COMPRESSIVE STRESS ON ULTRASONIC SENSORS IN DRY CONTACT FOR ELASTIC CONSTANTS MEASUREMENT AT HIGH TEMPERATURE AND DETECTION OF SURFACE PROPERTIES AT MICROSCOPIC SCALE.

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Abstract

Acoustic plane sensors adequately pressed against materials are suitable to measure their elastic constants from flight time measurement without any coupling fluid for high temperature with longitudinal and transverse acoustic waves in the megacycle range. Our sensors use a hard delay line with a mirror polished end surface. An experimental set-up is presented to perform acoustic reflection measurement in various contact conditions by increasing the applied mechanical load. The main results concern the surprising ability of this technique to detect surface properties modifications limited to thickness inferior to one micron. Tests on silicon etched by NH_4F have shown that samples with an average roughness respectively of 0.5 and 6nm were easily identified from their reflection coefficient versus load curves.

Introduction

Ultrasonic techniques enable mechanical properties measurement for bulk materials using echographic techniques. Indeed, from flight time measure, longitudinal and transverse velocities can be deduced if the sample thickness is well known. From these ultrasonic parameters and using the sample mass density, Young's modulus and Poisson's ratio can be calculated from classic linear elastic theory. Using specific signal treatments, this mechanical properties investigation is done in a non-destructive way with accuracy close to one percent. So, all these techniques need a coupling fluid to ensure ultrasonic wave propagation between the sensor and the sample to avoid the large attenuation presented by gazes. For high temperature ultrasonic measurements, specific fluids are necessary but they are often of complicate use due to their toxicity or corrosive behaviour. So, dry coupling techniques have been developed to overcome coupling fluid limitation. Hard delay lines have been also developed but they require a mechanical loading system to get the right contact conditions to enable ultrasonic wave transmission into specimen.

For this study, we have made various ultrasonic sensors working in the range of 10 to 120MHz and they generate longitudinal or transversal ultrasonic waves. Our sensors are ended by a plane hard delay line with a mirror polished end surface. In the first part of this work, we will validate our sensors and our mechanical loading system to get the adequate experimental conditions for longitudinal or transverse wave transmission through the sensor-sample interface at high temperature.

In the second part, we demonstrate experimentally the surprising ability of this probe to sense variations of surface properties, roughness, at a sub-microscopic scale despite the relatively low working frequency of our sensors in the range of 100 Hz. Surface state is analysed by Atomic Force Microscopy to provide density power spectrum (DSP) of rough surface. So, the variation of the acoustic reflectance versus pressure can be linked to the material state surface but it can be also a revealing sign of the quality of the mechanical contact or the friction conditions between the sensor and the sample. Models involving for instance the Hertz's theory will be used to deduce contact area versus applied load. A finite element model can provide reflection of an acoustic pulse versus contact area. Then we can follow theoretically reflection coefficient versus applied load for a roughness given.

Experimental set-up and method to elastic constants (E, n…) measurements up to high temperature

The first aim of these tests is to demonstrate the ability of our experimental device to provide acoustic data of the sample properties with a hard delay line pressed against sample surface without any coupling fluid from acoustic reflectance coefficient measurement (figure 1). For easier contact conditions, all the samples have been mechanically polished.

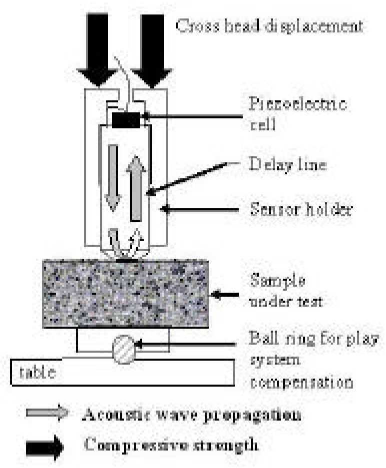

Figure 1: experimental set up to measure reflection coefficient on a compressed sample.

During these tests, the front wall echo and the back wall one are recorded to study wave propagation conditions. Figure 2 represents typical echograms or echo pattern recorded to an applied load of 10Mpa with a longitudinal sensor at 50MHz.

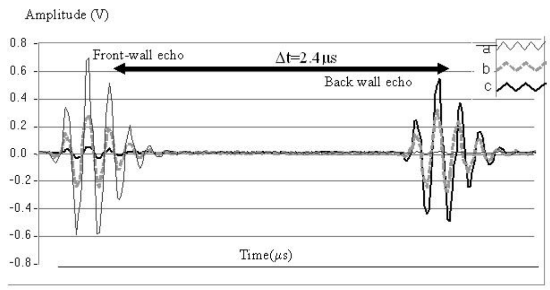

Figure 2 Echo pattern for a contact configuration

As the load is increased, the front wall echo gradually decreased whereas the back wall echo becomes visible. From these echograms analysed for the compression test, and with two sensors, longitudinal and transverse velocities $\mathbf{V_L}$ and $\mathbf{V_T}$ can be calculated from flight time measure Δt using known sample thickness. So with these velocities, elastic constants of material can be calculated [1]. So, elastic constants can be accurately measured up to high temperature. Figure 3 presents the Young modulus and Poisson's ration of an alloy of aluminium versus temperature.

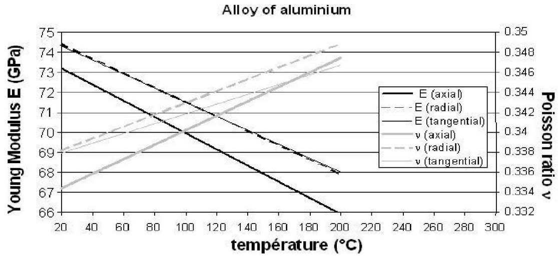

Figure 3: Variation of Young modulus and Poisson's ratio of an alloy of aluminium versus temperature using our dry coupling system

Acoustic reflection coefficient r measurement

When the sensor lays on the sample by its own weight, the incident wave is totally reflected at the end of the delay line as for an acoustic sensor drops in air. This suggests that we do not sense the underneath material. As the load is increased, we have shown that the front wall echo gradually decreased. So, we represent on figure 4 the variations of the acoustic reflectance coefficient "r" versus applied load.

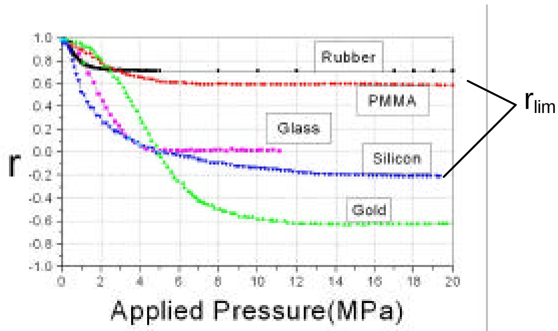

Figure 4: variation of the reflection coefficient versus normal stress for different materials using a 50MHz longitudinal transducer

This graph shows the same variations for any tested materials from soft ones (rubber) to hard ones (titanium alloy). Indeed, each curve starts from r=1 for a null applied stress (except sensor weight) and it gradually decreases to reach a minimum value (r_{lim}). First of all, this indicates that there is not any energy transmitted into the test sample for a mechanical stress inferior to 1 MPa, which roughly corresponds to a 30N load with our 50MHz sensor. Then, as the load is increased, the sensor-sample interface transmits more and more energy until it reaches a maximal value, which depends on the properties of the material under test.

The compression with our glass made delay line on the same material yields to a variation of the "r" from 1 to 0 that is to say from a complete reflection configuration at this interface to a complete transmission one.

Same results have been found using transverse waves at 5MHz. All these results validate our experimental procedure to study acoustic reflectance coefficient.
In Mechanics, two materials are in an ideal contact configuration when there is a total continuity of the stress and displacement components at this interface. This kind of boundary conditions corresponds in fact to a perfect adhesion situation. This property of elastic materials perfectly bonded and travelled by low amplitude waves yields to the following expression of the acoustic reflection coefficient (cf. Equation 1) [2].

$$r = \frac{P_r}{P_i} = \frac{Z_1 - Z_2}{Z_1 + Z_2} \qquad \textit{Equation 1}$$

1=upper medium, 2=lower medium

P_i corresponds to the incident acoustic pressure emitted by the sensor and P_r to the part reflected at the interface between the two media. "Z" corresponds to the acoustic impedance of a medium with $Z = r \times V$. "ρ" is the mass density of this material and "V" the wave velocity in this material
Equation 1 justifies that "r" becomes negative at high load when the delay line acoustic impedance is smaller than the material one (see results obtained for gold sample on figure 4).

In the specific case of our glass delay line in ideal contact with a glass sample, this theory also predicts a null reflection coefficient which is good agreement with the experimental curve corresponding to a glass sample pressed by a glass made delay line (cf. figure 4). This limit value "r_{lim}" of the reflection coefficient is reached for normal stress superior to 5 MPa. Next, for all the other tested materials, the final value of reached at high load confirms this theory of ideal contact with an error inferior to 2%. From r_{lim} value acoustic impedance of each material (Z_{sample}) can be deduced:

$$Z_{sample} = Z_{sensor} \cdot \frac{(1 - r_{lim})}{(1 + r_{lim})}$$

Consequently, this measure of the acoustic reflectance can be also used to evaluate the sample mass density "ρ" using the following equation:

$$r = \frac{Z_{sensor}}{V_{samp}} \cdot \left(\frac{1 - r_{lim}}{1 + r_{lim}} \right)$$

In this case, the sample velocity "V_{samp}" is available from flight time measure. This technique tested on glass, PMMA and different alloys has given experimentally the expected value of their mass densities with an error inferior to 5% but this method gives above all a local measure of this parameter. By comparison, incertitude of 2% on each input value induces theoretically a cumulated error of 7% for the mass density.

Acoustic reflection coefficient for detection of surface roughness

Surface properties can be modified in various ways. They can have different mechanical properties, roughness, etc. To separate these effects and to study the range of roughness affecting this dry coupling measure, small roughness levels of Si (111) p+ substrate etched by NH4F have been done. Roughness has been measured with an atomic force microscope (AFM). From AFM measurement, density power spectrum (DSP) can be calculated in order to have information about frequency level roughness. The figure 5 shows that small roughness levels may have a large effect on the variation of the reflection coefficient curve versus normal stress for longitudinal waves at 100MHz.

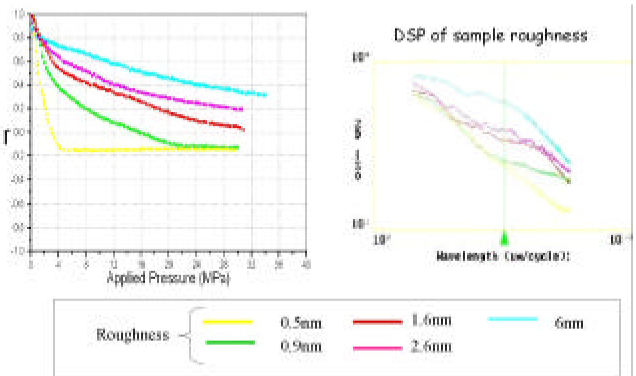

Figure 5: Variation of reflection coefficient versus applied load for different roughness levels on Si etched by NH_4F. Compare with the DSP evolution.

Indeed, silicon with a roughness as small as 6nm reflects the incident acoustic pressure up to 40% for a compression stress of 26MPa. This sample is very smooth even after the chemical attack. Only high frequency roughness is revealed. We develop a model where two surfaces parameters, roughness amplitude A and correlation length of roughness L can leave an element explanation about reflection coefficient response.

Theoretical model

In a first approximation, surface roughness can be modelled by a sinusoidal function with two significant parameters:

A : roughness level
L: correlation length

Figure 6 shows such surface.

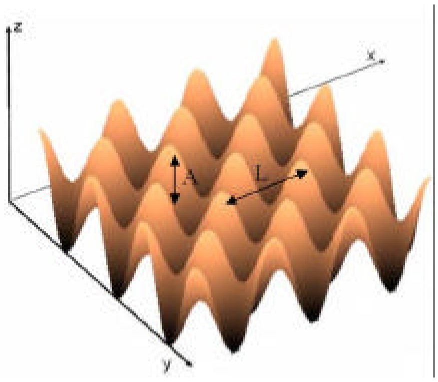

Figure 6: Surface roughness representation

So, using Hertz's theory, contact area between a flat smooth plane (sensor) with this rough surface can be calculated [3]. We shows that lower A/L ratio values is, lower the applied load required to obtain perfect contact is.

In a second part, we develop a finite element model in order to draw the reflection coefficient versus contact area. We demonstrate for a roughness given, when correlation length changes, reflection coefficient changes too. When L is greater than acoustic wavelength, a linear evolution of reflection coefficient versus contact area is shown. Quasi-static model based on contact stiffness [4] doesn't provide this evolution. With Hert's theory results and finite element ones, we can present comparisons of theoretical and experimental data of reflection coefficient versus applied load (Figure 7).

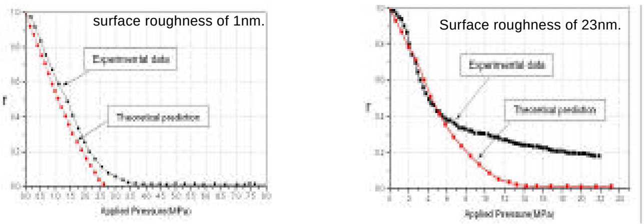

Figure 7: : Theoretical and experimental reflection coefficient versus applied load for two roughness levels.

These results show that the measured reflection coefficient from a partially contacting sensor-rough glass interface is in good agreement with that predicted by our model for small roughness.
So, when roughness is superior to 20nm, we can see that model predict smaller reflection coefficient that experimental data. We explain this by the fact that real surface contains much frequency roughness dependence and at this time our model take into account only one frequency of roughness.

Conclusion

Hard delay line sensors are suitable to measure in a non-destructive way the elastic constants on a large temperature range if these ultrasonic transducers are correctly pressed against the material under test in order to operate without any coupling fluid. Next, we have verified that, when the applied compressive stress reaches a sufficient level, the acoustic reflection coefficient corresponding to a perfect contact situation is correctly predicted by classic linear elasticity. This property can be also used to get an estimation of the sample acoustic impedance and mass density with a few percents accuracy.

For instance, measurements performed on silicon substrate etched coupled with AFM investigation have shown that a 100 MHz ultrasonic sensor was sensitive to very small roughness level in the range of 0.5 to 6 nanometers.
From acoustic reflectance measurement, hard delay line transducers can also provide tribological information such as the contact area parameters to understand contact, adhesion and friction between surfaces. A model based on the Hertz theory and our finite element has been compared with partial agreement to our experimental data for a glass sample characterised by AFM for roughness measurement.

References

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