Deliverable 3.3

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Cross-sectorial real-time sensing, advanced control and optimisation of batch processes saving energy and raw materials (RECOBA)

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Acoustic sensor for particle morphology monitoring

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Dissimination level

PU	public	☒
PP	restricted to other programme participants (incl. the Commission Services)	☐
RE	restricted to a group specified by the consortium (incl. the Commission Services)	☐
CO	Confidential, only for members of the consortium (incl. the Commission Services)	☐

CONTENT

1 Introduction

The aim of deliverable 3.3 is to describe the progress made and the results achieved on particle sensing using thin film bulk acoustic wave resonator (FBAR) technology. The work performed has been mainly focused on two different areas:

1. Fabrication of the piezoelectric FBAR transducer
2. Sensing experiments with solutions containing polymer particles

While the work on the fabrication of the piezoelectric FBAR transducer was performed in the laboratories of the University of Cambridge, the sensing experiments took place in the Universidad Politécnica de Madrid, in the facilities of the Group of Microsystems and Electronic Materials (GMME), during the first two weeks of December, enabled by a COST Action STSM. As it will be described in the corresponding section, they have set up a custom-made experimental arrangement that provides a reliable way to perform sensing experiments and handling liquid samples using FBARs.

2 Fabrication of the piezoelectric FBAR transducer

2.1 Deposition of ZnO layers

The use of FBAR as sensors is based on the change in their resonant frequency that occurs when a mass is attached to their surface, also known as gravimetric sensing. The resonance is achieved through the conversion of electrical energy applied to the device into mechanical energy (and vice versa) thanks to a thin piezoelectric layer, usually zinc oxide (ZnO) or aluminium nitride (AlN), that is sputter-deposited between the metallic electrodes. There are two main parameters that will indicate the goodness of a FBAR device: the electromechanical coupling factor (k^2) and the quality factor at the resonant frequency (*Q*). The first one indicates the efficiency of the conversion between the electrical energy applied to the device and the acoustic energy generated by the piezoelectric layer. It depends mainly on the quality of this piezoelectric layer. The second parameter, the quality factor, is related to the energy losses suffered by the resonator and depends on many factors, such as the contacts, the materials used in the device, its geometry, etc. The *Q* is the critical parameter in resonators intended for sensing applications, because it is related to the sharpness of the resonant peak. If it is not sharp enough it is extremely difficult to determine the value of the resonant frequency accurately. In that case it is not possible to track it; therefore identifying the shifts related to the mass being bond to the surface of the transducer is not feasible.

FBARs display two different modes of operation depending on the direction of the vibration of the particles with respect to the direction of propagation of the wave. If the vibration is perpendicular to the propagation, we will refer to the mode as the ‘shear’ mode. If it is parallel, the mode will be the ‘longitudinal’. The shear mode has a particularity that makes it especially suitable for in liquid sensing: it does not couple into the liquids, which means that it suffers a much lower attenuation (maintains a higher *Q* in liquid compared to the longitudinal mode). Figure 1 displays the frequency response of a resonator showing both modes and operating in air and in liquid.

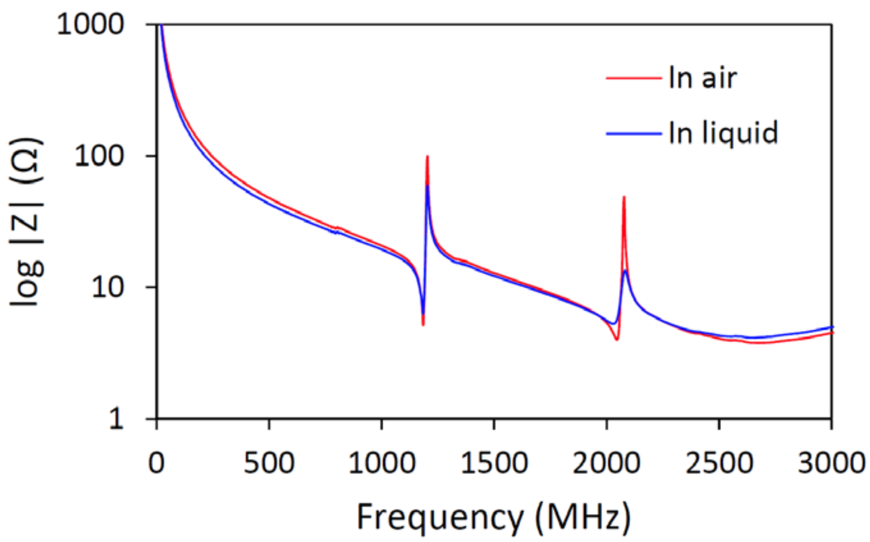

Figure 1 – FBAR displaying the shear (at ~1200 MHz) and the longitudinal mode (at ~2050 MHz) operating in air and in liquid.

While the shear mode is capable of maintaining a $Q \approx 150$ in liquid, the Q of the longitudinal mode reduces to values below 10, which is insufficient for sensing purposes.

The requirements set by the RECOBA project involve the detection of the particles in fluids, which essentially means that the FBARs must operate in the shear mode. However, the excitation of this mode is not straightforward, and is dependent on the microstructure of the piezoelectric materials. We have optimized a process for producing piezoelectric ZnO films with a microstructure to yield FBAR devices with the response shown in figure 2.

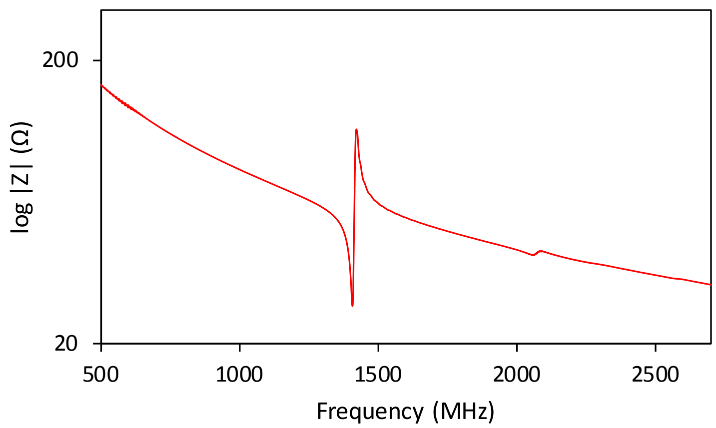

Figure 2 – FBAR operating in the shear mode and fabricated using ZnO thin films with an optimised microstructure.

The devices fabricated display a k^2_{shear}≈2.8% and a Q≈120, which ensures that the resonant peak is sharp enough to determine the resonant frequency accurately.

2.2 Design of the test transducers

Another challenge derived from the requirement of the operation in liquids is the actual handling of the samples and the compatibility with the devices. The design of the resonators should enable the fitting of a fluidic system that keeps the sample away from the measurement probes. This means that the contacts to the active area must be extended. Given the high frequency of operation of the FBARs, above 1 GHz, such extensions should be carefully designed to control the parasitic elements they add to the equivalent circuit of the device and that can cause serious damage to the resonance [1]. This has been achieved using a specific FBAR device structure and microfluidic system which is ready to be attached to a measurement setup to perform particulate detection.

3 Sensing experiments with solutions containing polymer particles

As it has been stated in the introduction, the sensing experiments were performed in the laboratories of the GMME in Madrid. The sensor is attached to the closed fluidic circuit that flows the samples using a fluidic pump. Figure 3 shows a picture of the setup, with the different parts highlighted.

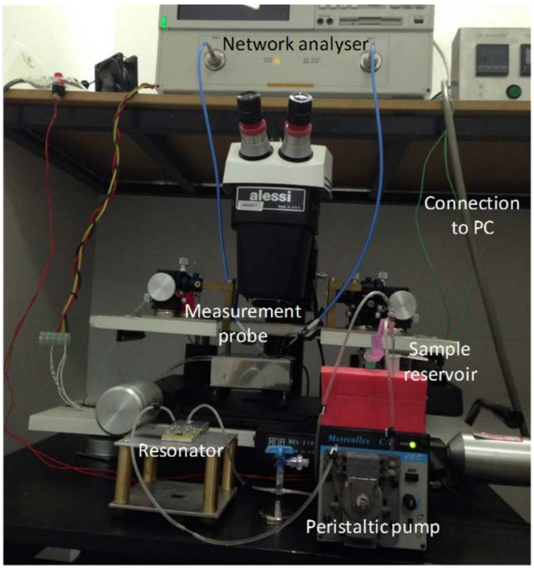

Figure 3 – Picture of the experimental setup with closed fluidic circuit.

The experimental setup enables recirculation of the samples if necessary. The tracking of the resonant frequency in time is performed using ad-hoc LabVIEW software that determines accurately the resonant frequency from the frequency response acquired with the network analyser and represents its evolution in time.

The polymer samples used for the experiments were provided by Polymat. They are structured particles with a core-shell structure. The test solutions where prepared from the samples diluted in deionised (DI) water. Prior to the feeding of the prepared sample DI water was introduced in the circuit while tracking the resonant frequency to stablish a baseline. Figure 4 shows the result of one of the described experiments.

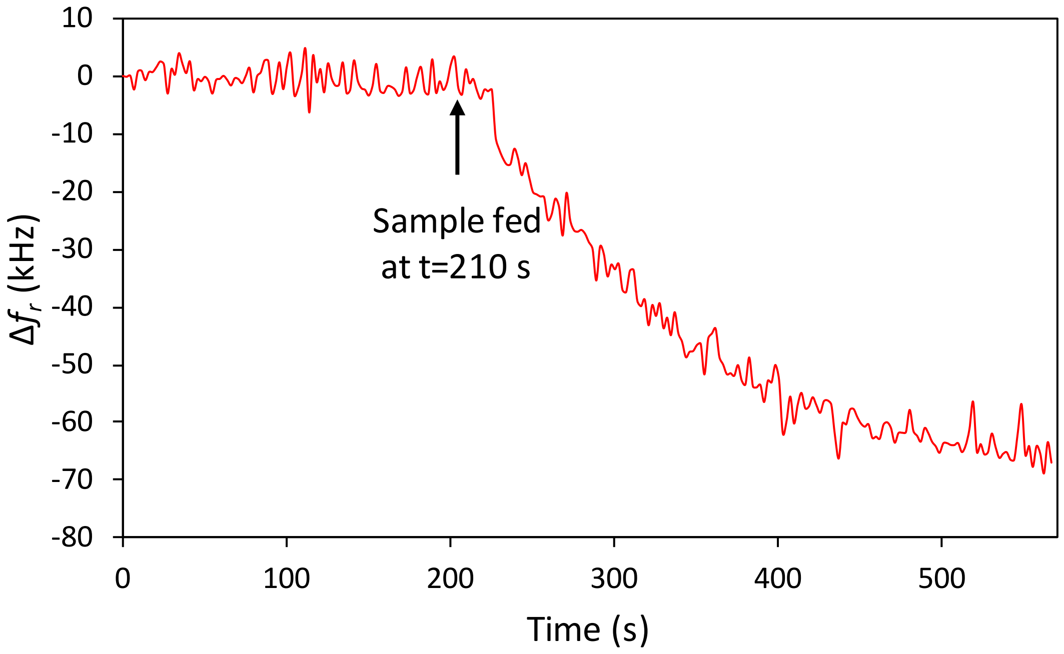

Figure 4 – Evolution of the resonant frequency of the transducer during the experiment.

When the sample contacts the active area of the device a progressive shift takes place until saturation. From previous experiences with this kind of devices, such a shift cannot be attributed to a change of the viscosity of the liquid, which suggests that the particulates must be binding to the active area. Rinsing with DI water with the same rate does not detach the particles.

Even though these experiments only constitute a proof of concept, they are extremely promising. The detection of the particles seems to be possible using FBARs in the light of the presented results. New experiments will be performed in the coming months using solutions with different types of particles, with the objective of finding a correlation between the frequency shift and the morphology (at least the size if not the shape) of the particulates.

4 Conclusions

As a summary of this deliverable 3.3 the following conclusions can be drawn:

- A novel method to deposit microstructured ZnO thin films has been developed. Using this method, it is possible to fabricate shear mode FBARs that display a k^2_{shear}≈2.8% and a Q≈120. Test transducers have been designed and fabricated to fit a custom made fluidic system necessary to handle the liquid samples.
- Sensing experiments of polymer particulates have been performed using FBARs with successful results. They constitute a proof of concept and further experiments need to be perform, but they are very promising and indicate that the detection is possible using FBARs.

The next months (M13-M19) will be focused on the development of surface acoustic wave devices (SAW) for the identification of the morphology of the particles. In parallel the work on the FBAR technology will continue to optimise the design of the transducers and test their response to different type of particles.

5 References

[1] M. DeMiguel-Ramos, M. Barba, T. Mirea, J. Olivares, M. Clement, J. Sangrador, and E. Iborra, “Influence of the electrical extensions in AlN-BAW resonators for in-liquid biosensors,” in *Proceedings - European Frequency and Time Forum (EFTF)*, 2014, pp. 301–304.