ULTRASONIC RESONATING SENSOR AND NEAR-FIELD INTERACTION : FROM RHEOLOGY TO MICROSCOPY.

JY. Ferrandis, G. Levèque, and J. Attal
Laboratoire d'Analyse des Interfaces et de Nanophysique, LAIN, UMR CNRS 5011, Université Montpellier 2, CC 082, Pl. E. Bataillon, F-34095 Montpellier Cedex
ferrandi@lain.univ-montp2.fr

Abstract
Horn shape ultrasonic sensors can be an alternative service with respect to acoustic integrated units in AFM-like microscope. When the tip or the horn is dived into the medium to be tested, the sensor generates propagating and standing waves and gives information on the viscoelastic properties of the paste. If the sensor is laid on the sample, information are related to local elastic properties. It is shown that the near-field interaction between the tip and the medium depends on the viscoelastic properties of the sample.

Introduction
Horn shape ultrasonic sensors can be an alternative service with respect to acoustic integrated units in AFM-like microscope. In contrast to most other sensors, the horn-like one is self-consistent and does not require any other kind of detection, the piezoelectric elements serves as both input and output electrical port. The main applications discussed here concern submillimetric to micrometric scales so modelling will be done in that range with operating frequencies of about 20 to 300 kHz.

Resonating Horns
The principle of the device is simple: the PZT excitation produces longitudinal oscillations of a resonating sensor composed of two cylinders .

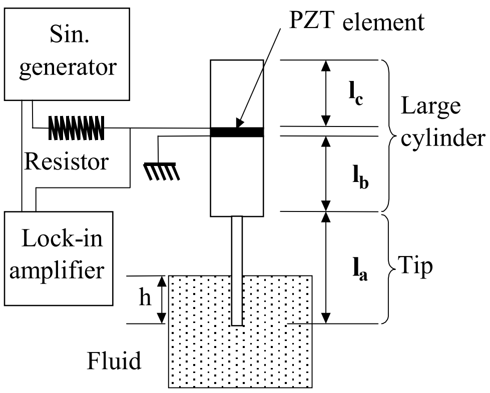

Figure 1 : Geometry of the sensor. The electric circuit is composed of a sinusoidal wave generator and a Lock In Amplifier

The large cylinder, which is free in the upper side and coupled to a much more lighter resonator (the tip at the lower side) oscillates approximately to a λ/2 mode. On the other hand, the tip which is nearly free at the lower end (the fluid action is supposed to be small) and strongly coupled to a greater mass on the other end, oscillates with a λ/4 mode. As (l_b + l_c) is roughly equal to $2l_a$, the two oscillators (the main cylinder and tip) are strongly coupled and vibrate in the same frequency range. This tuning to the two oscillators is essential in the good transfer of the acoustic load on the tip towards the electric system [1, 2]. The equation of the rod axial oscillations are well described by plane waves inside the cylinders, the fluid properties being taken into account via the boundary condition at the tip end.

The PZT element acts as a sensor for the acoustical impedances applied on its two sides (Z_1 and Z_2) and gives the measured electric impedance [3] :

$$Z_{elec} = \frac{1}{i\omega C_0}\left[1 + K^2 \frac{Z_c}{\beta l} \frac{i(Z_1 + Z_2)\sin\beta l - 2Z_c(1-\cos\beta l)}{(Z_c^2 + Z_1 Z_2)\sin\beta l - i(Z_1+Z_2)Z_c\cos\beta l}\right] \quad (1)$$

with C_0 the clamped capacitance of the PZT, K the piezoelectric coupling constant, β the wave vector, Z_c the characteristic impedance of the ceramic and l the length of the PZT .

The sensor uses the alterations in the resonance state of an acoustic horn which is in contact with the medium to be tested. The input signal is a frequency sinusoidal signal that excites extensional acoustic waves in the horn. The amplitude of the vibration at the end of the tip is typically 100 nm. The medium interacts with the sensor tip, loads the horn mechanically, and hence modifies the resonance. The response of the acoustic sensor is its impedance variation, measured with a lock'in amplifier. Two characteristics are detected by a frequency scan: the variation of resonance frequency (f') and the half width (f'') of the resonance curve (figure 2). The complex resonance frequency (f=f'+if'') is the pole of the Z_{elec}(f) function.

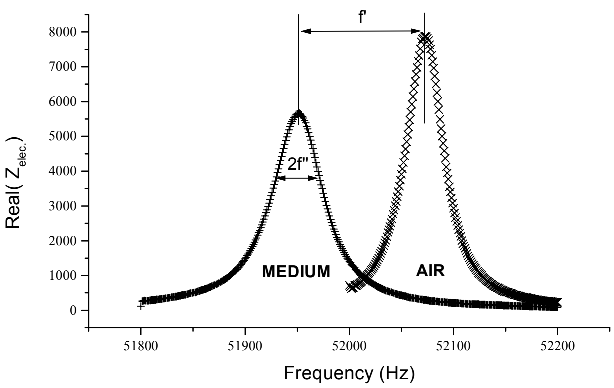

Figure 2 : The frequency shift of the resonance f ' and the half width of the resonance f '' measured by applying a frequency ramp to the piezoelectric element.

Among the numerous possible shapes, step horns were chosen consisting of two metallic rods of different diameters, the smallest one is the tip which focuses the elastic field down to the extremity.

The sensitivity of these horns is very attractive with respect to the load. Complete puts into evidence two peaks of resonance close to each other. Each peak are sensitive to a given range of elastic and viscoelastic properties of the load. So, it is a real two ways sensors with overlapping ranges. This is very convenient to monitor material that properties change with time (glue, paint, cement …).

Characterisation of materials

Two kinds of waves are emitted by the sensor. For a viscous fluid and for a sinusoidal excitation the equation governing the propagation of waves are given in term of longitudinal (compression) and transverse (shear) waves, satisfying:

$$\begin{cases} \Delta\vec{u}_L + k_L^2\vec{u}_L = 0 \quad with \quad k_L = \dfrac{\omega}{c_L} \\ \Delta\vec{u}_T + k_T^2\vec{u}_T = 0 \quad with \quad k_T = \sqrt{\dfrac{\omega\rho_f}{2\mu}}(1-i) \end{cases} \quad (2)$$

Liquid

We showed [4, 5] that the sensor is sensitive to the fluid density if partly immersed. Longitudinal waves (pressure waves) are emitted in the fluid by the flat extremity of the tip, which produce a shift in the resonance frequency of the sensor, due to the mass of the moving fluid.

Z_N is the normal acoustic impedance on the flat end of the tip which surface is S_1, ratio of the mean liquid pressure $\overline{P}$ to the tip end velocity v(d). This normal impedance Z_N is then calculated according to the following expression :

$$Z_N = \frac{\overline{P}}{v(d)} = \frac{1}{S_1 v(d)}\int_0^{r_1} p(r,d)2\pi r dr \quad (3)$$

We showed [6] that the impedance Z_N is nearly imaginary, and the flow is similar to a standing wave localized near the tip end. The small real part is due to the emission of the dipolar field at great distances. This part shall disappear if the experiment is performed in a finite size vessel, as there is no energy loss for longitudinal waves propagating in an ideal fluid.

As we are in the near field domain, the *kr* terms in equations velocity and pressure fields are small relative to the unity ($kr_1 \approx 0.22$); this imply that the flow velocity field close to the tip is nearly independent of frequency.

A second consequence is that the simple law :

$$Z_n \approx 0.66\, i\omega\rho_l r_1 \quad (4)$$

gives a good approximation of the acoustic charge. This agrees with the fact that the impedance of a vibrating piston is imaginary when its size is smaller than the wavelength. The interaction between the tip and the liquid then occurs in near-field configuration. By solving the reversed problem we are then able to measure density and viscosity of liquids [6].

Viscoelastic medium

Transverse waves (shear waves) are emitted in our geometry, when the tip is dived in the medium. In viscoelastic media, the main part of the acoustic load arises from this kind of interaction.
Experimental validation is presented on cement paste which is a viscous liquid at the early stage of the hydration and becomes a solid after a few hours. The tip is dived in the paste. The sensor is sensitive to the shear modulus of the medium and serves to test the workability of the paste during the hydration (figure 3). For instance, when the cement paste is fresh (<1h) the wavelength of shear waves is much shorter than the cylinders radius and the wave expands cylindrically around the cylinder. Later, the wavelength is greater than the tip, and the cement is then oscillating in a standing wave mode.

Such complex vibrations can not be represented by simple analytical expressions and a numerical analysis is needed. A finite element calculation (axisymetric, 9 points element) gives at the frequency of the experiment (around 45 kHz), the deformation and the strain everywhere in the vessel. The mechanical impedance of the immersed part of the tip is then calculated :

$$Z_{mech} = \frac{\textit{Strain in the section of the tip A at the surface of the sample}}{\textit{Velocity of deformation in this section}} \qquad (5)$$

This impedance (a complex number) describes precisely the interaction between the cement paste and the sensor. The imaginary part of the impedance is related to the standing waves in the medium, and the real part to the energy dissipated in the medium

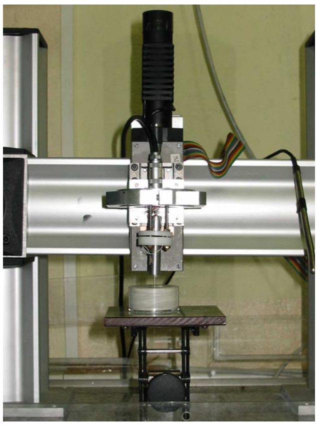

Figure 3: Experimental set-up : the tip of the sensor is dived in the analyzed medium.

In opposition to many other systems, the sensor is placed directly in the medium to be tested, without any preparation. This in-situ, non destructive test gives obviously information on the real state of the medium. With the described sensor, the range of the shear modulus G' of the medium, $10^2 < G' < 10^7$ Pa is tested.

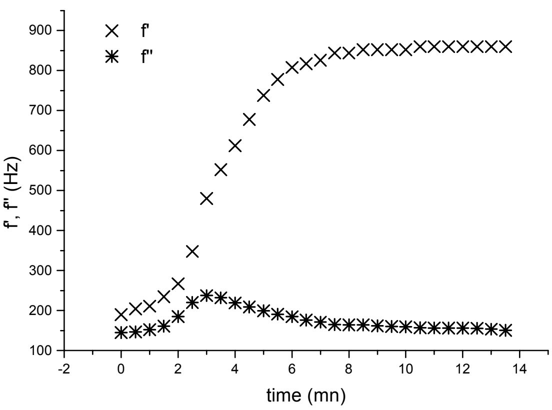

Figure 4: Experimental curves f' and f'' versus time of hydration.

According to applications required it is possible to adjust the geometry of the sensor to make it efficient the right ranges. Signal processing has also been improved to the point where it is possible to make real time data acquisitions (10 ms per point) for monitoring ultrafast changes.

Solid

When the sensor is just laid on the sample with a constant force, because the tip of the horn is much smaller than the wavelength of the acoustic waves, the interaction of the tip with the sample occurs in the near-field. The resonance frequency depends on the local elastic properties of the sample and is related to local inhomogeneities. The interaction depth directly depends on the contact surface and can be described by the Hertz theory : the ball glued at the end of the tip of the sensor is in contact with a plane (figure 5).

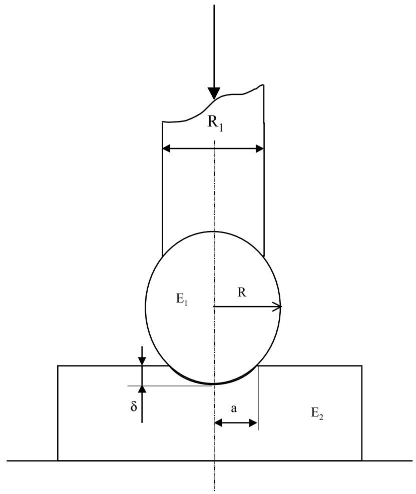

Figure 5: the ball glued at the end of the sensor is in contact with the flat surface of the sample.

δ is the penetration depth, a is the contact width, E_1 and E_2 are respectively the Young moduli of the ball and of the sample, F is the applied force on the sample.

The normal contact stiffness is [7] :

$$k = \sqrt[3]{6RE^{*2}F} \qquad (6)$$

As we have shown before, the interaction between the sample and the sensor is describe by the contact impedance :

$$Z = \frac{\sqrt[3]{6\ FRE^{*2}}}{i\pi R_1^2\ \omega} \qquad (7)$$

The sensor developed for microscopy applications is presented in figure 6.

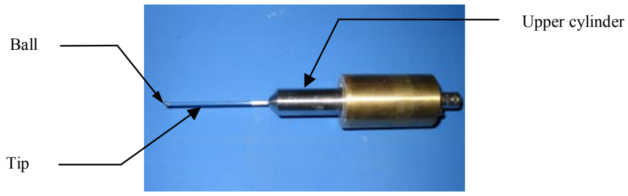

Figure 6: The sensor developed for microscopy is composed of a metal upper cylinder, a glass tip and a stainless steel ball.

Experiments were lead with different material covering a large scale of properties (from Plexiglas to gold). Different force were also applied allowing to get several measurement points of the contact stiffness and so, different values of the contact impedance. Results are presented on figure 7.

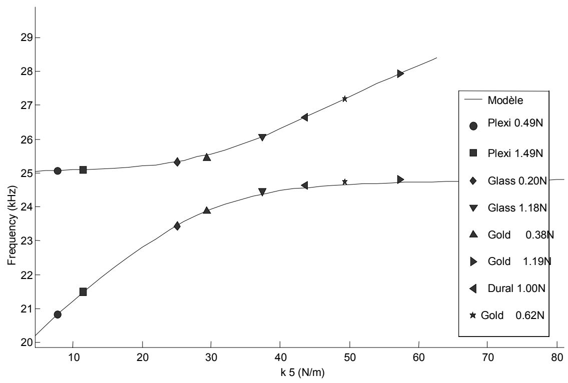

Figure 7: Resonance frequencies of the sensor versus contact stiffness for different forces and different materials.

These results show that the sensor is able to separate material as the function of the properties. Elastic properties of the sample can then be imaged with a resolution about the tip size.

Conclusion

Resonating horns are used as ultrasonic sensors in the range of about 20 to 300 kHz operating frequencies. When the sensor is dived in the medium, transverse waves are mainly involved. The wavelength then depends on the properties of the medium. A rheological characterisation is performed. When the tip is just laid on the medium, the interaction occurs in a near-field configuration with pressure waves.

The main interest of the technique is that it is a non destructive tool to characterise rheological properties of media from liquid to solid state. By changing the geometry of the sensor, a large range of properties can be investigated with a great sensitivity.

References

[1] A. Bangviwat, H. Ponnekanti and R. Finch, J. Acoust. Soc. Amer. **90**, 1223 (1991).
[2] E. Eisner, J. Acoust. Soc. Amer. **35**, 1367 (1963).
[3] G.S. Kino, *Acoustic waves : devices, imaging and analog signal processing*, (Prentice-Hall, Inc. Englewood Cliffs, New Jersey, 1987).
[4] C. Prugne, J. Van Est, B. Cros, G. Leveque and J. Attal, Meas. Sci. Tech. **9**, 1894 (1998).
[5] J. Van Est, C. Prugne, B. Cros, J. Attal and B. Cretin, Appl. Phys. A **66**, S357 (1998).
[6] G. Leveque, JY. Ferrandis, J. Van Est and B. Cros, Rev. Sci. Inst. **71**(3) (2000).
[7] K. L. Johnson, « *Contact mechanics* », Cambridge University Press, 1985.