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SU-8 photoresist and SU-8 based nanocomposites for broadband acoustical matching at 1 GHz

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Abstract. So as to integrate acoustic functions in BioMEMS using 1 GHz ZnO transducers deposited on silicon substrates, acoustic waves propagation through the silicon substrate and its transmission in water needs to be maximized (the insertion losses at the Si / water interface are about $6dB$).

In the context of integration, it is interesting for mechanical impedance matching to use photosensitive materials such as SU-8 so that patterns may be obtained. Nanocomposite materials based on SU-8 mixed with nanoparticles having adequate impedances were fabricated. These new materials are characterized in terms of their acoustic velocity, impedance and attenuation. For this, the nanocomposite layers are deposited on the substrate by spin coating to obtain a thickness of about 10 μm, in order to separate acoustic echoes from the material (even if $\lambda/4$ layer thickness is lower than 1μm).

The insertion losses of the device immersed in water can be simulated as a function of frequency for a given reflection coefficient between the silicon substrate and the photoresist. The characteristics of some nanocomposites made with SU-8 and various concentrations of nanoparticles like TiO_2, $SrTiO_3$ or W have been determined.

1. Introduction

Recent advances in biology, and more specifically biological detection, have revealed the need to work with higher resolutions on a micrometer scale. This implies an increase in the frequency range used, that is to say in the order of gigahertz [1-2]. This frequency range can be obtained using a ZnO piezo-electric transducer. However, as it can be considered that the cell's environment is mainly water, much of the energy of the acoustic waves emitted is reflected at the Si / water interface due to the huge impedance mismatch between these two materials. In addition, the attenuation in water is high (about 220dB/mm/GHz^2); the acoustic waves will thus have difficulty to reach the cells.

The aim of our study is to optimize the energy transfer between the silicon substrate and water, which implies good mechanical matching between these two materials. The standard solution is to match the impedance using one or more quarter wavelength transmission lines with specific acoustic impedance. It is well known that the more segments, the higher the bandwidth. As materials having the required impedance are generally not available we must create them [3 for example] and doing that, try also to minimize their absorption. The way we chose is to mix nanosized particles in SU-8. SU-8 is an epoxy-based negative photoresist that can be patterned for high ratio structures [4]. So it makes it possible to use it for Lab-On-Chip fabrication [5-6].

Theory of multi section quarter wavelength transformer [7] is used to compute the required values of acoustical impedance optimizing the matching of water to silicon. Mechanical impedances to use are about 5.5 MRayls for one section quarter wavelength transformer, 8.8 MRayls and 3.4 MRayls for a two section quarter wavelength transformer, 12.56 MRayls, 5.48 MRayls and 2.39 MRayls for a three section quarter wavelength transformer, and so on. The two sections quarter wavelength transformer can be easily realized by glass and polymers deposition.

In addition to pure SU-8, W, TiO_2 and $SrTiO_3$ nanoparticles are used to fabricate different composites with adjustable acoustic impedances. The fabrication process and characterization of high frequency ultrasound propagation in these materials are presented in this paper. We check that the photoresist property of the SU-8 resin remain in the low concentration mixture we use. The phase velocity and the attenuation coefficients of the acoustic waves are also measured.

2. Material fabrication and characterization

2.1. Matching layer fabrication

The fabrication of the 1GHz transducers has been presented previously [8]. The SU-8 2000 photoresist and TiO_2 nanosize particles for example (TiO_2: 35 nm diameter, *Degussa Corporation*; $SrTiO_3$: 50nm diameter, *Advanced Materials*) were ball-milled (*Retsch* PM100 planetary ball miller) in an agate jar in order to obtain an homogeneous nanocomposite. SU-8 2000 thinner (cyclopentanone, *Microchem Corporation*) was used as a solvent during ball milling. The mixture was deposited on the back of the silicon substrate by spin coating to obtain a thickness of about 10 µm. The substrate was then placed on a hot plate to evaporate the solvent. The thickness and surface roughness of the matching layer was characterized as less than 100 nm using a *Tencor Alpha step* profiler. Figure 1 shows a Scanning Electron Microscopy (SEM, *Zeiss* U55) surface image of a TiO_2/SU-8 nanocomposite and a cross section image of a W/SU-8 nanocomposite. It was revealed that the nanoparticles were homogenously distributed in the composite.

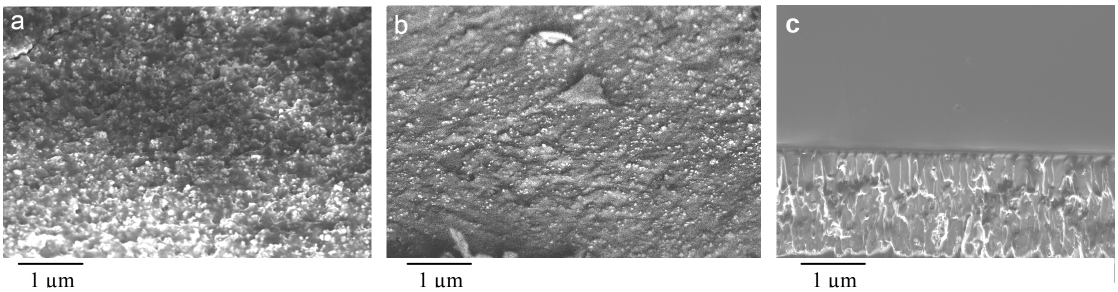

Figure 1. SEM micrograph of a TiO2 / SU-8 composite and a W/SU-8 composite;
(a) secondary electron image for TiO2/SU-8
(b) backscattered electron image for TiO2/SU-8
(c) Cross section of W/SU-8 nanocomposite.

2.2. Lithography of the nanocomposites

The lithographic efficiency of SU-8-based nanocomposites was studied. The lithography process is as follows: spin-coating (*Suss Microtech Spin Coater*, speed: 3000 rpm; time: 20 seconds) of the nanocomposite layer, then pre-baking at 95 °C for 4 minutes. The sample is then exposed to 365 nm UV for 20 seconds at 13 mW/cm^2, post-baking at 95 °C for 4 minutes. The development is made in SU-8 developer for 30 seconds. Isopropyl alcohol (IPA) was used to rinse the developed samples. Figure 2 shows the photo-defined pattern of an SU-8-based nanocomposite measured by FIB (*FEI Strata* Focus Ion Beam). As a consequence, the nanocomposites can be used in micro fabrications as SU-8 and also for the integration of acoustical characterization in lab-on-chip technology. These results show the possibility to use the nanocomposites also for high frequency piezocomposite for which it is necessary to adjust the mechanical impedance of the non piezoelectric material.

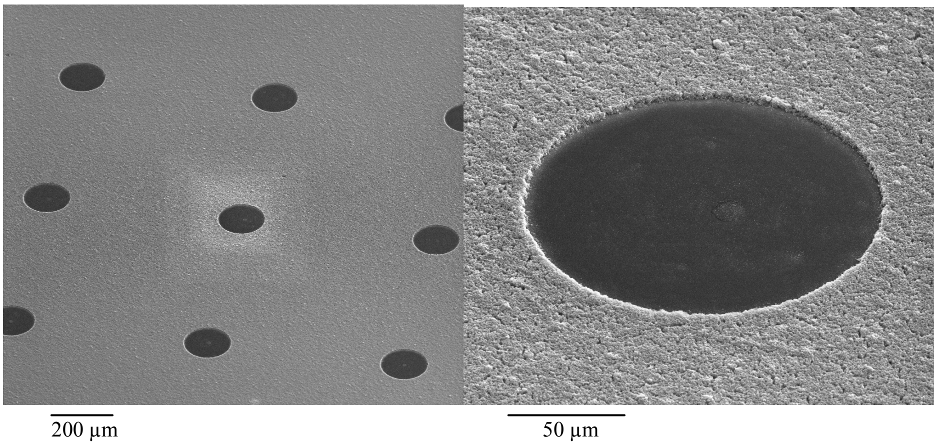

Figure 2. FIB images of the lithography of an SU-8-based nanocomposite. The holes patterned in the composite has a diameter of 150 µm

2.3. Determination of acoustic properties

To measure the acoustic properties of the materials, a broadband (300 kHz-8 GHz) vector network analyzer (*Rohde Schwarz,* ZVA 8) was used with a prober system (*Suss Microtech* EP4), as shown in figure 3.

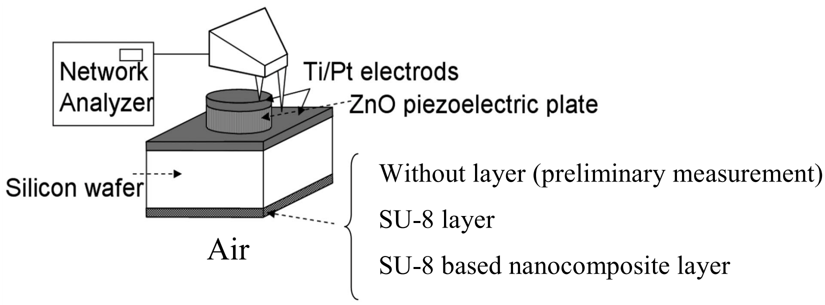

Figure 3. Scheme of the device under test
The S_{11} parameter measurement is obtained thanks to a prober system connected to a VNA

This measurement uses the parameter S_{11}, which is the ratio between the reflected and the incident electrical wave. In figure 4, the real and imaginary parts of S_{11} are plotted versus the frequency. This signal is extracted directly from the vector network analyzer. The real and imaginary parts of S_{11} show oscillations superimposed on slow variations. Oscillations are produced by the propagation of acoustic waves reflected on both sides of the silicon wafer and slow variations from the electrical behavior of the transducer. It has already been shown that [9]:

$$S_{11}(f) = S_{11}^{el}(f) + K\, S_{11}^{ac}(f) \tag{2}$$

Where:

K is the electro-acousto-electric conversion coefficient of the transducer.

S_{11}^{ac} is the sum of the acoustic waves that are formed from all the possible reflections before returning to the piezo-electric plate. This parameter can be used to estimate the acoustic characteristics of the materials.

S_{11}^{el} is the electrical reflection on the transducer. It allows the electrical impedance of the transducer to be measured. A Tchebychev type II high pass filter is applied in order to isolate the acoustical response (oscillations) from the electrical response (slow variations).

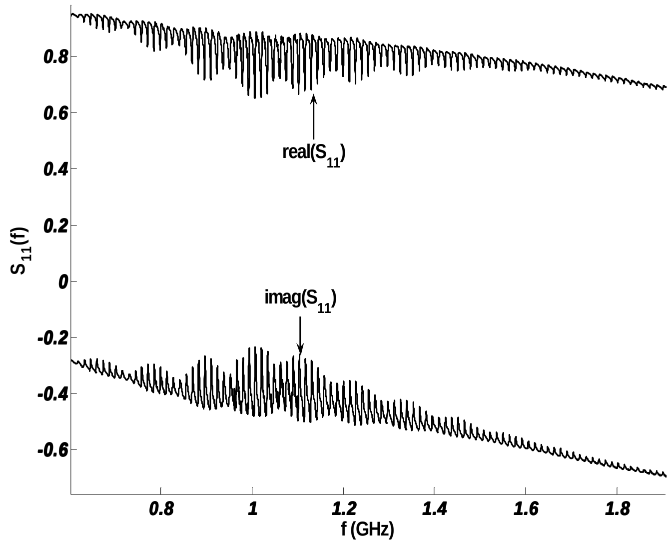

Figure 4. Real and imaginary parts of the measured S_{11} parameter versus frequency for the preliminary test (Transducer / Silicon / Air).

The impulse response of the system $S_{11}(t)$ is obtained by computing the inverse Fourier transform [10] of the $S_{11}(f)$ parameter. It is formed by a set of peaks centered at the delay time of the arrival of waves. A first measurement without layer is needed to characterize the electro-acousto-electric conversion coefficient of the transducer. Then an other measurement of the S_{11} parameter of the system for which the thin layer under test has been spin coated is performed. The comparison of the first echoes allows to evaluate reflection coefficient at the interface silicon / layer under test, the other echoes allow to evaluate the attenuation coefficient and the velocity of the ultrasonic wave propagating in the layer under test.

Figure 5 shows the impulse responses of signals associated with the silicon / air (preliminary test) and the silicon / SU-8 / air configurations. The comparison of the two curves shows that the first impulse decreases when the reflection coefficient at the Si/SU-8 interface is lower than that at the Si/air interface. It is obvious as a part of the acoustic energy is transmitted to the layer. Other impulses come from the reflection of ultrasonic waves on both of the SU-8 layer. As they are delayed and absorbed by each travel in the sample, they can be used as follows to characterize the different materials.

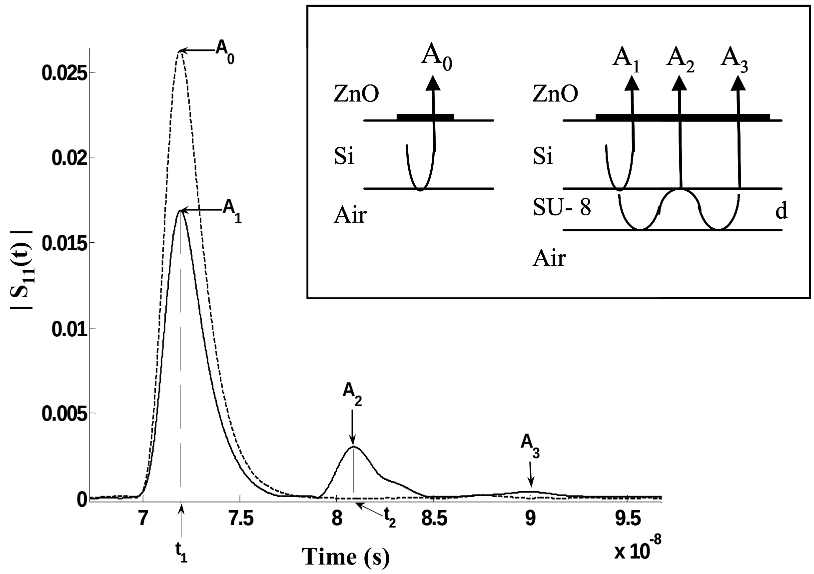

Figure 5. Impulse responses computed from S_{11} parameter versus time.
Dashed line : without layer (preliminary test)
Solid line : about 10 µm thin SU-8 layer
Insert : waves travels in the systems

The phase velocity in the material is given by:

$$V_a = \frac{2*d}{t_2 - t_1} \tag{3}$$

To calculate the impedance and attenuation, we proceed as follows:

The reflection coefficient of the Si / material interface is given by: $r = \frac{A_1}{A_0}$,

Amplitudes A_0 and A_1 correspond to the maximum amplitude of the reflected acoustic signal at the Si / Air interface and the maximum amplitude of the reflected acoustic signal at the Si / Material interface respectively. A_2 and A_3 correspond to the maximum amplitude of the reflected acoustic signal at the Si / material interface after respectively 2 or 3 reflections in the silicon.

The reflection coefficient r is also defined by: $r = \frac{Z_a - Z_{Si}}{Z_a + Z_{Si}}$ **(4)**

This thus gives the material's acoustic impedance:

$$Z_a = \frac{1+r}{1-r} * Z_{Si} \tag{5}$$

The attenuation in the material can be estimated by:

$$\alpha = -\frac{1}{2*d} * \log\left(\frac{A_3}{r*A_2}\right) \tag{6}$$

We use the same method to characterize all the other samples.

3. Results and Discussion

3.1. Acoustical characteristics of the materials

Nanocomposites made from SU-8 and W (or TiO_2 or $SrTiO_3$) nanoparticles have been characterized. The results are presented in Table 1. The concentration of SU-8 in the nanocomposite as shown in table

1 is the concentration of SU-8 with its thinner (SU-8 2000 thinner evaporates during ball milling) and not its "dry" concentration.

Table 1. Acoustic characteristics of nanocomposites at 1 GHz

Acoustic characteristics	SU-8	SU-8 90% TiO_2 10%	SU-8 80% TiO_2 20%	SU-8 95% W 5%	SU-8 80% $SrTiO_3$ 20%	SU-8 70% TiO_2 30%	SU-8 70% TiO_2 15% $SrTiO_3$ 15%	SU-8 70% $SrTiO_3$ 30%
Impedance (MRayls)	3.5	4.5	5	5.3	5.5	6	6.5	7
Phase velocity (m/s)	2890	2490	2710	2700	2640	2600	2500	2370
Attenuation (dB/µm)	0.3	0.5	0.6	0.4	0.3	0.5	0.6	0.7

The impedance of pure SU-8 is lower than the impedance of SU-8 mixed with a nanocomposite. In the case of a low concentration mixture, the phase velocity remains almost constant, so the impedance is governed by the increase in density. It was found that the concentration of particles in the mixture could not exceed 30% for TiO_2 and $SrTiO_3$ and 15% for W as the solution becomes inhomogeneous and therefore inoperable. So, an acoustic impedance higher than 7 MRayls could not be obtained and this technique is limited to a one section quarter wavelength transformer. We will therefore compare the efficiency of the impedance matching of one nanocomposite layer with the two section quarter wavelength SiO_2/SU-8/H2O commonly used and characterized in a previous study [8].

3.2. Acoustical matching

After obtaining the experimental results of the acoustic characteristics of the materials, the insertion loss at the interface between the different quarter wavelength matching layers and water as a function of frequency were determined as shown in figure 6. The insertion loss at the interface between silicon and water is close to 6 dB. When SU-8-based nanocomposites were introduced as matching layers, the insertion loss greatly decreased. The insertion loss is given by:

$$L = 10\log_{10}\left[1-\left|r\left(f\right)\right|^2\right] \quad \textbf{(7)}$$

r(f) is computed by filtering the oscillation of S_{11}(f) so as to conserve only these corresponding to the first arrival of the impulse response [8].

In figure 6, an example of two section acoustical layer matching is presented. A quarter wavelength SU-8 layer and a quarter wavelength SiO_2 layer are stacked. The experimental and theoretical results, based on transfer matrix calculation were compared. A simulation with only one nanocomposite layer is also presented. The bandwidth obtained is greatly improved compared to a single quarter wavelength layer.

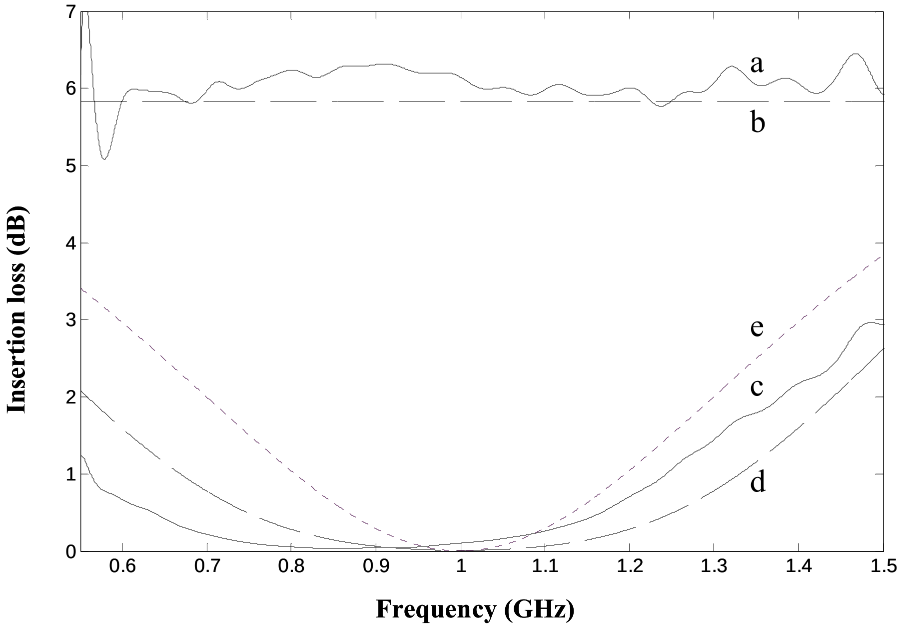

Figure 6. Insertion loss (dB) versus frequency (GHz) for:
the Si/water interface ((a) measured, (b) theoretical)
the Si/SU-8/SiO_2/water interface ((c) measured, (d) theoretical)
the Si/SU-8 $_{80\%}$ $SrTiO_{3\ 20\%}$/water interface (e) theoretical)

The minimum is centered around 0.9 GHz. This difference can be explained by an imprecision in the thickness the spin coating of the layer and by variation in the phase velocity in the layer, which is strongly dependent on the fabrication process.

4. Conclusion

This study has shown that new materials could be created. These materials have been characterized and we have presented their acoustic properties (velocity, attenuation coefficient and acoustic impedance). The acoustic impedance can be easily adjusted between 3 MRayls and 7 MRayls. The obtained materials remain photosensitive and so, can be patterned and have potential applications for the integration of acoustical characterization in lab-on-chip technology. The range of acoustic impedance we can get is not high enough for making a two section impedance transformer using only polymers. So we realize and characterize the two sections efficient impedance matching (Si / SiO2 / SU-8 / H2O) as shown on curves of the figure 6. The attenuations of ultrasonic 1GHz longitudinal waves in these new materials remain weak. So, used as matching layer, they can increase energy transmitted from silicon to water of about 5dB.

5. Acknowledgments

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6. References

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