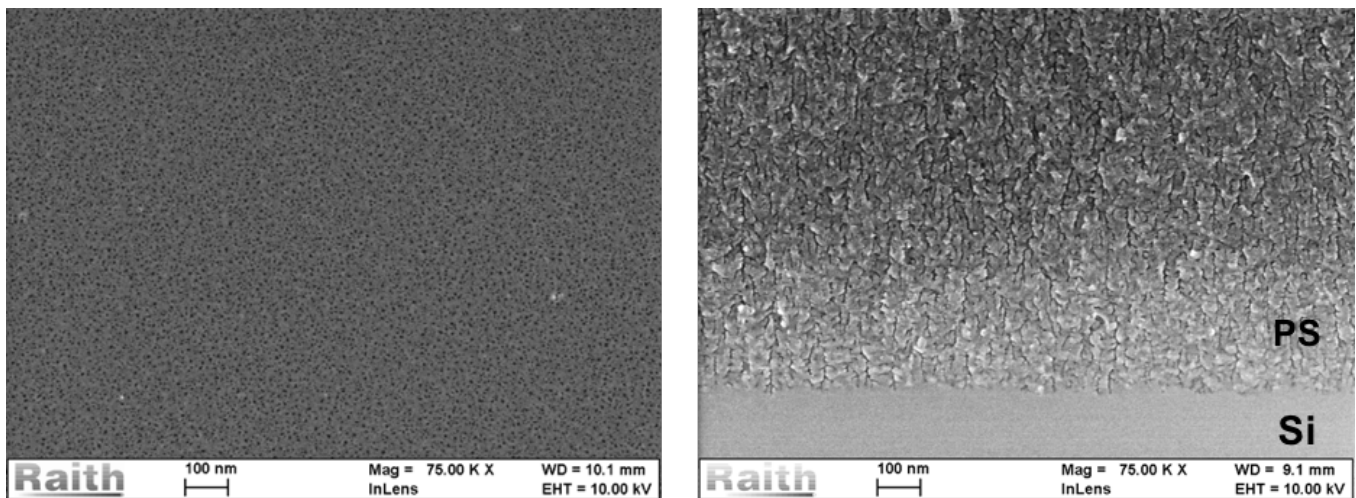


Fig. S1: SEM observations of the porous silicon layer obtained at 50 mA/cm² 11.5 min, left: top view and right: cross-sectional view.

II. Experimental setup

The measurement of the force applied between the ultrasonic transducer and the sample was performed thanks to a homemade setup as represented on the Fig. S2. The sample was held by a hollow plastic holder which was deposited onto a homemade plastic tip connected to a micro load cell (CZL635, from GOTRONIC). The load cell was electrically connected to a homemade electronic circuit including a Wheatstone bridge and an amplification stage. Finally, the output voltage amplitude was recorded using a multimeter with mV accuracy (ISOTECH-IDM103) and the applied force was determined using previously established calibration curves.

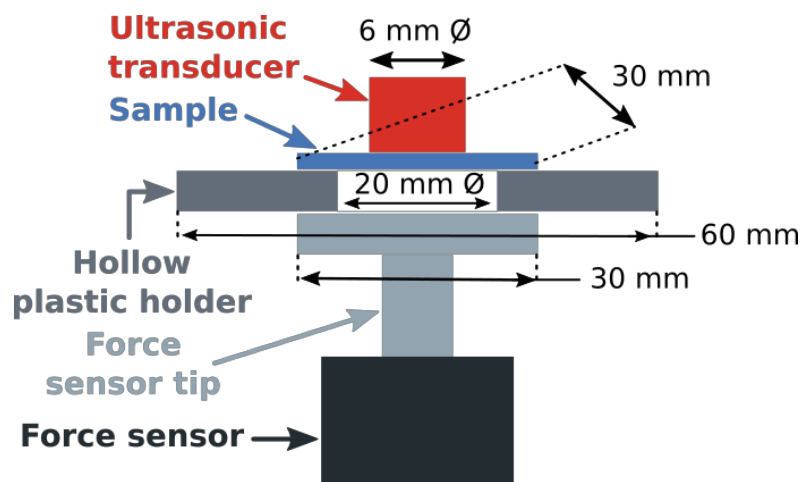


Fig. S2: Schematic representation of the sample setup on the force sensor with the relevant dimensions (unscaled drawing).

III. Estimation of the measurement uncertainties

The measurement on the Si plate was repeated five times in order to evaluate the consistency of the characterization method and to estimate the measurement uncertainties. Before each measurement, the calibration procedures were performed and the sample was moved and put again in place. The acoustic conductances deduced from the five measurements are presented in Fig. S1.

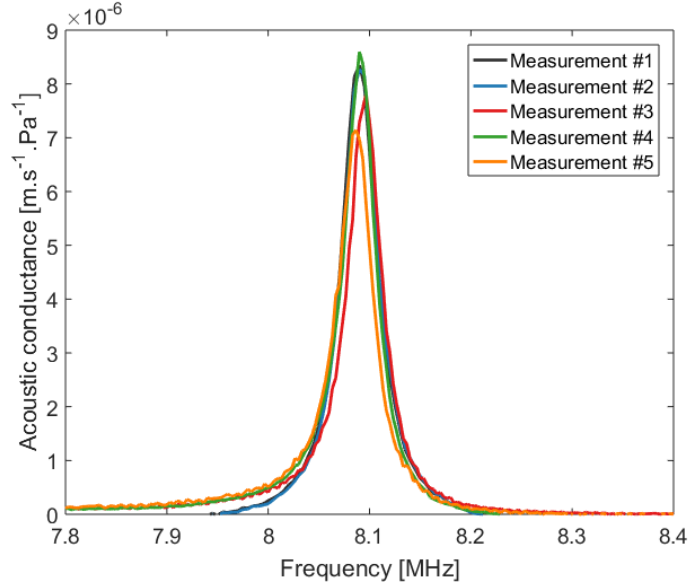


Fig. S3: Acoustic conductances deduced from the measurements of the Si reference plate.

The resonance frequencies and quality factors extracted from the resonance peak of the Si plate are detailed in the Table S1. These values were used to deduce the measurement uncertainties following the recommendations described in [ref_S1]. The distribution of the measured resonance frequencies and quality factors were supposed to follow a normal distribution. As a consequence, the standard uncertainties in the term defined in [ref_S1] were calculated by dividing the standard deviation by the root mean square of the number of measurements (i.e. 5). Finally, the expanded measurement uncertainties, presented in Table S1, were obtained by multiplying the standard uncertainties by a coverage factor. Here, the coverage factor was chosen to have a level of confidence equal to 99% and is equal to 2.576.

Table S1: Resonance frequencies and peak quality factor extracted from the measurements on the Si plate and the deduced measurement uncertainties.

	#1	#2	#3	#4	#5	Mean value	Standard deviation	Measurement uncertainty
Resonance frequency [MHz]	8.090	8.090	8.097	8.090	8.087	8.091	0.004	0.004
Quality factor	179.8	179.8	186.8	202.3	186.6	187.1	9.2	10.6

IV. Acoustic admittances measured on the Si/PS samples

All the real parts of the measured acoustic admittances on Si/PS samples are presented in Fig. S2 and compared to the measurement on the Si reference plate. Data are normalized to the maximum of the amplitude of the real part of the acoustic admittance of the Si plate measured prior to each measurement set.

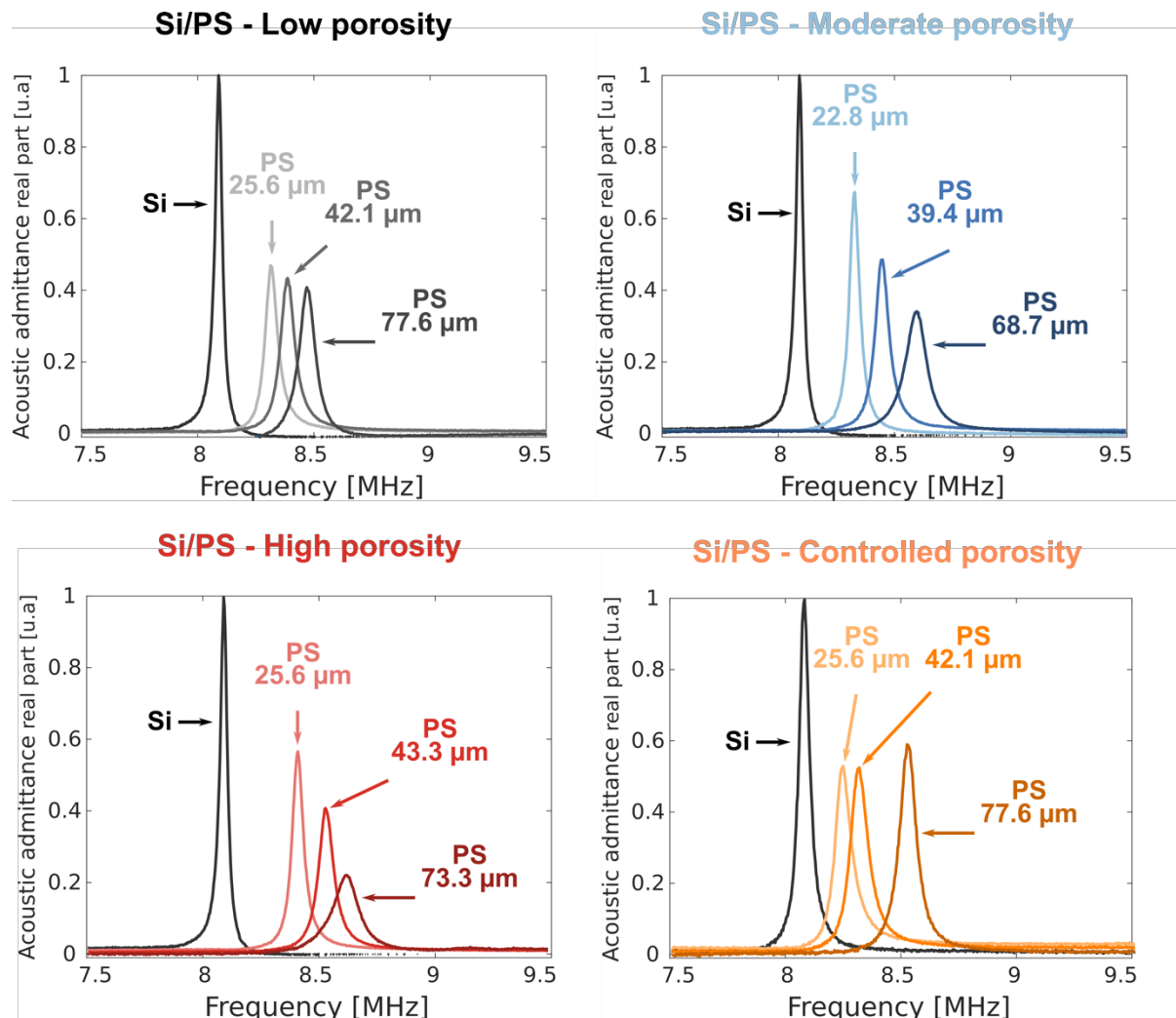


Fig. S4: Normalized real part of the acoustic admittances measured on all the Si/PS samples.

V. Theoretical evolution of the quality factor

The Fig. S5 shows the evolution of the quality factor as a function of the ratio between the PS layer thickness and the acoustic wavelength in the PS at the resonance frequency of the substrate (h/λ) for two porosity values of 30 % and 60 %. The thickness of the PS layer with a porosity value of 60 % was varied between 20 μm and 80 μm , to be in agreement with the experimental data; the thickness of the PS layer with a porosity value of 30 % was varied between 20 μm and 120 μm for a better comparison. The wavelength values were calculated at the resonance frequency of the bilayer; considering the variation of the longitudinal velocity in the PS as a function of the porosity value as defined in Eq. (9).

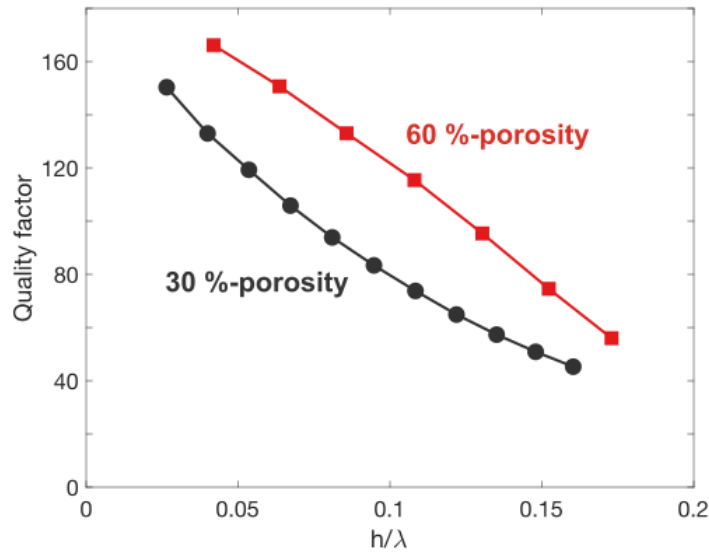
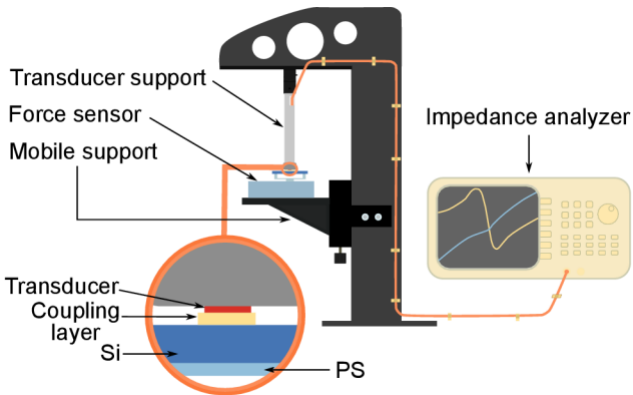


Fig. S5: Evolution of the quality factor at the substrate resonance as a function of the h/λ ratio, for two porosity values of 30 % (black circles) and 60 % (red squares).

References:

[Ref_S1] BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML. Guide to the Expression of Uncertainty in Measurement. International Organization for Standardization, Geneva. ISBN 92-67-10188-9, First Edition 1993, corrected and reprinted 1995.



Si/PS - Moderate porosity

