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Real-time on-line ultrasonic monitoring for bubbles in ceramic 'slip' in pottery pipelines ☆

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A B S T R A C T

When casting ceramic items in potteries, liquid 'slip' is passed from a settling tank, through overhead pipelines, before being pumped manually into the moulds. It is not uncommon for bubbles to be introduced into the slip as it passes through the complex piping network, and indeed the presence of bubbles is a major source of financial loss to the ceramics industry worldwide. This is because the bubbles almost always remain undetected until after the ceramic items have been fired in a kiln, during which process bubbles expand and create unwanted holes in the pottery. Since there it is usually an interval of several hours between the injection of the slip into the moulds, and the inspection of the items after firing, such bubble generation goes undetected on the production line during the manufacture of hundreds or even thousands of ceramic units. Not only does this mean hours of wasted staff time, power consumption and production line time: the raw material which makes up these faulty items cannot even be recycled, as fired ceramic cannot be converted back into slip.

Currently, the state-of-the-art method for detecting bubbles in the opaque ceramic slip is slow and invasive, can only be used off-line, and requires expertise which is rarely available. This paper describes the invention, engineering and in-factory testing across Europe of an ultrasonic system for real-time monitoring for the presence of bubbles in casting slip. It interprets changes in the scattering statistics accompanying the presence of the bubbles, the latter being detected through perturbations in the received signal when a narrow-band ultrasonic probing wave is transmitted through the slip. The device can be bolted onto the outside of the pipeline, or used in-line. It is automated, and requires no special expertise. The acoustic problems which had to be solved were severe, and included making the system capable of monitoring the slip regardless of the material of pipe (plastic, steel, etc.) and nature of the slip (which can be very variable). It must also be capable of detecting bubbles amongst the myriad solid particles and other species present in the flowing slip. The completed prototype was tested around several factories in Europe, and proved not only to be more versatile, but also more sensitive, than the state-of-the-art method.

1. Introduction

Around 1–2 million Euros per year are lost from the business of an average European medium size sanitary-ware factory through the unwanted presence of air bubbles in the 'casting slip' (the liquid suspension from which the ceramic items are eventually produced). Potteries for tableware, decorative and specialist items are also hampered by the presence of bubbles. The bubbles cause faults in the final ceramic articles, which are known as 'pinhole defects' [1]. The defective tableware is rejected, resulting in significant financial losses to the ceramic factory: the detection of pinhole defects often occurs hours after the bubbles were introduced, when the pottery is examined after firing, so that not only can hours of production time be wasted through use of persistently bubbly slip, but also the spoilt material cannot be recovered and recycled. The production process is vulnerable to the introduction of unwanted bubbles through pumps, piping and processes associated with mixing and injection: in the factory, the casting slip (also called 'slurry') is pumped from a settling tank into overhead pipes, from which slip is drawn off through a number of subsidiary pipes before being injected into a mould via a nozzle (in a process not dissimilar to the hand-operated nozzles used in petrol stations for refuelling cars) [1]. The filled moulds are then taken for firing in a kiln, during which process the bubbles expand, creating pinholes. If the air bubbles could be identified and quantified in the slip as it flows through the pipelines before the casting (that is, by continuous on-line monitoring), the slip could be diverted to a

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☆ Note that this work was presented by Mr. Yim in the student session at the 2006 Spring conference of the UK Institute of Acoustics, for which Mr. Yim received the Association of the Noise Consultants prize for "best paper presented at an Institute of Acoustics conference by a young person in 2006".

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settling tank or secondary device to eliminate the air bubbles. The object of this research was to provide the prototype for such a device, operating non-invasively and in real time. The device needs to be easy-to-use, mounted external to the pipes and portable, so that it could be relocated to track back to the source of bubbles [1,2].

The state-of-the-art method used by the industry for measuring gas bubbles in slip is called the 'compressibility test', and it does not meet these requirements. In this 'compressibility test', the expert removes a sample of slip from the injecting nozzle or the settling tank. A compressive force is then applied to the sample, and the amount by which it compresses indicates the total volume of gas present, and hence the void faction (the percentage of bubbly liquid which is made up of free gas). The 'compressibility test' is undertaken only on samples of slip, which represent a very small fraction of the slip flowing at any time; it is not on-line and not continuous, and indeed the expertise is not widespread. It is performed intermittently (indeed, rarely); it samples a tiny (and perhaps unrepresentative) volume of slip; it is slow and entirely manual, and requires expertise which is uncommon in the industry both for undertaking the test and interpreting the results. Furthermore, it can only be undertaken at the settling tank or nozzle, and therefore cannot be used to 'track' back along the pipeline to determine the source of bubble generation (such as a particular bend or junction in the pipe). Continuous monitoring of air bubble contamination is not possible, and the test certainly does not lend itself to averaging or to monitoring the performance of a factory over time, or in response to changes made to the pipeline. The test is invasive, and there is the potential for bubbles to be introduced by the measurement process (giving a false positive result) or to escape or dissolve during the measurement (giving a false negative result). The sensitivity of the compression test is so limited that even a void fraction which would be considered by factory as 'high' (with respect to its ability to ruin the product) is too small to be detected by the compression test. This is demonstrated in Table 1, which summarises the range of size of bubbles, and void fractions in the slip, required of the device.

This project was designed to provide the ceramic industry with a device which would monitor for the presence of bubbles, and determine the void fraction, on-line and real-time in various types of slip (of which even the bubble-free properties cannot be predicted by the authors, since the device was to be tested in this project around Europe in potteries where the authors had minimal *a priori* knowledge of slip or pipework). It uses sensors which are mounted outside of piping (so requiring no modification to the existing piping and, furthermore, be capable of being moved along the piping to track a bubble population to its source). It is designed to function robustly despite the wide variations in pipe thickness (both inner and outer diameter) and materials. The over-riding consideration was that any production unit based on this prototype should be designed to be economically viable (i.e. inexpensive, for example, no more than a few thousand Euros per unit) with respect to manufacturing, initial purchase, subsequent operation; and the device should be amenable for operation, installation, and the interpretation of results by the unskilled user. The end-device could not fail any of these criteria, but the research budget was very restricted. The eventual device was based on the interpretation of changes in the scattering statistics accompanying the presence of the bubbles, the latter being detected through perturbations in the received signal when a narrow-band ultrasonic probing wave is transmitted through the slip. However, before reaching this final solution, a number of other possible options were considered.

Table 1
Typical detection ranges requested by industry in the specification for the device. The void fractions listed in the table are based on experience using the compression test. Note that the bubble resonance frequencies are lower than the corresponding resonance frequencies for air bubbles of the same size in water, because of the differences in density and viscosity of slip. The maximum and minimum bubble sizes state the industry recommendations for the range of bubble sizes that should be detectable by a useful sensor: this does not necessarily mean that bubbles outside this range do not occur in slip, but rather than such bubbles appear not to cause problems in production.

Description given by the ceramics industry	Magnitude	Comment
Maximum bubble size	1000 μm	Resonant at 2.5 kHz
Minimum bubble size	50 μm	Resonant at 50 kHz
Minimum detectable void fraction	0.002%	Using the compression test
A 'huge' void fraction	0.03%	Detectable by compression
A 'large' void fraction	0.01%	Detectable by compression
A 'high' void fraction	0.001%	Not detectable by compression

Although there exists a wide range of acoustic techniques for characterising the bubbles in liquids [2–4], most of the technology currently does not fit all the requirements mentioned above, particularly the restrictions in terms of cost, simplicity of hardware, ruggedness, minimal user training and reliability for use with a range of pipes and their contents (the details of which would not be predictable). In many cases of bubble detection, bubble size information is gained through exploiting the bubble resonance (Table 1 shows the value of that resonance for the bubble populations of interest to the ceramics industry). However, with a few exceptions [5,6], the underlying theory to do this is based on free-field assumptions, which can cause large errors when applied to tanks and pipes because, for example, the geometry can affect the fluid loading [5–10] and constrain the fluid dynamics [11–15]. Furthermore, the deployment of bubble sensors across a pipe involves a geometry which can introduce a wide range of errors into the measurement, such that although the algorithm which interprets the received signal can produce an estimate of the bubble size distribution, that estimate is inaccurate [16].

All techniques for bubble detection have inherent advantages and disadvantages and so it is advisable to deploy several simultaneously for cross-validation [3,17–19]. However, in the current project this is clearly impracticable, given that it is difficult to find even a single technique to match the requirements above. That technique is described in the following section.

2. Methods

Passive acoustic techniques interpret the sound generated by bubbles upon entrainment to obtain a bubble size distribution [20] (although usually through an inversion based on free-field models, which are often inapplicable in the industrial environment [5,6]). Active acoustic techniques usually work by detecting the bubble resonance, using for example, the fact that scattering of ultrasound by the bubble has a local maximum at resonance frequency because of its oscillatory behaviour. To identify the resonant characteristics of bubble, the sound field needs to consist of a series of tones, or some other broad band signal (e.g. a chirp, a pseudo-random sequence, etc.) to find the bubble resonance [3,17]. This in turn requires a broadband sound source. Such sources are too expensive for this project's research budget, and a market analysis for the project indicated that incorporation of a broadband source into the design of the device would make the cost of any marketable sensor, developed from our prototype, more expensive than the potteries stated they would be willing to pay.

A solution which, for cost reasons, uses a narrow-band transmitter had to be found. Because the device will be used in a wide range of environments (in terms of materials, geometries, reverberation, liquid suspensions, etc.) about which the authors have no specific knowledge, it was decided to simplify its output commensurate with delivering a robust measure of the bubble population when deployed by an unskilled user. For these two reasons, the interpretation was not based on the bubble resonance, such that the output would be in terms of the void fraction, rather than the bubble size distribution. The most common way of obtaining void fraction is to appeal to a form of Wood's equation, assuming that all the bubbles are smaller than the bubble size that would be resonant with the driving frequency (whilst Wood's equation has a long history of application to two-phase suspensions, it has recently been applied to materials which are in reality three-phase [21–23]). However, that approach is difficult here, since it depends on knowledge of the properties (e.g. sound speed) of the bubble-free liquid, which as stated above will neither be known nor static; and it requires measurement the bubble-mediated sound speed. Direct measurements of sound speed through time-of-flight are difficult because the requirement is to send sound from an external transducer, across the short distance of the pipe diameter, to be received by a transducer external to the pipe; this raises the possibility for errors described elsewhere [16] and in addition, the frequency-dependent phase calibration of the receiver can introduce significant systematic errors (which can be partially mitigated if the receiver positions can be swapped, if two or more hydrophones are used, noting that immersion in sediment-like materials can change the calibration [24]). Indirect measurement of the bubble-mediated sound speed, for example, through the vessel modes, is complicated by coupling between the slip and the walls [25–29]. The largest problem arises because the materials and geometries of the walls, and the composition of the slip, can vary greatly, and the device must be sufficiently robust to provide accurate measurements regardless of changes in these factors, and lack of knowledge of the material properties.

For the ceramics industry, therefore, an alternative to use of Wood's equation had to be found. The device was consequently devised to interpret the scatter and attenuation of the sound field. Given the high levels of attenuation, a transmission measurement was used, so that in the absence of bubbles the amplitude of the directly-transmitted signal would be high. However, such an implementation is not simple, because of the transmission paths through walls [16], and because the slip already contains many millions of particles which can scatter and attenuate the sound field [30–34]. It is, however, in principle possible, when bubbles occur amongst scattering particles in suspension, to detect just the bubbles (and indeed just the bubbles of a specific size, which can be chosen by the operator, although this was too costly to implement for the ceramics industry). This is through the use of two insonifying frequencies, a technique which has successfully been used to measure bubble size distributions in the oceanic surf zone, where breaking waves can generate dense mixtures of bubbles and suspended sand [18,35]. This is particularly relevant, since the effect of suspended particles on the signal in question is very much less than the effect of the bubbles, as required here. The bubble population is diagnosed from the time-varying scatter of an imaging signal. This signal is scattered by a bubble which is driven to pulsate by a pump signal (a range of bubble sizes being detectable when the pump source is sufficiently broadband to cover the range of possible bubble resonances present) [36–41]. Such a technique would be impractical for the ceramics industry, primarily because of the cost of the hardware, but also because of the complexity of propagating a MHz imaging signal through various pipe walls and slip. However, bubbles of the sizes indicated in Table 1 offer another option, whereby the modulation of the scattering by a bubble is not the result of its being driven to pulsate by a pump signal, but rather comes by another source.

Whilst a major strength of the two-frequency technique described above is that it can ignore all other sources of perturbation in the signal and look only at those sources related to the pump frequency (and therefore which can be ascribed to resonant bubbles), the technique chosen for the ceramics industry exploits those other sources of perturbation. If a single 'carrier wave' frequency is projected into the pipe, then every second millions of slip particles will leave this acoustic sensing volume, to be replaced by millions more. The statistics of the scatter will not change. However, if a bubble enters this volume, the statistics of the scatter will change significantly, and it is this principle which is used for bubble detection by the device. The passage of a bubble through the sensing volume modulates the detected 'carrier wave' signal through a number of mechanisms involving bubble presence [16], pulsation [42,43] and shape oscillations [44] induced by flow or passage of the bubble through the inhomogeneous sound field [1,45,46] which can include modal functions that may generate feedback effects with the bubbles [45–49]. All these perturbations occur on characteristic timescales, which can be modelled (future work will exploit these models to enable the sensor to be used for absolute measurements, but currently the sensor is calibrated against the compression test). Project budget restrictions compelled a reliance on gratis transmitters and receivers, and a pair were found that were capable of supporting a 37 kHz 'carrier wave'. This frequency is higher than the resonant frequencies of many of the bubbles in which the industry stated they were interested (Table 1). However, these frequencies were not so high as to be unusable because of the attenuation by the slip. Therefore, whilst it would be impossible to exploit the bubble resonance to characterise all the bubbles of Table 1 using this narrow-band signal, these gratis transducers did possess the characteristics required to generate a carrier signal. The projector insonifies a region having a volume of O (litre). The envelope characteristics of the received signal are extracted and plotted as a time history, and the cumulative energy in this is then related to the void fraction present.

3. Results

A scaled version of a factory rig was built in the laboratory at ISVR to develop and characterise the prototype before it was tested in the factory environments. The rig included a circulating piping, slip reservoir, diaphragm pump and supporting structure. One of the vertical pipe sections was chosen for 'static' or 'flowing/pumped' tests (i.e. when the pump was, respectively, not activated, or activated). The rig could be filled with water or slip (although the latter could only be used in flowing (pumped) tests or else it would set). The inner diameter of the pipe was 55 mm and the wall thickness was 4.5 mm. The sensor was mounted on a vertical section of pipe. Air bubbles could be introduced through a hypodermic needle, inserted through the wall of this vertical section, below the level of the sensor. When water was used, the size of bubble could be estimated by optical observations (including photography) and cross-checked with the size calculated from the measured rise speed of bubble [50].

The initial tests were conducted in 'static' water, without pumped flow. The configuration for these tests (shown in Fig. 1) was obtained when the flow loop was opened above a vertical section of pipe. This meant that there was an accessible free surface 1.85 m above the bubbler, which allowed the void measurement to be undertaken simultaneously with the acoustic measurement by collecting air bubbles using the measuring cylinder and flask (Fig. 1). The inset photograph (at the top right of Fig. 1) shows the transmitter and receiver transducers mounted on the middle of test pipe when it is filled with ceramic slip. To enhance the impedance match with the pipe [16], uPVC adaptors matching the outer curvature of pipe were inserted between transducer head and surface of pipe. These are visible in the photograph. Coupling

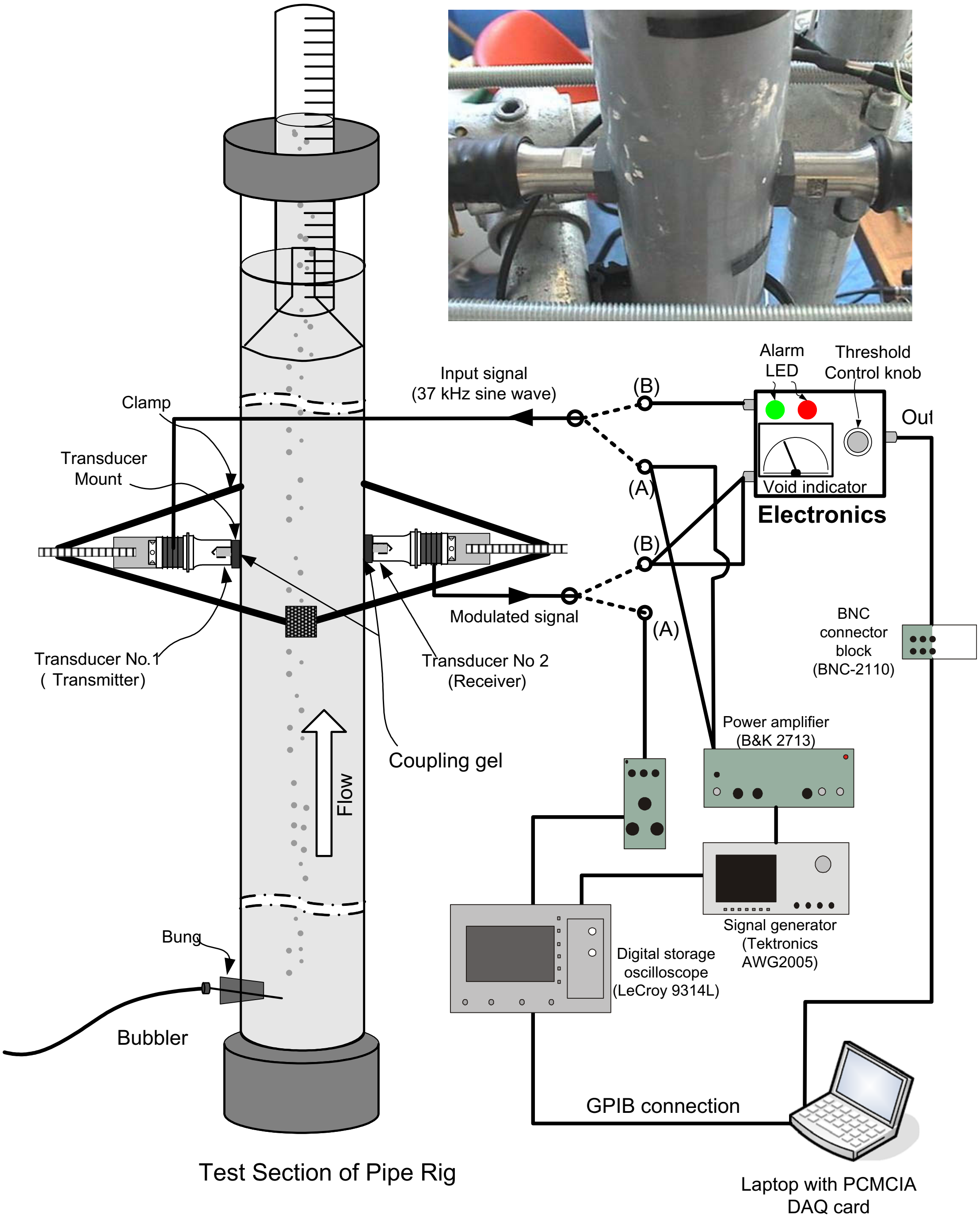

Fig. 1. Schematic (with photographic inset) of laboratory experiments with water-filled 'static' pipe rig. The rig could also be filled with slip and closed to form a pumping loop, and measurements were also made on this configuration.

gel was applied to these surfaces. In the initial experiments whilst the prototype was developed and refined, standard laboratory equipment (shown in Fig. 1, arrangement (A)) was used. Initially the 37 kHz sinusoid was generated using the signal generator (AWG2005 arbitrary waveform generator) and transmitted to the transducer via power amplifier (B&K T2713). The output signal from receiver transducer was conditioned by preamplifier (B&K T2635) and displayed simultaneously with the input signal on the oscilloscope (LeCroy 9314L). The data was sampled at 200 kHz and saved to a PC via GPIB connection. A Matlab program was used to control the equipments, data acquisition and analysis of data.

The final prototype could not make use of the commercial equipment listed above, as it would make the market device prohibitively expensive according to the market survey. Therefore the development of the prototype entailed the replacement of this equipment by a compact electronics unit (shown in Fig. 1, arrangement (B)). Not only does this unit generate the transmitted signal, and acquire the output of the ultrasonic receiver, but it also undertakes the processing and indicates, through the use of a 'traffic light' system, whether the device considers that the level of bubbles detected in the flow warrants cessation of mould filling and return of the slip to the settling tank (as indicated by the red light shown in Fig. 1) or not (as indicated by the green light shown in Fig. 1). In addition to the binary traffic light system, the unit was equipped with a needle which indicated a voltage output (labelled ‘void indicator’ on ‘B’ in Fig. 1) which is very roughly proportional to void fraction.

Fig. 2 shows the received time signal, with and without bubbles in the water in the ‘static rig’ pipe (i.e. without pumping, as in Fig. 1). In bubble-free conditions, the carrier frequency is detected

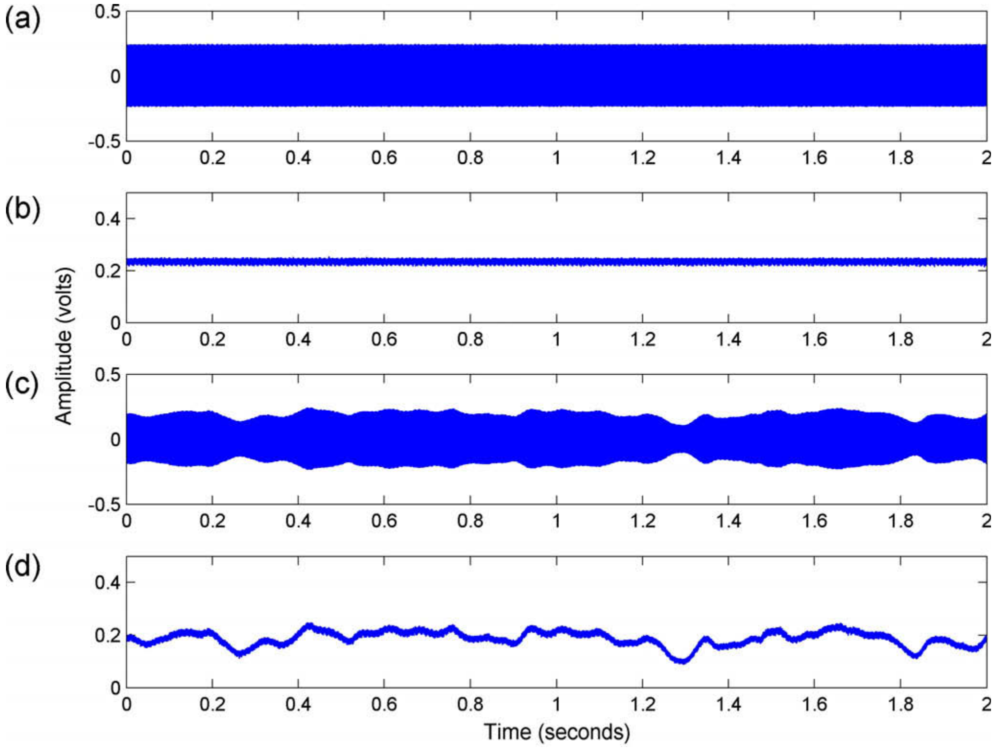

Fig. 2. Comparison of detected signals on a common time axis, with and without bubbles present, and both before and after processing for envelope features. (a) The raw 37 kHz signal from the receiver, when no bubbles are present. (b) The envelope calculated from the signal of part ‘a’. (c) The raw 37 kHz signal from the receiver, when bubbles are present. (d) The envelope calculated from the signal of part ‘c’.

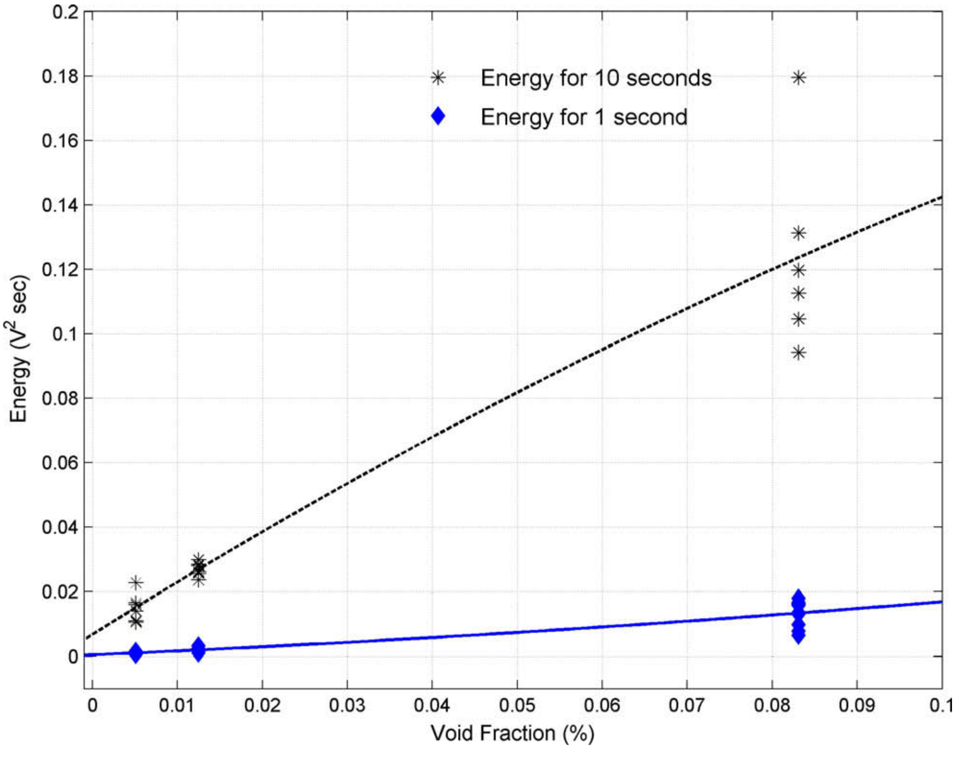

Fig. 3. Comparison of the acoustic measurement (the integration of the envelope, such that V^2 s provides an energy-like quantity) with the results of void fraction measurement, undertaken in the of water-filled static rig (the void fraction was measured by collecting of rising bubbles using measuring cylinder and flask, and correcting for the variation in rise speed with bubble size). The results are shown for integrations of the acoustic signal for both 1 s and 10 s.

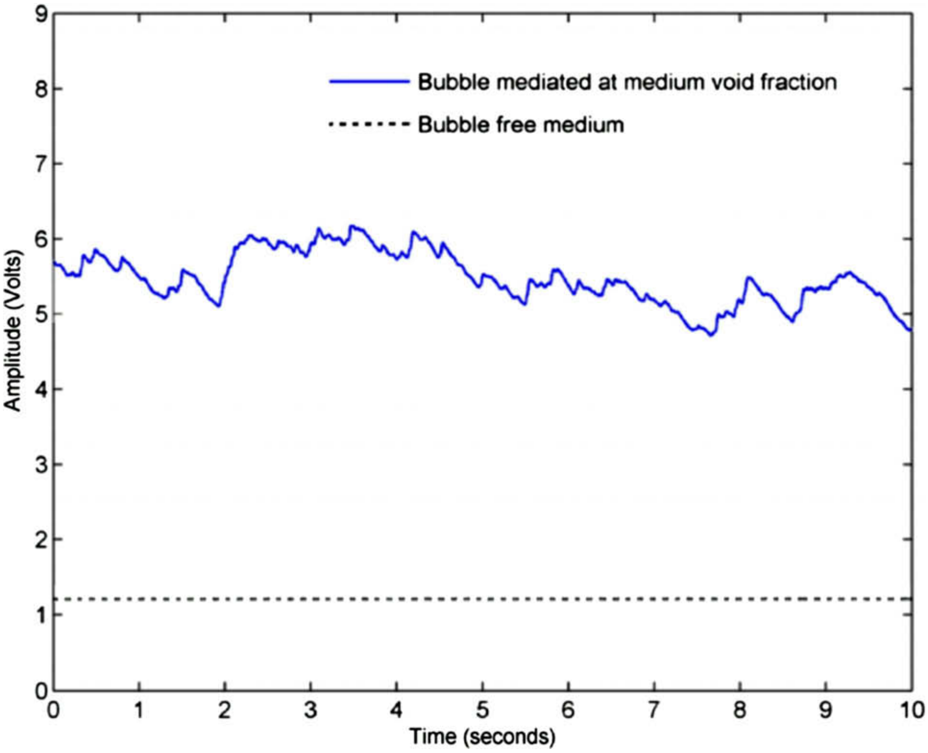

Fig. 4. The time history of the output of the prototype electronics.

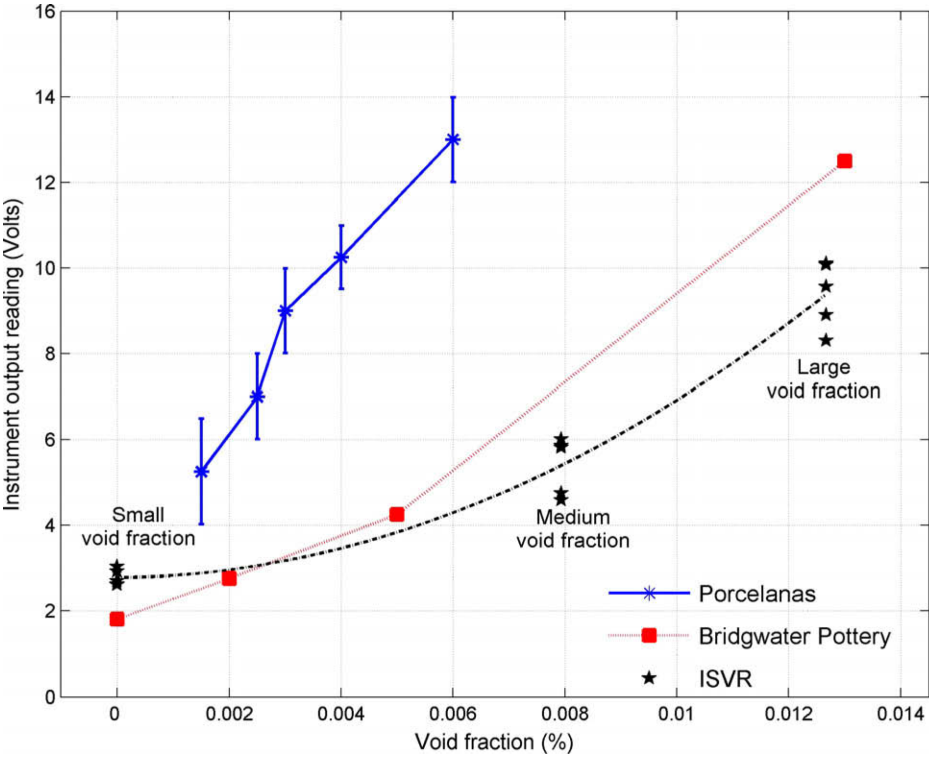

Fig. 5. Examples of factory trial results for the measurement of void measurement within pumped ceramic slip during factory operation (at Porcelanas and Bridgwater). The plot shows the calibration curves obtained using the compression test, compared against the voltage shown on the 'void indicator' needle of the finished unit ('B' in Fig. 1). Also shown are the results obtained within the test rig at ISVR in pumped slip. There are two important differences between the ISVR data and the factory data. First, unlike the factory data, the data in the rig at ISVR were not taken during real factory operational conditions. Second, unlike the factory data, the void fractions against which the ISVR data are plotted were not measured with the compression test, as this was not compatible with the ISVR rig. Therefore it would be inappropriate to compare the void fractions of the factory data on this plot with those against which the ISVR data are plotted. Consequently, the ISVR data points are labelled as having 'small', 'medium' or 'large' void fractions, and no correspondence with the void fractions on the abscissa should be inferred.

without amplitude modulation (Fig. 2a). Using the Hilbert transform, the envelope component is separated (Fig. 2b). When bubbles are introduced into the pipe, the carrier signal begins modulating (Fig. 2c), and, after processing, its envelope can be plotted as a time history (Fig. 2d). The ultrasonic estimation of void fraction was undertaken by using the square of demodulated voltage, which is plotted with measured void fraction on Fig. 3. Here the void fraction was measured at the free surface in the open static rig, where the bubbles were collected for void measurement using measuring cylinder and flask.

After testing and development on the ISVR test rig, the device was tested in the following factories: Bridgewater Pottery (Stoke-on-Trent, UK); Quality Ceramics (Arklow, Ireland); Koninklijke Sphinx (Maastricht, Netherlands); Porcelanas Bidasoa (Irun, Spain). Fig. 4 shows time history of the output signal from the finished prototype. To make this output, the time history of the envelope is divided into consecutive time bins or windows, typically of duration O (10 ms). Within each time window, the envelope modulation is squared and integrated. This provides, within each time window, a single value which is then plotted against the start-time of that window. Whilst it gives the constant voltage of a little over 1 V for the bubble-free medium, bubble-mediated changes are clear, giving in this case a time-varying signal (representing the time-varying void fraction) at a higher value, here between 4.5 V and 6 V. The results were well correlated with the compressibility results and were effective on both metal and plastic pipeline systems in factories (Fig. 5).

4. Conclusions and future work

This paper describes the conception and engineering of a sensor for use in potteries (other industries could make use of the device). Following laboratory experiments, carried out to detect bubbles in water (in both static and flowing conditions) and ceramic casting slip (flowing conditions only), an inexpensive and compact prototype detection unit was produced for use in practical trials at four ceramic factories around Europe. Whilst the earlier stages of the prototype were PC-based, a self-contained unit was engineered for the later stages. This unit was designed for ease-of-use, reliability and within the cost constraints indicated by a market survey.

The prototype was used in parallel with a compressibility test to detect the level of air contamination in the slip systems studied. The results of the two measurement systems correlated well on both metal and plastic pipeline systems in factories (Fig. 5). However, the ultrasonic unit proved to be considerably more convenient to operate, and could be used to monitor a moving slip supply continuously, which is not possible using compression techniques. Furthermore, it is non-invasive. In the factory trials, the device was able successfully to monitor a slip supply on a semi-permanent basis, automatically signalling an alarm if excessive amounts of air were present. Moreover, the factory trials also demonstrated how the device could be sequentially relocated along a pipeline to track down the source of bubble generation.

The device is now also being tested on pipework to determine whether it could be deployed to measure helium bubbles in liquid mercury, for a project where such bubbles will be introduced to reduce erosion damage in a spallation neutron source by attenuating shock waves generated when the proton beam interacts with the mercury.

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