Metal composite as backing for ultrasonic transducers
dedicated to non-destructive measurements in hostile

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Abstract. Our team is specialized in ultrasonic measurements in hostile environment especially under high temperatures. There is a need for acoustic transducers capable of continuous measurement at temperatures up to 700°C. To improve the performances of acoustic sensors we focus our works on the realisation and characterisation of transducer backings able to operate under very high temperature. Commercially, they are produced by the incorporation of tungsten powder in a plastic matrix, which limits the working temperature. The realisation of ultrasonic transducers for non-destructive measures at high temperatures requires adequate materials, manufacturing and assembly processes. To produce the backings, composites were made using very ductile metals such as tin and tungsten. These composites are manufactured by uniaxial hot pressing. First, we studied the influence of temperature and pressure on the densification of tin pellets. Then, several specimens made of tin/W were made and characterised by measuring the specific weight, speed and attenuation of sound. The acoustic measures were realised by ultrasonic spectroscopy. This test-bench was designed and tested on control samples of PMMA and on standard backings (epoxy / tungsten).

1. Introduction

Currently it does not exist acoustic transducers capable of operating continually at temperatures up to 700°C. However, the need is real in many industrial sectors (Nuclear, Oil, ...). The Institute of Electronics and Systems (IES) of Montpellier has been working for many years, especially with the Commissariat of Atomic Energy (CEA) for the development of acoustic instrumentation in hostile environment (High temperature, neutron flux).

The realisation of ultrasonic transducers devoted to the non-destructive measurement in hostile environments requires finding adequate materials, manufacturing and assembly processes. The ultrasonic transducers are composed of 3 elements: an active element such as a piezoelectric element, a front layer to inject a maximum of acoustic power into the investigated material and finally, a backing. The backing permits to control the bandwidth, the temporal resolution and the sensitivity of the transducer. To increase the bandwidth, the acoustic impedance $Z$ (which is the product of the speed of sound to the density) of the backing must be equal to those of the active element and the backing must be strongly attenuating.

The backings are particulate composite materials made of a matrix with a low acoustic impedance in which spherical scatters with high acoustic impedance are dispersed. The quantity and diameter of scatters control the impedance, attenuation and velocity of the backing. Standard backing mostly used in industry are made of polymer and inclusions of tungsten, because it is simple to fabricate. These mixtures allow acoustic impedances ranging from 0.1 MRayls to 100 MRayls depending on the volume fraction of tungsten introduced [1-2].

However, whichever the polymer used, none of them is able to work at temperatures up to 700°C. In order to increase the work temperature, we propose to use metallic composites. To obtain a suitable
protocol of manufacture, we start our study on tin-tungsten composite because tin is a very ductile metal allowing working at moderate temperatures and pressures. The melting temperature is the only limiting factor for the sensor. In addition, the protocol implemented using tin may be applied to other ductile metals having higher melting temperatures (zinc, copper ...). In this study all specimens were made by temperature uniaxial pressing.

The study conducted by Sayers [2] on such metallic composites is only theoretical. In the reference [3], the author does not give the attenuation of the composites made nor results on the densification. Thus, the mechanical strength of composite are proposed. It was then decided to start our work by studying the densification of tin pellets as a function of temperature and pressure. Then we give the explanation of the manufacturing process and the characterisation method of Tin-Tungsten backing.

Three backings were made and characterised. In this paper we present for each specimen the speed of sound, the attenuation and impedance as a function of the volume fraction of inclusion.

2. Realisation of tin pellets

We manufactured tin pellets with a white tin powder of 99.5% purity, mean diameter of about 149 microns and a density of 7.29g / cm$^3$. To determine the pressure and temperature leading to samples with good mechanical strength and relative density, it was decided to vary some parameters to have a density close to pure tin. The parameters are the pressing load, the pressing speed and the pressing temperature to observe their effects on the final properties (relative density and mechanical strength) of the pellets.

The pellets and the backing were made by simple uniaxial hot pressing processing, by a single moving piston [Fig 1]. We choose this kind of process for its simplicity, to reduce the costs of manufacturing and to increase density.

The samples were loaded in compression using 1196 mechanical testing machine with 250kN maximum load capacity. It is controlled by a software allowing to choose the maximal pressure and the pressing speed.

Heating is provided by a heating element wrapped around the matrix. To minimize heat losses, an insulating glass wool is wrapped around the heating element. The temperature is controlled by a PID. A thermocouple, inserted into a hole filled with thermal grease drilled into the matrix measures the reference temperature [Fig 1].

The desired amount of powder is weighed and then introduced into the matrix. To determine the amount of powder $m_{Sn}$ to be weighed, we arbitrarily fix a final height $h_{Sn}$ which is used to calculate the final volume of the pellet knowing the diameter $D_{Sn}$ of the matrix.

Two various tests are carried out:
- Two cold tests at various pressing speeds (0.1 mm/min, 1mm/min),
- Three tests at constant speed and different temperatures (25°C, 50°C, 100°C).
2.1. Influence of the pressing speed
When pressing, there are frictions on the rim of the pellet which depend on the pressing speed. The pressing speed is then an important parameter to study. Two samples of tin powder were pressed under the same temperature and maximum pressure (1600 Bars) but with different pressing speeds (0,1 and 1 mm/minute).

![Figure 2: Influence of the pressing speed of the relative density at ambient temperature](image)

It is observed in figure 2 that for both pressing speeds, the changes in relative density as a function of the pressing force is the same. It seems that the pressing speed has not influenced the densification of the tin samples. However, the sample pressed at 1 mm/min had a bad mechanical wear, the sample breaks during demolding because of the important radial frictions and therefore cannot be characterized. It was then decided to press at 0.1 mm/min.

We can now proceed to the second study which is the influence of temperature on the densification.

2.2. Influence of Temperature
A few works show the dependence of the densification used to make metallic samples [4-5]. However the papers we cite use an isostatic press. We need to check the influence of the temperature in our conditions (uniaxial press). In this part, we will highlight the importance of the temperature on the realization of dense pellet. Three tin pellets were realised at 25°C, 50°C and 100°C.
The relative density as a function of the pressure is shown in figure 3. We observe a significant difference between an ambient pressing temperature and a 100°C pressing temperature. The relative density grows faster and its value is higher at 100°C and reaches 99% of relative density. We do not find significant difference between the ambient temperature curve and the 50°C curve. However, it would be preferable to press at ambient temperature. At this temperature there is no delaminating. Indeed, the fact that tin is more ductile at high temperature increases the radial friction and consequently the differences of compaction increase the phenomena of delaminating.

Finally we retained the following protocol. The sample is:

1. shaped with a 1600 bars maximum pressure at room temperature,
2. reversed and pressed again (double action),
3. annealed at 100 °C.

This tin study allowed choosing the pressure, temperature and the pressing speed for the realisation of tin/tungsten backing.

3. Realisation and characterisation of transducer’s backings

3.1. Realisation of tin/tungsten backings

The backings with tungsten particles (size between 140 to 200 micron diameters) are realised according to the following protocol:

1. Weigh the quantity of tin and tungsten desired,
2. Mixture of tin and tungsten,
3. Introduction of the mixture into the matrix, then pressed dynamically until 1600 bars, with holding pressure,
4. Annealing at 100°C.

3.2. Estimation of the phase velocity and attenuation

The method used to characterise the backings is the ultrasonic spectroscopy using the insertion/substitution method [6]. It consists of two measurements [Fig 4], a first reference measurement is performed through water, then a second measurement through the material being studied.
If only two echoes are used, this method requires knowing the thickness "d" of the sample and its density "ρs", the temperature "T", the density "ρw" and the speed of sound "cw" of the water during the experiment.

Figure 4: Principle of the ultrasonic spectroscopy immersion method

4. Results and discussion

To characterise our backings, we used a pair of 5MHz plane sensor central frequency, covering the frequency range between 4MHz and 6MHz. We used a pulse generator Olympus 5052 PR and a Tektronix oscilloscope. The sampling frequency is 1GHz and the acquisition is performed on 1000 points.

The characteristics of the three backings are:

Tableau 1: theoretical and experimental densities for three backings with different volume fraction of tungsten

|          | Theoretical densities (g/cm³) | Experimental densities (g/cm³) |
|----------|-------------------------------|-------------------------------|
| Backing1 | 7.91                          | 7.76                          |
| (5%W)    |                               |                               |
| Backing2 | 9.11                          | 8.98                          |
| (15%W)   |                               |                               |
| Backing3 | 10.91                         | 10.77                         |
| (30%W)   |                               |                               |

For each sample, there is a reference measurement and a measure through the sample. The phase velocity and the attenuation are respectively deduced from the phase and the amplitude.

The figure 5, 6 below compare the experimental and theoretical speed of sound and attenuation depending on the frequency for the three samples (5% W, 15%W and 30%W).

Figure 5: Speed of sound of 3 different fractions (5%, 15%, and 30%) of tungsten in backing as a function of ultrasound frequency at 26°C, for a transducer with a 5MHz center frequency
It is found that the higher the volume fraction of tungsten, the higher the phase velocity. However, the phase velocity for the backing having a volume fraction of 30% tungsten is above the speed of the other two with a lower volume fraction, it is in accord with the theory [1-7]. The average relative differences between the theoretical and the experimental measures for each backing are:

| Backing | Average relative difference for the speed of sound [%] |
|---------|-------------------------------------------------------|
| Backing1 (5%W) | 2.94 |
| Backing2 (15%W) | 4.20 |
| Backing3 (30%W) | 2.22 |

Although the theoretical curve does not seem correctly fit the experimental data the relative difference is quite low. The relative difference is more important for the 15% backing it is probably due to a quantity of porosity more important than the two other.

Concerning the attenuation coefficient shown in figure 5, it is found that the higher the volume fraction of tungsten, the higher the attenuation. The average relative differences between the theoretical and the experimental measures are:

| Backing | Average relative difference for the attenuation (%) |
|---------|-----------------------------------------------------|
| Backing1 (5%W) | 17.63 |
| Backing2 (15%W) | 13.57 |
| Backing3 (30%W) | 8.57 |
The differences between the simulation and the experiment are more important for the attenuation than for speed of sound. These differences can be due to several parameters:

- The presence of porosity which reduce the value of speed of sound in the matrix
- The experimental diameter of the diffuser is between 140 and 200 µm, whereas the simulation was made with a single diameter 170 µm (mean of 140-200 µm). Indeed, during the simulation we observed that the values of the attenuation coefficients change with the theoretical values of the particles diameter [Fig 6].

- Simulation does not take into account the attenuation in the tin matrix which reduces the attenuation coefficient of the backing. It is found an important average relative difference of 17.63% for the attenuation in the backing1 (5%W). Indeed, the backing1 has the lowest volume fraction of tungsten. The attenuation of the backing1 is close to the attenuation of the tin matrix and in this case we cannot neglect the tin matrix attenuation. For the backing3 (30%W) the average relative difference for the attenuation is 8.57%. This result is justified by the high concentration of tungsten and in this case we can neglect the attenuation in the tin matrix because attenuation is a consequence of the scattering on a spherical obstacle (tungsten).

5. Conclusion
We studied the influence of the pressing speed and of the temperature on the manufacture of pure tin pellets which allowed us to deduce a protocol. It was shown that although the pressing speed does not seem to change the densification, it is important to slowly press to have good mechanical strength. The metal is more ductile at 100°C and significant densification rates are achieved quicker. Yet, it is better to press at room temperature and to anneal after pressing at 100 ° C. This significantly reduces the delamination.

The realisation of backings by uniaxial pressing with powder allows increasing the particle size and the volume fraction of tungsten because there is no sedimentation. We realised several samples and characterised them by ultrasonic spectroscopy to determine their velocity, attenuation. We concluded on the effectiveness of the method of manufacture. Even if they are differences between simulation and experiment results, we have acceptable relative differences.

6. Bibliography
[1] M.G. Grewe and T. R. Gururaja 1989 IEEE Ultrasonics symposium Vol 173 Acoustic properties of particle/polymer composites for transducer backing applications
[2] C.M .Sayers and C.E .TAIT 1984 Ultrasonics Vol 22 .p57-60 Ultrasonic properties of transducer backings

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**Figure 6: Attenuation of sound in 4 different backings with different diameters of tungsten particle (150 µm, 170 µm, 180 µm and 200 µm) as function of ultrasound frequency at 26°C, for a transducer with a 5MHz center frequency**
It is found between these two signals a decrease in amplitude and a phase shift [Figure 5]. The phase shift is due to the fact that the material at a different speed of the water \( \varDelta \phi = \omega V_s(f) \times d \)

The decrease in amplitude is related to two phenomena: the attenuation characterized \( \alpha \) and losses inserts characterized by the energy transmission coefficient \( T \) which is:

\[
T = \frac{4(Z_w Z_s)}{(Z_w + Z_s)^2}
\]

where

\[
Z_w = \rho_w V_w \quad \text{et} \quad Z_s = \rho_s V_s
\]

Experimentally we deduce the velocity and attenuation from the acoustic transfer function.

\[
H(\omega) = \frac{P_s(\omega)}{P_w(\omega)} = T \exp(-\alpha(\omega V_s d) \exp(-j\omega d)} \frac{1}{V_s} - \frac{1}{V_w}
\]

From the phase \( \Delta \phi \) of \( H(\omega) \) is deduced the speed and amplitude \( |H(\omega)| \) the attenuation coefficient.

The velocity is given by the following relationship:
\[ V_s \omega = \frac{V_w}{1 - \frac{\Delta \theta V_w}{\omega d}} \text{ en m/s} \quad (3) \]

Where \( \Delta \theta = \theta_s - \theta_w \). Thus it is freed from the phase shifts introduced by the cables and the impulse responses of the transducers.

From the amplitude spectrum we have:
\[ \alpha_s \omega - \alpha_w = \frac{1}{d} \ln \frac{P_s}{P_w} = \frac{1}{d} \ln T - \ln H \omega \quad (4) \]

The attenuation of the water \( \alpha_w \) being negligible compared with that of the material \( \alpha_s \).