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Research and development of radiation resistant ultrasonic sensors for quasi-image forming systems in a liquid lead-bismuth

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Abstract

The problems of the development of high-temperature radiation resistant ultrasonic sensors operating in a liquid lead-bismuth alloy are analysed. Experimental diffusion bonded bismuth titanate sensors for operation up to 450 °C were developed. For acoustical coupling with a liquid metal the sensor protector is polished or coated by a diamond like carbon layer. The developed sensors survived high irradiation doses and experienced only 4 % decrease of the transfer coefficient after gamma irradiation by 22 MGy dose. Experiments in a liquid lead-bismuth were provided, including sensor long term wettability tests, determination of ultrasound losses and measurement of the ultrasound velocity temperature dependence.

Keywords: ultrasonic sensor, high-temperature, radiation, liquid metal.

Introduction

Objective of the work was development and investigation of ultrasonic sensors suitable for operation inside the MYRRHA reactor, filled with Pb-Bi alloy. MYRRHA is an accelerator driven system with non-critical fission core providing protons and neutrons for R&D (nuclear waste transmutation, production of radioisotopes, irradiation facility, etc). The core is cooled by a liquid Pb-Bi eutectic, vessel diameter is 4 m, high 8 m. MYRRHA is the project of the Belgian Nuclear Research Centre SCK-CEN [1-3]. The final tasks are establishment of quasi-visibility in a cylindrical space of several meters in diameter and height, surveys and close-up inspections, reconstruction of images and their recognition (Fig. 1.).

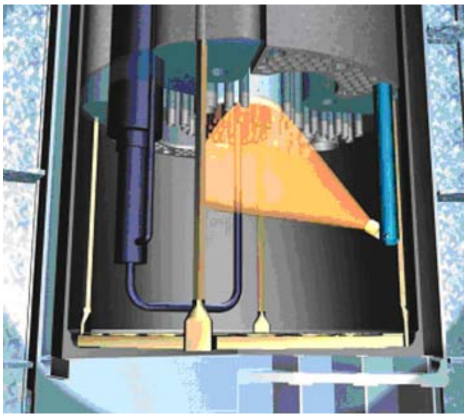


Fig. 1. **Manipulators with ultrasonic camera will provide a general overview and close-up inspections**

Such an ultrasonic viewing system is not only needed to inspect the inner vessel structures, to help in maintenance/recovery tasks, but also to monitor robotic manipulators which are required to de - and re-fuel the core.

The sensors must operate under liquid metal at high temperatures (150-450 °C) in a highly radioactive environment. They should be able transmit and receive short high frequency ultrasonic pulses in a liquid Pb-Bi alloy without cooling. Development of such sensors is a challenge as a lot of problems must be solved [4-9]. They are associated with selection of high-temperature piezoelectric material, radiation proof (γ-ray, neutron), design acoustic coupling and electrical connections inside the sensor, good performance in a pulse mode, selection of electrical cables, wetting of sensor face by a liquid metal, protection from a high pressure and corrosive lead-bismuth liquid alloy, dissolution/oxidation of structural materials, etc. There are known attempts to develop high temperature ultrasonic sensors for specific needs, e.g. for non-destructive testing of very hot steel plates, thickness measurements, welding inspection, operating in metallic melts and in ultrasonic Doppler velocimetry systems [10-17]. However, to our knowledge, at the moment are no ultrasonic sensors suitable for long-term continuous operation in the MYRRHA type environment.

General

Optimum performance of the ordinary ultrasonic sensor is obtained by minimizing ringing after the pulse response as well as the insertion losses. Usually this is achieved by a proper choice of the piezoelectric material, backing impedance, acoustic matching layers, technological procedures, excitation type, etc. In our case this choice is quite limited as, e.g., optimum acoustic matching layers and electrical matching networks inside the sensor can't be used at all at high temperatures. The necessary pulse response, ideally consisting of a single pulse, may be achieved only by a high damping of the piezoelectric material. This requirement as well as radiation resistance and other specific operation conditions limit the sensor design variety. Thus the sensor in the common case must consist of some combination of the piezoelectric element, front protector and backing (Fig. 2), and the sensor design must guarantee the necessary and reliable damping of the active element in the wide temperature range. It is necessary to emphasize that the high temperature and resistance to radiation exclude a lot of materials. For example, in this type of sensor copper can not be used. The front protector and the housing must be made of the stainless steel AISI 316, backing - metallic as well. Its geometry must be optimised in order to obtain efficient dissipation of the reflected wave.

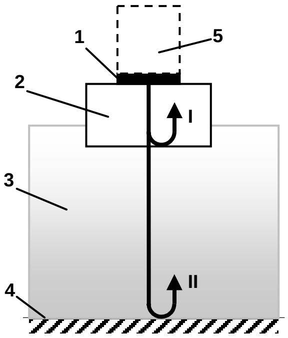


Fig. 2**. Ultrasonic sensor operating in a pulse-echo mode in a liquid metal: 1-piezoelement; 2- stainless steel protector (thin or thick membrane or a buffer rod); 3-liquid lead-bismuth; 4-reflector (non-wettable plane); 5-backing; I - reflection from the interface between the protector and a liquid metal; II - reflection from the reflector**

The main piezoelectric material for ultrasonic sensors operating up to 250 °C usually is PZT ceramics. Its Curie temperature T_C, depending on manufacturer, can reach 365°C. Typically its thickness mode electromechanical coupling coefficient k_t=0.5, but some modifications of PZT have k_t=0.7. For these high-temperature sensors piezomaterials with much higher Curie temperature are necessary. A number of such ferroelectric/piezoelectric exists. However, their piezoelectric response is smaller or even poor.

We have selected bismuth titanate material Pz46 (Ferroperm), as it exhibits a low dielectric constant, low dielectric losses and properties are stable up to 550 °C, its T_C=650 °C, k_t=0.2, however, the sensitivity is lower than PZT. Bismuth titanate belongs to the group of silenite structure-based ceramics. Its electromechanical coupling coefficient and Curie temperature vary depending on material modifications, so may be k_t =0.13÷0.23, T_C=500÷920 °C (Noliac, Seacor Piezoceramics, Piezo Technologies).

Some other materials were investigated as well, the rest were rejected after the literature analysis. Good results lithium niobate (T_C=1210 °C, k_t=0.30) showed also, but it may be unreliable due to the oxygen losses at high temperatures. The electro-acoustic efficiency of galium orthophosphate and aluminium nitride was poor. Galium orthophosphate is a new piezoelectric material for a wide range of high technology applications. It belongs to the same group as quartz; its piezoelectric constant d_{11} is only about twice than of quartz. As compared with bismuth titanate, its sensitivity is significantly lower. Aluminium nitride sensors are capable of operation at temperatures exceeding 1100 °C. This material is a strong candidate for high frequency ultrasonic sensors as the deposition of thin films is easily done. Thin aluminium nitride films were deposited on a titanium substrate, they had a rather low fundamental frequency (15 MHz), were wideband and demonstrated a good pulse response. These films were manufactured at the Dayton Research Institute, U.S.A. using chemical vapour deposition (CVD) technique. Unfortunately, due to the technological problems, such as unsatisfactory electrode and substrate adhesion, aluminium nitride at the moment can not be used.

Some other scientific achievements are known in the field of high temperature ultrasonic sensors. For example, sol-gel based technology is the novel approach to directly deposit high-temperature sensor material on e.g. steel substrates thus avoiding coupling problems (as well as in CVD technology). High Curie temperature ferroelectric crystals powders are incorporated in a sol-gel solution, which is sprayed onto substrate at room temperature, after that thermal treatment and poling succeeds. Piezoelectric combinations consist of lithium niobate, lithium tantalate, bismuth titanate and PZT powders. Two different piezoelectric materials are usually used in sol-gel solutions: one is responsible for high piezoelectricity, while the other for high Curie temperature. Unfortunately, according to the literature [18÷20] thicker piezoelectric films (e.g.>100 μm) are not dense, their piezoelectricity is low compared with that of bulk piezoceramics. More, these technologies are limited in the maximum thickness obtainable due to the build-up of internal stresses during deposition which lead to film cracking. There are no illusions about the commercialization of this scientific knowledge in near future.

Suitable commercial sensors are not available as yet. Several high temperature ultrasonic sensors for hot surfaces are supplied by companies such as Etalon, Panametrics, Ultran, Sigma transducers, etc., but standard high-temperature sensors are designed with a duty cycle in mind. Although the delay line insulates the interior of the sensors, the recommended duty cycle for surface temperatures between 90 °C and 425 °C is no more than ten seconds with the hot surface, followed by a minimum of minute of air cooling. The best sensors can operate up to 230 °C continuously.

Design of the sensor depends on its application fields. Some main realisations of the high temperature sensor for liquid metals follow from Fig. 1. Most simple is the sensor with a thin membrane. Typically, its thickness must be λ/2, what is 0.6 mm for the stainless steel AISI 316 at 5 MHz. Such sensor has some shortcomings. It is insufficiently pressure robust as the membrane is rather thin. Ultrasonic waveform in a liquid metal consists of numerous cycles and is suitable only for ranging but not for visualisation purposes (Fig. 3). The piezoceramic element is damped only moderately from one side by a thin membrane and liquid metal whose acoustic impedance is not high (18.7 MRayl), what is more than twice lower than of stainless steel (46.4 MRayl). In the case of worse wetting of tne sensor the ringing increases.

For visualization purposes backing is necessary in a sensor with a thin membrane. It is known that in the optimal case in a heavily damped sensor the backing acoustic impedance must match with active element acoustic impedance. Then the best resolution is achieved but at the expense of signal amplitude. Acoustic impedance of bismuth titanate piezoelement is 26.2 MRayl. From the radiation requirements the best material for backing is stainless steel AISI 316, so its acoustic impedance is not optimal from the point of view of acoustic performance. For visualization purposes backing is necessary in a sensor

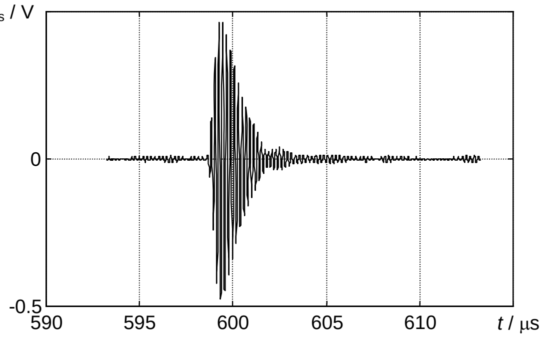


Fig. 3. **Ultrasonic waveform of the most simple sensor with a thin protector in a liquid metal**

with a thin membrane. It is known that in the optimal case in a heavily damped sensor the backing acoustic impedance must match with active element acoustic impedance. Then the best resolution is achieved but at the expense of signal amplitude. Acoustic impedance of bismuth titanate piezoelement is 26.2 MRayl. From the radiation requirements the best material for backing is the stainless steel AISI 316, so its acoustic impedance is not optimal from the point of view of acoustic performance. Another suitable metal in the sensor design is Inconel 600 but it possesses nearly similar acoustic impedance (49.5 MRayl). The sensor with a thin membrane and metallic backing, if properly bonded amongst, is characterised with an acceptable pulse response in a liquid metal (Fig. 4). Geometry of the backing body must be optimised in order to achieve maximal dissipation of energy from the piezoelement back face. Residual reverberations in a damping body are seen along the main short pulse in Fig. 4 as the signal measurements were performed at near distances. Reflector in a liquid metal was situated at 1 cm from the sensor. At longer distances these reverberations are absent and a pure pulse is observed. The problem of pressure robustness of this sensor remains but it can be solved by a proper sensor design, e.g. if backing is reliably fixed inside the sensor.

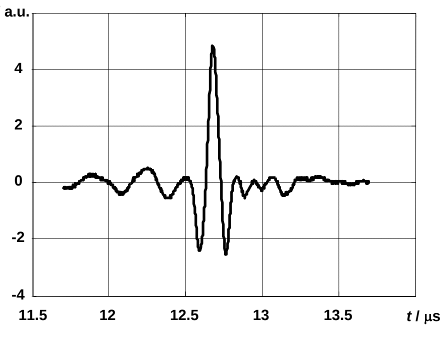


Fig. 4. **Frequency response of the sensor with a thin membrane and metallic damping body**

The buffer rod sensor is suitable for research and development experiments of sensor design, for investigations of ultrasonic properties of liquid metals, including wetting experiments, etc. This sensor possesses unique property as in a pulse-echo mode in a liquid metal two signals are observed: the first is the reflection from the interface rod tip/liquid metal and the other–from the reflector (Fig. 1). Comparative evaluation of the amplitudes of these two signals allows providing various precise quantitative measurements. The typical rod diameter in these experiments was 20 mm, length 50 mm. Both rod ends were polished, side surface - grooved. With this buffer rod different piezoelement acoustic coupling methods were investigated as well as an optimal coupling with the liquid metal was found. This buffer rod sensor is not intended for quasi-image forming systems.

The sensor with a thick protector is a modification of a buffer rod sensor, it is pressure robust. As well as in buffer rod sensor, backing is not necessary if the piezoelement is reliably bonded to the protector. The obtained pulse is sufficiently short; the advantage of the sensor is a simple design. Unfortunately, additionally some decaying pulses are observed in the time intervals which depend on the protector thickness, what is a shortcoming of such sensors (Fig. 5). These additional pulses may be reduced or eliminated in two ways. Protector layer with nonparallel surfaces may be used in order to suppress these multiple reflections inside the protector, but the structure of the ultrasonic field radiated through such protector becomes complicated. Appropriate computer generated excitation waveform may be used for the same purpose, but it is not simply made.

Bonding methods

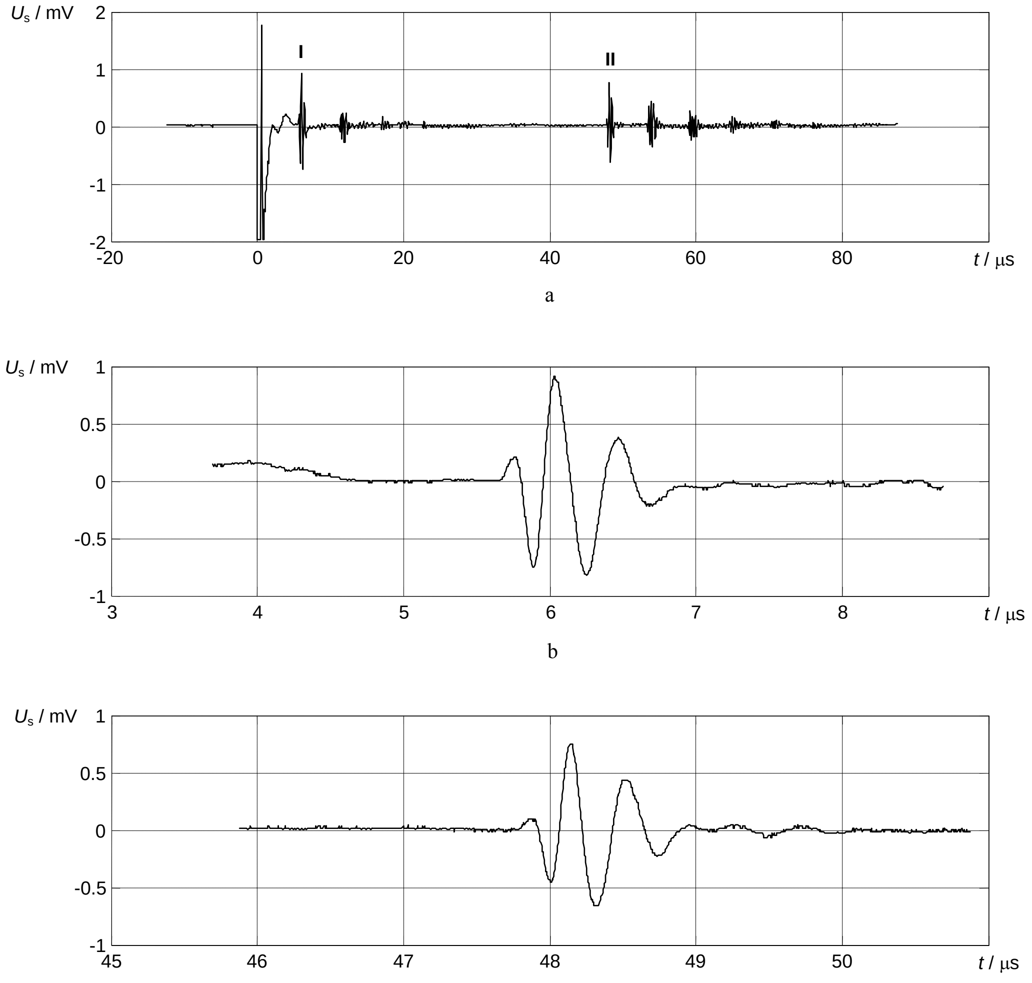
Bonding of piezoelectric element with a stainless steel protection layer and backing is crucial for high-temperature ultrasonic sensors. Three main concepts of acoustic coupling are known: dry, liquid and solid coupling.

In a dry coupling a high surface finish and a soft metal (gold) foil interlayer are required. It is impossible implement in the sensors with the λ/2 membrane, as the thin membrane would not withstand the necessary coupling pressure. Moreover, the high temperature springs necessary to create a high pressure are problematic as well.

In a liquid coupling thin acoustical membranes may be used. The liquid couplants can be divided into two groups: the couplants which are liquid at the room temperature and glass solders, which are solid at the room temperature and melt prior to operation at high temperatures. For this purpose silicone oil is widely used, which may operate successfully only up to 250°C, but it evaporates gradually and acoustic coupling is lost. Specialized high temperature couplants must be used for a short period only since they tend to dry out or solidify and no longer transmit an ultrasonic energy. Liquid couplants for continuous use at the temperatures above 350°C are difficult to find. Besides that their resistance to radiation is poor. During long lasting operation the liquid couplant may flow out of the bonding area due to vibrations of the used piezoelement.

On the contrary, the glass solders are not applicable up to 250°C. Hence, there are no radiation resistant liquid couplants in the required temperature range suitable for a long term operation.

For a solid coupling several approaches can be used: soldering, diffusion bonding, ultrasonic welding, cementing, as well as sol-gel or chemical vapour deposition (CVD) technology. The common requirement in



a

b

c

Fig. 5. **Experimental signal in a liquid Pb-Bi at the temperature °C: a-the ful signal; b-the first reflection I from the interface protector/liquid metal and the first reflection II from an aluminum plate in a liquid metal.**

a solid coupling is elimination of influence of inevitably different temperature expansion coefficients of the piezoelement, protector, backing and solid couplant, such as a solder, alumina cement, etc. This requirement excludes many solid coupling methods. For example, we have used successfully soldering of the piezoelement to a thin metallic membrane and a metallic backing with a soft solder only up to ~ 200°C. At higher temperatures (the solder Pb93Sn5Ag2 with the melting point 300°C) the piezoelement partially disbonded from the membrane and the backing after some heating/cooling cycles (20-290°C). The commercially available 88Au12Ge solder alloy is known [21], which is ductile and suitable for the joints comprised of dissimilar materials having different thermal expansion properties. Unfortunately, its melting point is too low, only 356°C. The only practically available solder around 450°C is the Au82In18 with the melting temperature 451°C. However, it is extremely hard has a poor elasticity and a low ductility. Hence, this solder is not suitable for such applications. Cementing with the alumina cement is rather simple, but according to the literature data it is not reliable as the lithium niobate array disbonded from the metallic substrate after heating to 456°C [22]. Moreover, our experiments revealed bad irradiation resistance of the alumina cement. The sol-gel or CVD technology is used only for thin high frequency sensors. These technologies are being developed recently and are far from a final solution. There is no information about the adherence quality, influence of thermal shocks, etc. It is known that CVD AlN films disbonded from Ti substrate upon reheating and that the stainless steel substrate is also not suitable.

Dry coupling was successfully used by us up to 450°C only in a waveguide type sensor. It was obtained by pressing the 5 MHz, 12.7 mm diameter bismuth titanate piezoelement to the stainless steel waveguide by an external force. Electrodes of the piezoelement were electroplated up to 2 µm thickness and the contacting surfaces of the piezoelement and the waveguide were flat and polished up to the optical quality. The gold foil was 10-20 µm thick and annealed. The pressure was increased gradually until maximal damping of the piezoelement was observed and the duration of the ultrasonic signal did not shortened further. Fig. 6 shows the ultrasonic pulse reflected from the free end of the buffer rod in air at 300°C. This is a typical ultrasonic signal in the temperature range 20-450°C.

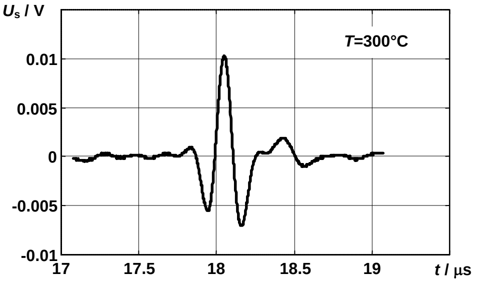


Fig. 6. **Ultrasonic pulse reflected from the free end of the buffer rod in air at 300°C (dry coupling).**

Diffusion bonding

After numerous experiments with various bonding methods it was decided that the diffusion bonding might be the best solution for sensors with thin membranes. Gold-to-gold is a most suitable pair for a diffusion bonding, because gold is plastic and ductile, especially at elevated temperatures. The joints are reliable even when the joined materials have different thermal expansion coefficients. In a thermocompressive bonding the main parameters affecting the bonding quality are: the surface quality, the bonding temperature, pressure and duration, protective inert atmosphere or vacuum. Gold-to-gold diffusion bonding may be provided in an open air as well. In order to estimate the quality of the bonding, the first experiments were carried out with a few pairs of stainless steel rods of the same 12 mm diameter. The bonded surfaces were polished up to optical quality and gold plated with chromium or nickel interlayers. Sputtering technology was rejected as tensile strength tests revealed unsufficient adhesion of these coatings. So a special gold (99.99 %) electroplating technology for stainless steels with nickel interlayer developed in Lithuania was applied. The bonding temperature reached 400°C, the pressure 300 MPa. The Ar protective atmosphere was used. The annealed gold foil interlayer made easier bonding of these rather large surfaces. Coplanarity of the bonded surfaces and pressure uniformity must be guaranteed. The bonded stainless steel rods are shown in Fig. 7. The bonding quality was tested by means of ultrasound in a through-transmission mode. For that purpose an additional uniform reference rod of the same dimensions was manufactured and the test results of ultrasound transmission were compared.

Fig. 7. **The bonded stainless steel rods**

Metallographic picture of the joined zone at the edge of the rod (see the polished area, Fig. 7) revealed that three gold layers with two interfaces are bonded perfectly, there are no bonding defects. Only surface irregularities are seen.

The tensile strength tests of the bond were provided. They revealed the bond strength 60 MPa (Fig. 8), while the tensile strength of the pure gold is 120 MPa.

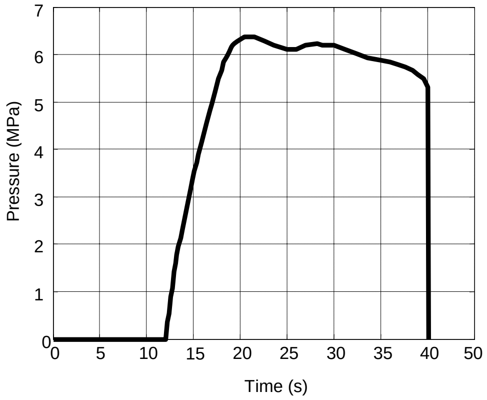


Fig. 8. **The tensile strength diagram of the bonded rods**

The bonded specimen broke partially due to the separation of the electroplated nickel interlayer from the stainless steel rod, partially gold layers separated from the nickel and which were bonded amongst. These tests demonstrated as well insufficient alignment of the interfaces and disclosed non-bonded areas (Fig. 9, dark zone at the left).

Fig. 9. **View of the broken bond after the tensile tests**

As it was pointed earlier, the sensor with a thin membrane and damping body possesses the best frequency response for visualisation purposes. Thus in this sensor the piezoelement must be bonded both to the protector and the backing. This bonding process is more sophisticated as two pairs of interfaces must be bonded, at the same time or in turn. Moreover, significantly lower temperatures and pressures must be applied due to the danger of piezoelement fracture and development of local stresses and cracks just under the surface of the piezoelement at the temperatures exceeding half the Curie temperature.

Temperatures and pressures may be lowered if a high surface quality is achieved. The overall flatness of polished gold coated protector evaluated by Newton`s rings method was less than $\lambda_{opt}/2$ (Fig. 10).

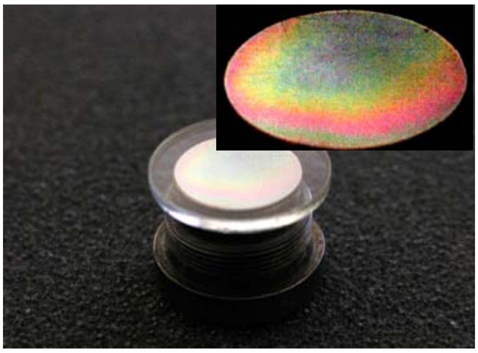


Fig. 10. **Determination of overall flatness of the sensor protector**

The remained roughness of the polished surfaces was additionally measured by the "QUEFNT" atomic force microscope. In a surface segment of 40×40 µm it was within 200 nm.

Experiments demonstrating the ability to create good metallic diffusion bonds between PZT piezoceramics and stainless steel for piezoelectric motors are known [23]. It was demonstrated how critical the flatness of the samples and the fixturing is to the ability to obtain good bonding throughout the sample.

The first bonding experiments we carried out with a sensor having a thick membrane. Such a choice has some advantages from the technological point of view because only one surface of the piezoelement must be bonded. In this configuration ultrasonic monitoring of the necessary pressure and bonding process was used in order to optimize it. For that purpose the same piezoelement was exploited for transmission and reception of ultrasonic waves, and the reflection from the outer surface of the thick protector was observed during the bonding process. This reflection can be seen if the protector is not thinner than 4-5 mm. A good bonding means that the amplitude of this signal is close to the theoretical value, the pulse is short and consists of not more than 1.5 period. Pressure was decreased more than twice in this experiment. The quality of the diffusion bonding was tested with a miniature wideband ultrasonic probe. Two testing configurations are possible: the sensor is scanned by a miniature probe from the piezoelement or from the protector side.

The thick protector with diffusion bonded bismuth titanate piezoelement is shown in Fig. 11. The golden foil strip for the interconnections inside the sensor is diffusion bonded to the hot electrode of the piezoelement as well. The experimental signal of this sensor was shown earlier in Fig. 5.

Diffusion bonding of monolithic sensor consisting of membrane, piezoelement and damping body (Fig. 12) is more complicated and higher precision is required especially for piezoelement surfaces.

Besides that in this bonding configuration ultrasonic monitoring of the process is practically impossible.

Fig. 11. **Thick protector with the diffusion bonded piezoelement and gold foil strip**

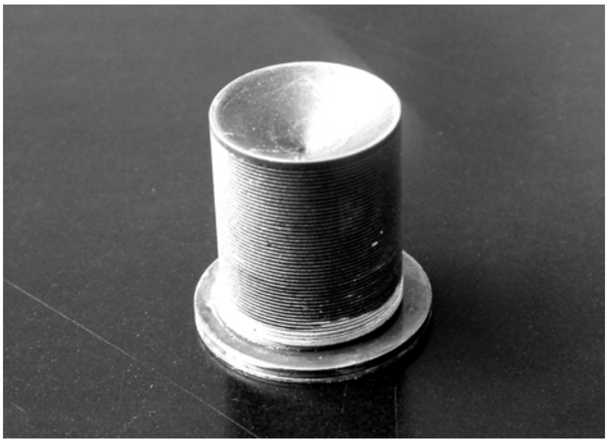


Fig. 12. **Monolithic design of the sensor**

In order to suppress multiple reflections of ultrasonic waves inside the backing, the end surface of it is concave and cylindrical surface has special grooves. So no reflection can be seen from the backing side of the piezoelement. The pulse response of this sensor was shown in Fig. 4. Unfortunately, technological problems associated with sometimes observed cracks and delaminations in bismuth titanate still remain, so the sensor technology must be improved. Fig.13 shows typical delamination in bismuth titanate when the pressure or/and the temperature were too large for this piezoelectric material. In a tensile strength

Fig. 13. **Delaminations in bismuth titanate piezomaterial**

test the electrode separated from the bulk piezomaterial with a thin layer of it. One possible solution of this problem may be application of thermosonic diffusion bonding. It is based on an additional use of an ultrasonic energy, what allows reduce the necessary pressure, temperature and duration of bonding. For example, the conventional thermocompression bonding of gold wires in electronics requires 300-500°C, while the thermosonic – only 100-240°C. These experiments are initiated now.

Experiments

Depending on the goal, experiments in a liquid Pb-Bi were performed with all three versions of the sensor: with a thin protector (with and without backing), with a thick protector and a buffer rod sensor. There were four major directions of research:

- investigations of sensor acoustic coupling with the aggressive media, e.g., sensor front face wettability tests;
- irradiation tests;
- determination of ultrasound losses;
- measurement of ultrasound velocity temperature dependence.

In order to get a reliable transmission of ultrasonic waves from the sensor to liquid metal and back the most essential property of the sensor is wettability by the liquid metal. Wettability due to a large surface tension of the liquid metal is a serious problem. An oxide film on the surface of a liquid metal may also drastically decrease the acoustic contact during the immersion of the sensor into a liquid alloy. Therefore, a special immersion procedure was developed protecting the tip of the waveguide from oxides. The good wetting was achieved by electroplating the sensor protector by the Sn-Bi alloy, but it dissolves gradually. This method was used successfully in experiments lasting up to 100 h. A number of various coatings of the rod sensor front face were investigated: Pt, Al, IrO, Mo, Ta and DLC. Before the coating the rod tip was polished precisely. Many of them demonstrated poor or better but not stable acoustic coupling. Satisfactory results demonstrated Mo coatings, good long-term performance showed DLC coatings and polished to the optical quality AISI 316 surfaces.

Quantitative wetting experiments are feasible with the waveguide sensor or the sensor with a thick protector. The quality of the acoustic contact with the liquid Pb-Bi was estimated from the ratio of the amplitudes of the signals, reflected by the non-wettable titanium reflector immersed in the liquid Pb-Bi alloy and by the waveguide tip (Fig. 2). The results of a typical wetting experiment at the 200°C are shown in Fig. 14.

From the acoustical impedances of stainless steel and liquid lead-bismuth follows that in the case of ideal wetting this ratio should be 1.9. During the experiment we have obtained k_w=1.1-1.3. It was assumed that smaller values of this coefficient are because not the whole face surface of the sensor but only some parts of it is wetted. More clearly the wetting efficiency may be evaluated by a coefficient $S_w=A_w/A$, where A_w is the total area of the wetted parts, A is the area of the front surface of the sensor, corresponding to the piezoelement diameter. In Fig. 14 the initial wetting is characterized as S_w=0.88, the minimal and maximal values are 0.84 and 0.90. Consequently, this example shows that a stable long lasting acoustic coupling may be achieved.

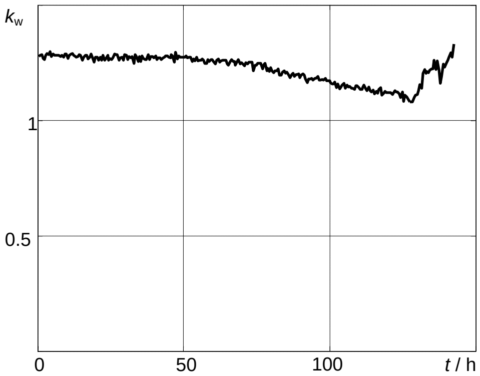


Fig. 14. **Time variation of the ratio $k_w=U_2/U_1$ in a liquid lead-bismuth alloy, where U_1 is the amplitude of the signal reflected by the sensor waveguide tip, U_2 is the amplitude of the signal received from the reflector in the Pb-Bi.**

Similar experiments were performed with sensors of different designs, such as with polished or DLC coated front surfaces, at various temperatures up to 400°C. The longest experiment lasted 978 hours. These experiments revealed that at moderate temperatures (÷300 °C) the wetting coefficient in long lasting experiments was rather stable (e.g S_w ~0.6). Microscopic examination of the sensor front surfaces, both polished or DLC coated, showed only minor corrosion indications. Experiments were provided with minor protective atmosphere and without an oxygen control. For better wetting it is necessary to supply e.g. Ar gas to prevent oxidation of liquid lead-bismuth because creation of oxides disturbs the propagation of ultrasound.

Irradiation tests were provided with the waveguide sensors especially made for these experiments (Fig. 15). Measurements were performed on-line during the cycle of the irradiation, and the reflection of ultrasonic pulse from the waveguide tip was registered. The underwater gamma irradiation facility BRIGITTE is situated in the side pool of main BR2 reactor pool at SCK-CEN. PZT, lithium niobate, galium orthophosphate, bismuth titanate and aluminum nitride piezomaterials received different γ total doses up to 33 MGy. Irradiation by neutrons was performed for

Fig. 15. **Stainless steel wave guide with the bonded piezoelement after the γ-irradiation**

aluminium nitride sensors so far. The total thermal neutron dose was 8.8×10^{18} n/cm^2. The bismuth titanate sensor experienced only a modest 4 % saturated decrease of transfer coefficient.

Determination of losses in a liquid Pb-Bi was carried out at various distances up to nearly 2 m, and the results were compared with similar experiments in water. Sensor with a thin protector was used. The measurements were carried out using the cylindrical Ø 20 mm Al reflector, which was positioned at different distances from the sensor: 12, 23, 50, 64 and 76 cm. Additionally, a planar reflector was placed at 78 cm. It was determined, that the total signal losses, including attenuation and diffraction, at 2 m distance were 42 dB for water and 31 dB for Pb-Bi alloy. That means that ultrasonic signal propagates in this liquid metal with lower losses than in water. Experiments were performed at 160 °C.

The developed sensors were exploited for ultrasound velocity measurements in the liquid Pb-Bi (50-50 %) alloy. Ultrasound velocity dependence on a temperature is necessary for ranging and imaging purposes. This dependence was measured using a pulse-echo technique and a waveguide sensor with a dry coupling. The actual length of the measurement cell was 24.956 mm at 15.99 °C. The thermal expansion of the measurement cell was evaluated. Measurements were provided every 10 °C. It was found that the velocity temperature dependence in the range 160÷460°C is nearly linear. The ultrasound velocity value at 300°C is 1740 m/s and its temperature coefficient is – 0.21 m/s×deg. The results obtained in the same specimen at the same temperature with a time interval of a few days were identical within ±0.7 m/s [4].

First versions of the sensor

Two types of diffusion bonded sensors for visualisation purposes were designed, investigated and manufactured: with a thick protector and with λ/2 membrane (with or without backing). Their housing is made of stainless steel and is laser welded. Inner interconnections are made of the gold foil strips. Sensors are supplied with a high-temperature mineral cable (THERMOCOAX, France). Their diameter is 22 mm. (Fig. 16).

Conclusion and further work

Prototype radiation resistant high-temperature ultrasonic sensors were developed using the gold-to-gold diffusion bonding method. The sensors demonstrated a good long time performance in a liquid lead-bismuth alloy. Their pulse response is suitable for visualisation purposes. For ranging and imaging possibilities ultrasound velocity dependence on temperature and losses were determined in this liquid alloy. Bonding technology must be improved in order to get a higher reliability. It is intended to use thermosonic bonding instead of a thermocompression bonding.

a

b

Fig. 16. **First versions of diffusion bonded sensors: a-polished sensor with a thick protector; b-DLC coated sensor with a λ/2 membrane.**

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Jonizuojančiajai spinduliuotei atsparių ultragarsinių keitiklių, skirtų vizualizacijai skysto švino bismuto lydinio aplinkoje, kūrimas ir tyrimas

Reziumė

Nagrinėjamos problemos, susijusios su aukštatemperatūrių ultragarsinių keitiklių, veikiančių skysto metalo aplinkoje, kūrimu. Sukurti pirmieji ultragarsiniai keitikliai, veikiantys iki 450 °C temperatūros. Keitiklio viduje panaudota pjezoelemento ir metalo difuzinio sujungimo technologija. Akustiniam kontaktui su skystu metalu užtikrinti ir apsaugoti nuo korozijos keitiklio nerūdijančio plieno protektorius padengtas deimanto struktūros anglies sluoksniu. Sukurtieji keitikliai atsparūs didelėms gama spinduliuotės dozėms, siekiančioms 22 MGy. Atlikti ilgalaikiai keitiklio drėkinimo eksperimentai skystame švino ir bismuto lydinyje. Nustatyta ultragarso greičio temperatūrinė priklausomybė ir signalo nuostoliai skystame metale. Ultragarsinio keitiklio nerūdijančio plieno korpusas suvirintas lazeriu. Panaudotas aukštatemperatūris aukštojo dažnio kabelis su mineraline izoliacija.

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