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Acoustical properties characterization of a composite made of SU-8 and nanoparticules for BioMEMS application

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Abstract

The photoresist SU8 often used in microtechnology has been acoustically characterized at the frequency of 1 GHz thanks to ZnO transducers. This material will be used to achieve acoustical matching between silicon and water at this frequency. The acoustical characterization of a nanocomposite material made of this photoresist SU8 doped with TiO_2 nanoparticles is also presented. The aim is to achieve matching layers with specific mechanical impedance around 5 MRayls and depending on the nanoparticles concentration. The targeted application concerns the integration of high frequency ultrasonic transducer in Lab-on-Chip for biological cell characterization. The mechanical impedance and the attenuation of this composite material are characterized around 1 GHz thanks to a standing wave ratio method measuring S_{11} parameter and extracting the targeted parameters thanks to signal processing.

1 Introduction

Acoustic microscopy/imaging has been a suitable tool for imaging or mechanical characterization of biological materials. The recent trend toward biological detecting demands higher resolution at the scale of micrometer pushing the operating frequency of acoustic devices up to the gigahertz range [1,2]. These high frequency acoustic waves can be enhanced using thin piezoelectric layers deposited on a substrate and this is a nice way to make some high frequency acoustics functions in BioMEMS (from simple detection to imaging of cell). Nevertheless, regarding the longitudinal acoustic wave attenuation in high frequency range (220 $dB/mm/GHz^2$ in water), quarter-wavelength acoustic matching layers between silicon, whose acoustic impedance is usually ~20 MRayls, and water, whose acoustic impedance is ~1.5 MRayls are necessary to enhance transmitted power.

Because single-phase materials that meet the need of such acoustic impedance do not exist in nature, most of the matching layers are made of composite materials. By mixing the high acoustic impedance powder with low acoustic impedance polymer, the acoustic impedance of the composites can be tailored by varying the volume fractions of the components to satisfy the requirements of ultrasonic transducers [3-5]. Nanocomposite matching layers of thickness about 10µm have been made for transducers (~ 50 MHz) by mixing nanosized silicon oxide/alumina and polymer [6-8].

Sizes of particles have to be very small compared to the wavelength. In such a case, for low concentration, particles increase density of the material with weak change in its stiffness. So acoustical impedance increases, velocity decreases and absorption remains acceptable for thin layers. For a 1 GHz transducer made of ZnO piezoelectric thin films, thickness of matching layers needed between substrate and propagating medium is less than 1 µm and size of particles is in order of a ten of nanometers. It is a challenge to fabricate such proper matching layers with homogenous properties for such GHz transducers by conventional mixing process method.

In this paper, we report on mechanical matching efficiency between silicon and water with SU8-based matching layers. The interest of SU-8 is that it is a negative photoresist that can also be used for high ratio structure [9] that can be integrated in Lab-on-Chip devoted to high frequency acoustical microscopy. Center frequency of the transducers is ~1 GHz. Acoustic properties of these SU8-based matching layers are also studied.

2 Material and method

2.1 Matching layer fabrication

The fabrication process is as follows. 10 nm Titanium layer and 100 nm Platinium layer were deposited by sputtering as the bottom electrode on n-type (100) silicon wafer successively. Then, 3.5 µm thick PMGI SF19 (*Microchem Corperation*) resist layer was spin coated on the bottom electrode. Patterns with diameter about 150µm in PMGI layer were obtained thanks to commercial MF 319 developer (*Microposit*) through a mask made of S1818 resist (*Shipley Corporation*). After that, 2.5µm thick ZnO thin film and 100 nm thick Platinium top electrodes were deposited on the patterned wafer. Finally, transducers were achieved through a lift-off process.

The well-known epoxy-based SU8 2000 resist (*Microchem Corperation*) and nanosized TiO_2 (~35 nm, *Degussa Corperation*) were ball-milled (Retsch PM100) with an agate jar in order to obtain homogeneous mixture. SU8 2000 thinner (*Microchem Corperation*) was used as solvent during ball milling. The mixture was deposited on the back side of the silicon substrate by spin coating so as to obtain a thickness of about 10 µm (in order to separate acoustical echoes, so as to get the speed and the attenuation of the acoustic waves in this material). The substrate was then placed on a hot plate to evaporate the solvent.

The surface roughness of the matching layer was characterized to be less than 100 nm by using a Tencor Alpha step profiler. Figure 1 shows the cross-section scanning electron micrograph of the matching layer with 30 wt% TiO_2. It revealed that nanosized TiO_2 particles were homogenously distributed in the composite.

2.2 Acoustic properties determination method

Electro-acoustic measurements are performed thanks to S_{11} scattering parameter using a Suss Microtech prober coupled with a Hewlett Packard 8753 Vector Network Analyzer (Figure 2). S_{11} parameter is the reflected to incident electrical waves ratio. It is simply linked with the electrical impedance of the transducer by $S_{11} = \frac{Z - Z_0}{Z + Z_0}$. We have already described this electrical impedance in a previous work [10] which emphasizes the fact that the electrical impedance can be split in two parts: a pure electrical part re presenting the reflected electrical wave upon the piezoelectric plate, and an electro-acoustic part representing acoustic wave propagations inside the wafer. This latest part contains acoustical information on all mediums in which acoustic waves propagate. The same is for S_{11} parameter which can be wrote as:

$$S_{11} = S_{11}^{el} + K\, S_{11}^{ac} \tag{1}$$

with K the electro-acousto-electric conversion coefficient of the transducer and S_{11}^{ac} the sum of acoustic waves traveling along all possible paths before returning to the piezoelectric plate.

In the case of normal incidence layer S_{11} parameter can be represented as:

$$S_{11} = S_{11}^{el} + K\, D_1\, e^{-2i\theta_0} + K\, D_1\, r_{0p}\, e^{-4i\theta_0} + \ldots \tag{2}$$

with labels 0 for the silicon wafer and p for the piezoelectric plate. D_1 describes the effect of waves reflected at each interface (Figure 2) of the medium labeled '1'. It is useful to express it with a Debye-series expansion.

$$D_1 = r_{01} + \left(1 - r_{01}^{2}\right) r_{12} e^{-2i\theta_1} + \ldots + \left(1 - r_{01}^{2}\right) r_{10}^{N-1} r_{12}^{N} e^{-2Ni\theta_1} + \ldots \tag{3}$$

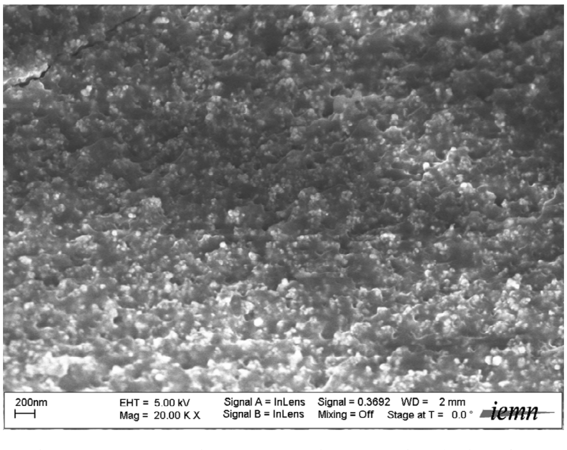

Fig 1. Cross-section SEM micrograph of TiO_2 / SU8 nanocomposite films.

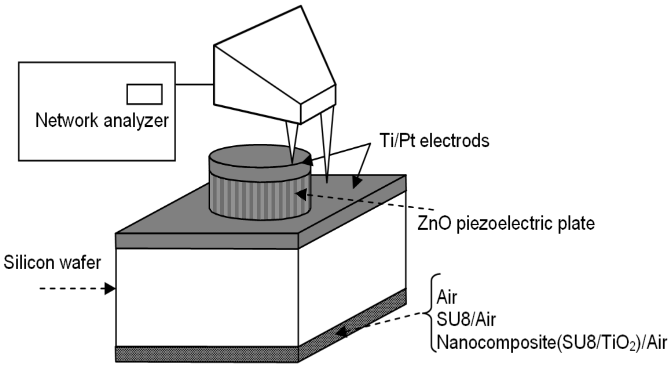

Fig 2. Scheme of the device under test

with $r_{ij} = \frac{Z_j - Z_i}{Z_j + Z_i} = -r_{ji}$ the reflection coefficient of the wave propagating from medium labeled 'i' to medium labeled 'j', $\theta_i = k_i\, e_i$ the phase of the wave propagating through a thickness e_i of the medium i, $k_i = \frac{\omega}{v_i} - i\,\alpha_i$ the wave number of this wave, v_i and α_i its phase velocity and its coefficient of absorption, $i^2 = -1$.Measurement are done with air as material 2 so $r_{12} = -1$.

In the frequency domain in which the measurement is performed, the reflected waves on multiple interfaces interfere making their analysis difficult. Nevertheless the time domain representation of the S_{11} parameter computed via an inverse Fourier transform algorithm presents a pure electrical impulse at null time since network analyzer is calibrated in the plan of ZnO plate and acoustic impulses delayed at the time of arrival of acoustic waves. They can overlap or not according to the length of the path of propagation and to the available bandwidth. Thickness of the silicon wafer being very large compared to the wavelength, we can spin coat a thicker layer of SU8 to prevent overlap of the waves traveling insides it and making so the acoustical characterization easier.

Characterization of materials is processed in time domain using ultrasonic pulse-echo technique [11] obtained by inverse Fourier transform. Figure 3 represents an example of time domain representation of S_{11} parameter. Time spacing between impulses labeled 'i' or 'j' allow us to evaluate phase velocity:

$$v_1 = \frac{2(j-i)e_1}{t_j - t_i} \tag{5}$$

The inset shows the description of multiple acoustic paths)

We compute coefficient of absorption using level of impulses

$$\alpha_1 = -\frac{\log_{10}\left(\frac{1}{r_{01}^{\;j-i}}\frac{A_j}{A_i}\right)}{2(j-i)e_1};\; i \neq 0 \tag{6}$$

With A_j the amplitude of the pulse j, and $r_{01} = \dfrac{A_0}{K}$ from which one can compute impedance of material 1: $Z_1 = Z_0 \dfrac{1 + r_{01}}{1 - r_{01}}$ then its density, $\rho_1 = \dfrac{Z_1}{v_1}$.

3 Results and discussion

The acoustic characteristics of pure SU8 (epoxy based resin) are close to else of epoxy (2860 m/s, 1190 kg/m^3) [12]. Our measurement method give acceptable values (2880 m/s, 1142 kg/m^3) and give in addition the attenuation coefficient of the material (0,33 dB/µm at 1 GHz).

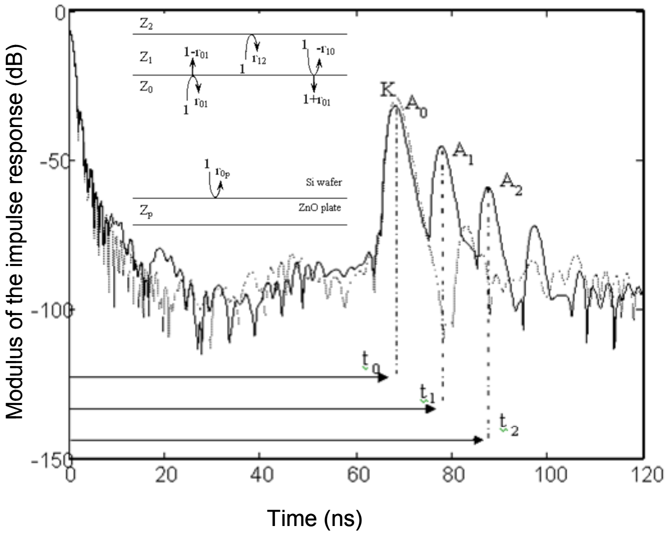

Fig.3. Impulse response modulus of the 1GHz resonant frequency transducer (Doted line: reference transducer, ZnO/Silicon/Air; Solid line: ZnO/Silicon/15 µm SU8/Air

The characterization of the nanocomposite with 30 wt% TiO_2 and 70 wt% SU8 shows an acoustic impedance of 5.5 MRayls which is the target for the matching layer.

The longitudinal acoustic wave speed in the composite is 2600 m/s (compared to 2880 m/s in pure SU8) and the attenuation in the composite is 0,53 dB/µm (compared to 0,33 dB/µm in the SU8). As we could predict TiO_2 particles increase density of the composite with weak change of stiffness but they increase attenuation. Nevertheless thickness of the quarter wavelength nanocomposite layer being smaller than the one of pure SU8, global effect is positive. As we can see in Figure 4, the transmitted power at the interface Silicon / water will be enhanced using these materials:

~ 6dB are loss at the interface Silicon/water;

~ 2 dB (~1.5 dB reflection and ~0.3 dB absorption) at 1GHz using 720 nm of SU8 as quarter wavelength matching layer;

~ 0.5 dB (~0 dB reflection and ~0.5 dB absorption) at 1GHz using 650 nm of SU8-TiO_2 nanocomposite as quarter wavelength matching layer.

4 Conclusion

We have described the fabrication and characterization of a nanocomposite made of TiO_2 nanoparticles and SU8 for 1 GHz mechanical matching layer conception between water and silicon. Longitudinal acoustic wave speed and attenuation have been measured. We have shown the interest of a composite based on SU8 compared to pure SU8. In fact, a good matching (the gain is about 5 dB) between silicon and water has been obtained with one photoresist layer.

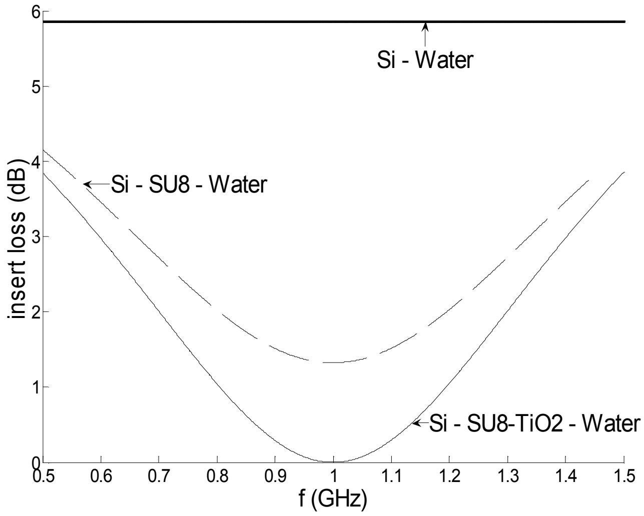

Fig 4. Simulation of the insertion loss vs frequency for SU8 and nanocomposite (SU8/TiO_2) as λ/4 matching layer between silicon and water

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