

High temperature ultrasonic transducers: review

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Abstract

The problems of development of high-temperature ultrasonic transducers for modern science and technology applications are analysed. More than 10 piezoelectric materials suitable for operation at high temperatures are overviewed. It is shown that bismuth titanate based piezoelectric elements are most promising. Bonding methods of piezoelectric elements to a protector and backing are discussed. Thermosonic gold-to-gold bonding is most modern and possesses unique features. Our achievements in this field are analysed in the context of world progress. In sol-gel and chemical vapour deposition technology bonding and sometimes coupling problems are avoided at all. Design peculiarities of the transducers are reviewed. Commercial sensors in the meanwhile are often characterized with poor performance in extreme conditions.

In conclusions recommendations for high temperature ultrasonic transducers design are formulated.

Keywords: ultrasonic transducer, high temperature transducers, piezoelectrics, bonding, acoustic coupling.

Introduction

Modern science and technology very often demand application of high temperature ultrasonic transducers. Two major requirements for ultrasonic measurements at high temperatures are: to possess transducers that themselves withstand the heat and to also to provide a suitable acoustic coupling between the sensitive element of the transducer and the object under a test.

First ultrasonic transducers for use at high temperatures have been described in literature since 1940 and were widely employed within nuclear power industries for a big variety of non-destructive testing (NDT) and non-destructive evaluation (NDE) applications. Ultrasonic methods are effective means to inspect the internal state of materials, both in laboratories and industrial environments. For instance, ultrasonic NDT of steel components in industrial plants is performed at temperatures up to 400°C. Progress in material science has enabled to create a lot of advanced composites and engineering materials. Ultrasonic monitoring is often used to characterize these materials during manufacturing at elevated temperatures, for example, during polymer extrusion and curing processes of graphite/epoxy composites. Ultrasound Doppler velocimetry of hot melts flows is an important field of high temperature ultrasonic transducers applications. Until now, no velocity measuring technique for liquid metals flows is available commercially. Apart from high temperatures, wetting and corrosiveness are the main difficulties of the measurements in liquid metals. Wetting behaviour can be evaluated by means of ultrasonic technique. In this case a positive feature is ultrasound propagation in liquid metals without significant attenuation. Therefore, ultrasonic methods are promising for on-line monitoring of molten metal properties. Possibility of ultrasonic ranging and imaging in liquid metals is very important for establishment of quasi-visibility inside modern reactors, cooled by Pb/Bi alloy [1-3]. Space exploration technology challenge requires new actuator and sensor designs. For example, there are no commercially available piezoelectric rotors for manipulators, operating in Venus. Its

environment represents several extremes: high temperatures (460 °C), high pressure (9 MPa) and corrosiveness [4, 5].

Poor performance of commercial sensors at elevated temperatures is still a significant obstacle in many applications. This problem is associated with different thermal expansion of various sensor elements.

This review is devoted to a detailed survey of development and achievements in the field of ultrasonic high temperature transducers.

Overview of piezoelectric elements suitable for high temperature conditions

There are many ferroelectric/piezoelectric materials that possess high Curie temperatures however the piezoelectric response of these materials is generally low in comparison to PZT ceramics. We shall overview the following materials: lead zirconate-titanate PZT, modified PZT, bismuth titanate $\text{Bi}_4\text{Ti}_3\text{O}_{12}$, modified bismuth titanate, lithium niobate LiNbO_3 , LNN based on LiNbO_3 , lead metaniobate PbNb_2O_6 , modified lead titanate, modified lead metaniobate, gallium orthophosphate GaPO_4 , aluminum nitride AlN , BMT-PT, BS-PT, sol-gel sprayed films, as well as some commercially available high temperature ultrasonic transducers.

The lead zirconate-titanate PZT is the main widely used piezoelectric material in the lower temperature range: the Curie temperature $T_C=350^\circ\text{C}$; the recommended operation temperatures are up to $T_0=150\div 200^\circ\text{C}$; the thickness mode coupling coefficient $k_t=0.5$. Some modifications of PZT have somewhat higher Curie temperature, e.g. PZT-5a $T_C=365^\circ\text{C}$, $k_t=0.45$ [6, 7]. The piezocomposite PZT may be used up to 180°C [8].

The bismuth titanate $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ has a very high Curie temperature and consequently operating temperatures. This material exhibits a low dielectric constant, low dielectric losses and properties are stable up to very high temperatures. Sensitivity is lower than of the PZT. The bismuth titanate belongs to the group of sillenite structure-

based ceramics ($\text{Bi}_{12}\text{MO}_2$, where $M=\text{Si, Ge, Ti}$). The features of some commercially available materials are the following:

Pz45 (Ferroperm), $T_C=500^\circ\text{C}$, $k_t=0.13$ [9,10];

Pz46 (Ferroperm), $T_C=650^\circ\text{C}$, $T_0=500\pm 550^\circ\text{C}$, $k_t=0.20$ [9,10];

B8613 (Noliac), $T_C=\text{N.A.}$, $T_0=500^\circ\text{C}$, $k_t=0.23$ [4];

PzS90 (Seacor Piezoceramics), $T_C=670^\circ\text{C}$, $T_0=500^\circ\text{C}$ [11];

PzS96 (Seacor Piezoceramics), $T_C=920^\circ\text{C}$, $T_0=700^\circ\text{C}$ [11].

The modified bismuth titanate Kezite K15 (Piezo Technologies, Keramos) is a revolutionary piezoelectric ceramic developed for high temperature applications previously not possible. It has an extreme piezoelectric stability, its $T_C=600^\circ\text{C}$, $k_t=0.15$ [12, 13].

Lithium niobate LiNbO_3 has the Curie temperature 1210°C , $k_t=0.30$ [7]. Unfortunately, at 600°C lithium niobate starts to lose oxygen to the environment, what makes it impossible to use [14].

Lithium niobate 1-3 connectivity composite material is made with pillars of Z-cut crystal in a matrix of alumina cement Al_2O_3 (Cotronics Resbond 989). The composite material produced shorter pulses than a monolithic plate of lithium niobate, it can operate at temperatures above 400°C [15].

The LNN is a piezoceramic newly developed by Ferroperm based on LiNbO_3 . The working temperature is a little higher than LiNbO_3 ($T_0=650^\circ\text{C}$), sensitivity is higher as well; the pulse shape improves at high temperatures [16].

The lead metaniobate PbNb_2O_6 possesses the high Curie temperature of 540°C , a low mechanical quality factor and is suitable as a sensitive element of a sensor. However, it is difficult to fabricate dense PbNb_2O_6 ceramics that have good piezoelectric properties. Ceramics with a high density and a high piezoelectric effect were fabricated by adding various elements such as Mn and Ca to PbNb_2O_6 and by examining the sintering process [17]. The high Curie temperature warrants the use of PbNb_2O_6 up to 300°C . This piezoelectric ceramic material has a good thermal shock resistance [18]. The thickness mode coupling coefficient is $k_t=0.30$ [6].

The modified lead titanate Pz32 (Ferroperm) has the Curie temperature 400°C , $k_t=0.43$, the operating temperature at the moment is non-available [9]. It has high mode cancellation compared to other solid solution piezoelectric materials such as zirconium titanates.

The modified lead metaniobate K-81, K-83 (Keramos) has $T_C=570^\circ\text{C}$, $k_t=0.41$ and is intended for high temperature applications up to 300°C [7, 13].

Galium orthophosphate GaPO_4 is a new piezoelectric material for a wide range of high technology applications. The sensitivity and a high thermal stability make it a very attractive choice for a wide range of uncooled high temperature applications up to 900°C . GaPO_4 belongs to the same point group as quartz. Up to 700°C the piezoelectric constant d_{11} shows no measurable deviation from its room temperature value which is about twice that of quartz [19]. As compared with bismuth titanate its sensitivity is significantly lower.

Aluminum nitride AlN polycrystalline ceramic film has no known Curie point. It is similar to quartz in that it is structurally piezoelectric and does not require poling. AlN ultrasonic transducers are capable of operation at temperatures exceeding 1100°C . This material is a strong candidate for high frequency ultrasonic transducers as the deposition of very thin films is easily done. To date, ultrasonic transducers in the 10 – 100 MHz range have been produced and tested [20]. Chemical vapor deposition (CVD) technique was developed to produce AlN layers on a suitable substrate. A good adherence is achieved on tungsten carbide and graphite [21]. The substrate must be electrically conductive [22]. Sensitivity of AlN is significantly lower than of the bismuth titanate.

A more detailed information about piezomaterials BMT-PT and BS-PT (TRS Technologies Inc) is not available. It is known, that BMT-PT is characterised by $T_C=450^\circ\text{C}$, $k_t=0.44$, while BS-PT by $T_C=400^\circ\text{C}$, $k_t=0.47$. Their piezoelectric activity is of the same order as PZT, while mechanical and electrical losses are about an order of magnitude higher. Dependencies presented in [4, 5] show their stable operation up to the Curie temperature.

The sol-gel based technology is a novel approach to deposit high-temperature ultrasonic transducer directly on steel substrates thus avoiding acoustic coupling problems. High Curie temperature ferroelectric crystals powders are incorporated in a sol-gel solution, which is sprayed onto the substrate at a room temperature. Multilayer coatings are used. After the thermal treatment, poling is performed using a corona discharge method. The layers obtained are up to $200\ \mu\text{m}$, typical resonance frequencies are in the range 3.3-30 MHz, the working temperature is up to 500°C . As a rule, two different piezoelectric materials are used in sol-gel solutions: one is responsible for high piezoelectricity, while another for a high Curie temperature. The typical piezoelectric combinations are the following: $\text{LiNbO}_3/\text{LiTaO}_3$ [23, 24], PZT/ LiTaO_3 [25], $\text{Bi}_4\text{Ti}_3\text{O}_{12}/\text{PZT}$ [26-28].

A new ceramics based on LiNbO_3 was fabricated using only the sol-gel based technology. It is known, that LiNbO_3 starts to lose oxygen at 600°C . An idea was to block oxygen loss by replacing some of the Li with another element. Sodium offers a better piezoelectric sensitivity than Li. An exhaustive examination of the $\text{Li}_{1-x}\text{Na}_x\text{NbO}_3$ family was provided with an atomic percentage of sodium between 0 % and 25 %. The new piezoelectric material had no oxygen loss and unique performance was achieved. But there should be no illusions about the speed with which this new scientific knowledge is likely to be commercialized [14].

Analysis of bonding techniques of piezoelectric elements to stainless steel protection layer and backing

Three different concepts of acoustic coupling at high temperatures are known [16]: dry coupling (with a high pressure); liquid (fluid) coupling; solid coupling.

In a dry coupling a high quality surface finish is required. Very small airgaps ($>0.01\ \mu\text{m}$) between a piezoelectric element and a front protective layer

substantially reduce the acoustic energy entering the material and pressures up to 300 MPa are required to expel the air even at an interface between highly polished surfaces [29]. Dry coupling is impossible with the transducer $\lambda/2$ membrane, as it can not withstand the necessary coupling pressure. Surface irregularities can cause deformations and failure of the piezoelement. Soft metal (best-gold) interlayer application makes easier the realisation of the method. It is problematic to keep constant the coupling pressure in the whole required temperature range. Over twenty years ago, Argonne National Laboratories demonstrated a soft metal foil as an effective long-term (>5 years) couplant in ultrasonic flowmeters, in experiments up to 600°C on liquid sodium [30]. It was used for clamp-on coupling of waveguide buffers. In tests of short duration a satisfactory economical couplant is an aluminum foil. For permanent coupling a gold foil is generally preferred. The foil usually is rolled and must be annealed, e.g. in an alcohol lamp flame. The optimal coupling pressure must be applied at higher temperatures as a metal foil couplant is not soft enough at a room temperature. It is reported the use of a metal foil for the coupling of ultrasonic energy between the AlN transducers and the object at elevated temperatures [31]. Dry coupling of a piezoelectric transducer with AISI-316 ultrasonic waveguide was used by us successfully and good experimental results were obtained [1, 3]. The contacting surfaces of a piezoelement and a waveguide were flat polished to the optical quality. The soft gold foil was of 99.99 % purity, 20 μm thickness. The pressure at elevated temperatures was increased gradually and measured until maximal damping of the piezoelement was observed and the duration of the ultrasonic signal did not shorten further. Perfect signals were observed up to 410°C at the pressure 60-70 MPa. Dry coupling with thin $\lambda/2$ or λ membranes was not quite successful as the membrane bent when the applied pressure increased.

In a liquid coupling thin acoustical membranes can be used. However, in spite of numerous attempts an optimal coupling fluid, concerning corrosives properties especially, is not yet available. Liquid couplants can be divided into two groups: liquid at the room temperature (silicone oil, specialized high temperature couplants, etc); glass solders, which are solid at the room temperature and melt prior to operation.

Silicone oil as a couplant was used successfully up to 250 °C [8, 18], but evaporated gradually. During a long lasting operation of the transducer the liquid couplant can flow out of the interfaces due to vibrations of a piezoelement. The chemical stability of this couplant is lost when the temperature increases for a longer period [8]. An original high temperature ultrasonic couplant is offered in [32]. It is non-flammable and consists of grease-like silicone fluid heavily filled with zinc oxide. The material was used in a nuclear reactor at radiation levels up to 50 R/h. The couplant material is capable of withstanding thermal cycling.

Specialized high temperature couplants must be used quickly since they tend to dry out or solidify and no longer transmit an ultrasonic energy. Couplant E (Ultratherm) is recommended for use between 260°C and 540°C [33]. It is

reported, that couplants for continuous use (scanning) at the temperatures above 350°C are difficult to find [34]. In a higher temperature range a solid couplant material Sono 1100 Film [35] is designed to melt at the temperature 370°C and is intended for use up to 593°C, but only for 15 seconds.

Internal acoustic coupling in principle may be accomplished with glass solder, which is fluid in the temperature range required. Low melting metals had to be excluded because they may cause short circuiting. The choices of the glass solder are made and balanced with respect to medium, chemical reactivity and compatibility, diffusion, evaporation, thermal expansion, mechanical behaviour under solidification, etc. Broad parametric studies were provided and glass solder "NaPoLi", consisting of sodium oxide, phosphor oxide and lithium oxide was selected with respect to its thermal expansion coefficient [16]. However, at higher temperatures the glass solder reacts chemically with the probe's components, except of platinum and gold. "NaPoLi" can be used as a coupling medium up to 500°C. There are known non-alkaline glass solders (LG-K1, LG-K2), which are much less chemically reactive. Unfortunately, a good adhesion between the transducer elements is an unsolved problem, as 60 % revealed adhesion problems [16]. The coupling began to emerge at a temperature 300 °C, but was subjected to changes, which ceased at around 350°C, where the probe showed its best sensitivity. It decreased later, and at 530°C no signal was observed. An unusual bonding material which is conductive and stable up to 600°F temperature and 50 R/h radiation is reported in [36]. It comprises four components: a carborane polysiloxane, a vehicle-solvent which will evaporate, a wetting agent and conductive materials. The bonding material has extremely stable ultrasonic signal properties.

In a solid coupling several approaches can be used: soldering; diffusion bonding; ultrasonic welding; cementing; epoxy bonding; sol-gel or CVD technology.

Compact and stable ultrasonic transducers for under-sodium scanner were designed using crystal and transducer face soldering technology. A solder with the melting point 220 °C was used. The transducers were tested in silicon oil at the temperature of 200 °C and were found to perform well [37]. At higher temperatures only a limited number of suitable soft solders are found. A solder should be elastic in order to compensate different temperature expansions of protector, backing and piezoelement; it should not dissolve thin electrodes of the piezoelement, etc. The commercially available 88Au-12Ge solder alloy offers the best opportunity for making dissimilar metal joints (the melting point 356 °C). The 88Au-12Ge alloy is effective in relaxing residual stresses generated during thermal processing. It is ductile and suitable for the joints comprised of dissimilar metals having different thermal expansion properties [38].

There is known a soldering (solder Ag based, m.p. ~500°C) technology for the bonding of LiNbO₃ crystal to the steel block using Ti foil interlayer [39]. In an integrated ultrasonic probe the piezoelement is welded to the rear end of the acoustic waveguide fabricated from a stainless steel foil [40].

There are known some attempts of diffusion bonding of a piezoelement. Los Alamos National Laboratory patented ultrasensitive ultrasonic transducer [41]. It is formed as a silver-to-silver diffusion bonded assembly of piezoelectric crystal, backing material and ceramic wear surface. Unfortunately, there is no information about its high temperature operation. The bonding can be carried out at the atmospheric pressure with silver sputtered pieces at temperatures over 150 °C and pressures about 50 MPa depending on samples. However, at higher temperatures ($T > 0.5 T_C$) due to local stresses the cracks in the piezoelectric ceramics originate and develop just under the surface. For PZT type ceramics it happens at the temperatures higher than 170°C [42]. Honeywell provided PZT to PZT, PZT to metal bonding for piezoelectric motors. Silver-to-silver thermocompressive bonding took place in a light vacuum environment, at the temperature <250 °C and pressures 10÷40 MPa [43]. It is emphasized that the flatness of the samples and more consistent loading are very critical. The bonded samples were ultrasonically tested and showed good-looking bonds in the ultrasonic imaging. In an indium cold welding technology only a mild pressure is required to bond an extremely thin piezoelectric crystal to a sapphire lens to achieve high ultrasonic transmission (NASA Lewis Research Center) [44]. Unfortunately, the indium melting temperature is only 156 °C.

For high temperature operation we provided bismuth titanate piezoelement bonding to the stainless steel protector using thermocompression gold-to-gold technology. The temperature was 400°C, the static pressure >200 MPa. Sometimes results were good, sometimes cracks and delaminations occur [3]. Therefore, for the diffusion bonding of piezoelectric ceramics, the temperatures and the pressures must be applied as small as possible. This became possible when we used custom built thermosonic bonding equipment. Introduction of 15 kHz vibrations into the bonding process allowed reducing temperature and pressure (230 °C, 60 MPa). Excellent results were achieved. It is necessary to emphasize, that reliable bonding happens only when a number of conditions are met in an optimized parameter window. The best combination of parameters depends on a surface treatment of bonded metals, pressure, temperature and ultrasonic power. "Trial and error" method must be used, as no modelling can lead to satisfactory results [45].

Temperature compensated piezoelectric transducer assembly for operation up to 350°C is diffusion bonded [46]. It comprises a lithium niobate piezoelectric element which is mounted on a metal base by means of a structured copper interlayer. The structured copper is a bundle of parallel filamentary strands of copper closely packed together (density ~90 %). This bundle easily accommodates different thermal expansions of the assembly.

Diffusion bonding of gold-to-gold may be made by wetting the bonding surfaces with mercury and clamping the wetted surfaces together using a moderate pressure, for example, at the temperature of 100°C for a period of 22 to 30 days [47]. The bond tensile strength exceeds the yield point of gold.

For the possibilities of ultrasonic welding Morgan Electro Ceramics [48] produces piezoelements with the

silver electrodes, polished to the surface flatness within 5 light waves, the surface finish within 0.3 µm.

1-3 connectivity composite material made of lithium niobate and cement for ultrasonic condition monitoring at elevated temperatures was bonded permanently to the test object with the same cement Al₂O₃ [15]. Unfortunately, after reaching 456°C and cooling, the composite transducer disbonded from the metal test block. However, the composite material itself was still intact and had survived. Further investigations are necessary to determine at what stage the material disbonds from the test block and whether this can be prevented. The same Al₂O₃ cement was used for bonding of LiNbO₃ crystal to the Ti block and operated up to 600°C in the experiments with liquid Sn [39].

Epoxy as a bonding material can approach the high heat resistance of phenolics, in some cases up to 300°C, without brittleness. This capability has been most fittingly used in an advanced space technology [49]. The high temperature epoxy (up to 300°C) was used to bond PbNb₂O₆ piezoelectric material and a backing material [18]. New commercially available inorganic silver epoxy Aremco-Bond™ 597 is silver filled with one part paste and is intended to use up to 927°C. It is thermally and electrically conductive; its volume resistivity is 0.0002 Ohms/cm [50]. This technology is used for the development of high temperature piezoelectric wafer sensors, based on ALN and GaPO₄. They can function in a harsh environment, e.g. for aircraft jet engine and Space-Shuttle diagnostics, space reentry vehicle thermal protection systems, etc. [51].

As there is no good solution for a high temperature ultrasonic couplant, sol-gel [23-28, 52] and chemical vapor deposition (CVD) [20-22] technologies were developed in order to avoid coupling problems. It is necessary to emphasize that these technologies are being developed newly and are far from a final solution. Until now experiments are quite limited and there is no information about the adherence quality, thermal expansion coefficients, influence of the thermal shocks, etc. It is known only that CVD AlN films disbonded from Ti substrate upon reheating [22] and that stainless steel for this purpose is not suitable.

Smart material/actuator needs in extreme environments in space are analyzed in articles [4, 5]. Future mission in Solar System will require new technology. In addition to the extreme temperatures the technology may have to withstand high radiation and corrosive environments. Motors for mobility platforms, deployment devices, actuators for sampling tools, composite manipulators, ultrasonic drills are required. They are based on high temperature piezoceramics technology.

Analysis of design of ultrasonic transducers for operation at high temperatures

Conventional ultrasonic transducers will tolerate temperatures up to approximately 50°C. At higher temperatures they will eventually suffer permanent damage due to internal disbonding caused by a thermal expansion. If the medium being tested is hotter than 50°C then high temperature transducers and special techniques should be

employed [53]. Design of high temperature ultrasonic transducers depends on a piezoelectric element selected. PZT piezoceramics with the high thickness mode coupling coefficient $k_t=0.5$ are usually restricted to maximum temperatures of $\sim 150^\circ\text{C}$ ($T_c=350^\circ\text{C}$). To overcome the thermal restrictions of PZT piezoceramics a waveguide (buffer rod) approach was developed and widely used. A big variety of waveguide design solutions is known.

An alternative approach is a direct contact transducer (with $\lambda/2$ or thicker protector, delay line, immersion, etc.) in which only a high temperature piezoelement must be used, possessing a significantly lower coupling coefficient and requiring new solutions in the transducer design.

In a waveguide design a couple of problems must be solved: acoustic coupling between the ultrasonic transducer and the waveguide, transmission of the ultrasound through the waveguide, etc. The waveguide material has to meet several requirements [8] with respect to the desired acoustic, physical (thermal shock resistance, low heat conductivity, etc.) and chemical properties (erosion and corrosion resistance). For high temperature applications, e.g. in flow measurements, the "transducer" end of a buffer waveguide is cooled down enough, so the sensor is not affected by the temperature of a hot end. Various measures are used to isolate it thermally without corrupting the signal. This relatively simple way to avoid having high temperatures at the transducer was already known to ultrasonic researchers more than 50 years ago. The rod was usually made of solid metal, sometimes steel, occasionally tungsten. The diameter is typically 15-25 mm. The external surface is threaded or grooved to break-up unwanted mode conversions. This solution works well at high frequencies, say 2-10 MHz, where the rod radius is $> 3\lambda$, λ being the ultrasound wavelength in the rod [30]. In low frequency applications (0.1-1 MHz) this requirement can not be fulfilled, and a "bundle waveguide technology" (BWT) was proposed. The waveguide consists of hundreds of wires enclosed inside a corrosion-resistant sleeve thus creating a rigid dispersion-free bundle [30]. The portion of BWT assembly within the insulated nozzle operates uncooled at the object temperature. The external part can be as long as necessary to convection-cool down to a temperature such as 100°C , well within the range of ordinary piezoelectric materials and constructions. It is reported that BWT assembly worked well at temperatures $> 200^\circ\text{C}$, later at 340°C and can withstand 450°C during extreme conditions. Wires or single fibers in the bundle can be made of steel, alumina, etc. It has been shown [8] that even a large drop in a temperature will change the transmitted pulse only insignificantly. The ratio of the fibre diameter and the frequency should be chosen in accordance to the desired acoustic transmission behaviour. For example, at the 4 MHz the maximum diameter for a steel fibre is 0.4 mm. The bundle has to be covered by a shell; the active waveguide diameter is 10 mm. For shear waves, obliquely incident, instead of using many thin wires one thin plate was proposed. The plate has certain topological features for generating reference echoes or for measuring temperatures at corresponding points. It resembles a small "hockey stick" at its abbreviation is OKS. OKS waveguides are clamp-on, and coupling is provided by the soft metal foil, which is thick enough.

An integrated ultrasonic sensor with an acoustic waveguide has been developed to overcome the thermal limitations of ultrasonic transducers. This sensor can be presently applied at maximum temperatures up to 600°C , and an extension of the temperature can be expected [40]. The acoustic waveguide is fabricated of a stainless steel foil with the thickness of 0.1 mm and the length of 200mm which is wrapped axially around a capillary tube. For measurements of the Doppler shift in the signal a dispersionless propagation of the ultrasonic wave of the frequency f inside the waveguide has to be required resulting in a restriction of the thickness d of the waveguide of the thickness d of the waveguide: $d \times f / c < 0.1$ [54]. The sound velocity c for longitudinal waves propagating in steel is about 5000 m/s. Therefore, at 4 MHz the thickness d has to be limited to a maximum of 0.125mm to provide the mono-mode behaviour of the waveguide. The waveguide has an outer diameter of 7.5mm. The waveguide is closed at the front end by means of laser beam welding. The 4MHz piezoelement is welded directly on the rear end of the waveguide. The welding technology is not disclosed. This waveguide sensor is applied to ultrasound Doppler velocimetry of liquid metal flows.

Clad steel buffer rods consisting of a core and a cladding were developed for ultrasonic monitoring of industrial process. The core is made of low ultrasonic loss steel, and the stainless steel cladding fabricated by thermal spray techniques ensures proper ultrasonic guidance [55]. These rods ranging from tens of centimeters to 1 m long can function under temperature up to 960°C . The ultrasonic transducer end of the buffer rod is air cooled so that the high performance room temperature transducers can be used. It is reported, that a short buffer rod has a length of 131 mm and a probe end diameter of 7.8 mm. This length is long enough such that the air cooling would not affect the temperature at the probing end of the clad rod, but it is short enough to have a small propagation losses and size. The air cooling pipes are installed, and the cooling keeps the temperature at the ultrasonic transducer end below 50°C . The diameter of the transducer is 6.35 mm. The signal-to-noise (SNR) is excellent. It is noted that the clad buffer rods also have superior performance in shear wave pulse echo measurements. For liquid metals, e.g. liquid aluminum at 967°C , a clad steel rod has a length of 1 m. At 5 MHz 50 dB SNR is reported.

Development of instrumentation and high temperature ultrasonic transducers has to be considered as a very difficult problem [8]. The waveguide concept enabled to apply these transducers in various fields in industry and scientific investigations: high temperature ultrasonic Doppler velocimetry, including flows of liquid metals; monitoring of polymer extrusion and thickness of metals, including liquid metals, at very high temperatures; measuring flows of hot gas at elevated pressures, as well as hot flows of various liquids, etc.

Design of a direct contact conventional ultrasonic transducer must be radically changed for operation at high temperatures. It depends on a piezoelectric element type as well as on a bonding method to the protector layer and backing.

Commercially several high-temperature ultrasonic transducers are supplied by companies, such as Etalon [56], Panametrics [33], Ultrason [57], SIGMA transducers [58], etc. All standard Panametrics high temperature transducers are designed with a duty cycle in mind. Although the delay line insulates the interior of the transducers, the recommended duty cycle for surface temperatures between 90°C and 425°C is no more than ten seconds of the contact with the hot surface (five seconds is recommended), followed by a minimum of minute of air cooling [33]. For design reasons there are no high temperature contact or immersion transducers in the standard product line. Most of SIGMA transducers can be fabricated to operate at higher temperatures than it is generally available in the market place at large. Selection of the proper materials other than epoxies and solders allows operation of these units up to 230°C continuously. Delay lines can prolong the operation a little.

The same situation is with the commercial transducers of other companies.

The internal design of commercial high temperature ultrasonic transducers is not disclosed. Ultrason [57] high temperature direct contact transducer HTC is intended for 250°C continuous operation, is supplied with a special high temperature-resistant 2 m wire terminated into a standard BNC connector. The compression-held piezoelectric assembly in a ceramic chamber is patented. The frequencies are between 0.5 and 5 MHz, the active diameters 6.3 and 12.5 mm. The transducer HTD is intended for 600°C continuous operation and is equipped with a fixed delay line. The transducers HTC and HTD are characterized by bandwidths 30÷50 % and 40÷70 % respectively (-6 dB), pulse widths 3÷6 and 2÷4 periods.

Hagisonic [59] high temperature transducers are single element contact transducers which are designed to operate up to 500°C. Their application is detection of defects at high temperature, high temperature thickness and corrosion measurements. The main features are the following: super hard alumina inner tube and matching plates, built in a high temperature resistant coaxial cable, corrosion resistant stainless steel housing. It is not clear if they are intended for continuous operation up to 500°C, or in duty a cycle mode. The pulse diagrams show that peak-to-peak voltage of the first reflection (near 10 decaying cycles) in 40 mm steel block with a gold couplant decreases nearly 10 times (from 1.7 V to 0.18 V) at 500 °C as compared to the room temperature 25°C. Endurance test under high temperature 500°C shows decrease of the signal in successive repetitions as well.

Imasonic [60] single element high temperature transducers are intended for permanent contact only at 130°C.

Panametrics [53] emphasizes that at > 50°C high temperature transducers and special test techniques should be employed. Conventional ultrasonic transducers are recommended only up to approximately 50°C, at higher temperatures they will eventually suffer permanent damage due to internal disbonding caused by thermal expansion. High temperature transducers are capable to operate up to approximately 500°C, but in a duty cycle mode. High temperature transducers fall into two categories: dual

element and delay line. In both cases the delay line material which is internal in the case of duals serves as thermal insulation between the active transducer element and the hot test surface. For design reasons, there are no high temperature contact or immersion transducers in the standard product line. The D790 (dual) series transducers can be used on surfaces as hot as 500°C. In challenging applications the delay line transducers can be used in contact with surfaces as hot as 480°C. These transducers are used with specialized high temperature couplants, which must be used quickly since they will tend to dry out or solidify and will no longer transmit ultrasonic energy.

Delay line high temperature ultrasonic transducers were developed in Federal Institute for Material Research and Testing (BAM, Germany) which could withstand continuous heating up to 250°C, above this value the probe must be cooled off after measurement. The delay line material of the probes is a special ceramic and it can be used up to 800°C [61]. New developments are carried out in transducers for continuous scanning up to 400°C and in phased arrays [34]. Novel wedge materials like polyamides can withstand extremely high temperatures and still have good ultrasonic properties. Different combinations of crystals backings and matching layers must be tested to benefit from the maximum sensitivity and the signal to noise ratio. Passive cooling is achieved by new designs of the transducer housings, while the other option is an active cooling system. An independent system can be built into the probes which provide the necessary cooling. It is emphasized that the couplants which can be used above 350°C still are a problem and improvements in this area would be welcome [34].

Optel [62] offers high temperature transducers with a water cooled delay line made from stainless steel or ceramics.

High temperature immersion type ultrasonic imaging probes are presented in [18]. These probes consist of piezoelectric transducers and short buffer rods and operate in a pulse-echo mode. The operating temperature is up to 215°C and there is no need for a cooling system. For imaging purposes one end of the clad buffer rod is attached to a piezoelectric transducer and another end is machined into a semi-spherical concave shape that provides the focus. The operating frequency is 5 MHz. The piezoelectric material is a lead niobate compound (PbNb_2O_6) that has the Curie temperature of about 540°C. This temperature warrants the use of this transducer up to 300°C. This piezoelectric ceramic material has a good thermal shock resistance. The backing is made of a solid polyamide and has acceptable ultrasonic damping. The PbNb_2O_6 piezomaterial is bonded to the backing through a high temperature epoxy, while to the rod with a thin layer of silicone oil as couplant in between.

The solid backing makes the probe broadband but ultrasonic echoes in it become the major spurious signals in the pulse-echo mode. In order to overcome such a drawback, the clad buffer rod consisting of a steel core and a stainless steel cladding is used as a delay line. It was shown that in a non-clad buffer rod may propagate many different modes, while in the clad buffer rod propagate much less modes. It is reported, that silicone oil may be

used up to 250°C with little evaporation provided a proper sealing was achieved. Ultrasonic images obtained by mechanical scanning and plotting the time delay of the echo and the echo amplitude are presented.

Several commercially available high temperature ultrasonic transducers attached to non-clad buffer rods were also investigated. IIICL (Tokyo, Japan) LiNbO₃ transducer was bonded to the 20 mm long 10 mm diameter titanium non-clad buffer rod via a brazing technique. Although this high temperature ultrasonic transducer with titanium buffer rod could function up to 450°C, the measurements were stopped at 205°C. Experiment failed as the signals in the reflection mode were good only till 140°C, in some measurement modes only till 50°C. The couplant made by the brazing technique and a poor propagation of ultrasonic waves in the non-clad titanium rod caused a low signal-to-noise ratio in this probe, while the brazing technology is not discussed. Probably the probes could be damaged. Similar observations were obtained when other commercially available probes were evaluated. The authors did not disclose the manufacturers.

Piezoelectric elements in delay line transducers may be at a lower temperature than the test object if they are intended to use in a duty cycle mode, e.g. [53], or approximately at the same high temperature in a continuous operation mode [18].

In high temperature contact or immersion transducers with thin or thicker protectors the piezoelements are always at the same high temperature, thus they must be characterized by a high Curie temperature. However, information about these transducers is quite limited.

A compact and stable ultrasonic transducer for under-sodium scanner was developed for Fast Breeder Test Reactor (FBTR), where liquid sodium is used as the coolant [37]. In the first versions of the transducer a high temperature grease was used as a couplant within the transducer as an acoustic bond between the piezoelectric crystal and the transducer face. It had a limited life around few hundred hours at the operating temperature of 200°C. In the second version the crystal was soldered to the transducer face with a high temperature solder (the melting point 220°C). The welded construction provided the helium-leak-tight integrity. The transducers were tested in silicone oil at the temperature of 200°C and were found to perform well. The transducers have only a thin membrane and the damping body is absent, thus the piezoelement is damped only by silicone oil or liquid sodium. Later the transducer was developed for continuous immersion in sodium up to a temperature of 250°C [63].

A comprehensive study of high temperature ultrasonic transducers and the development of a prototype ultrasonic probe is described in [16]. It is emphasized, that at temperatures > 200°C ultrasonic testing is based usually on electromagnetically generated ultrasound using EMAT transducers. No new way of developing piezoelectric transducers was found in literature. The developed prototype ultrasonic probe had the Inconel-600 $\lambda/2$ membrane (~0.6 mm), which is closing the front of the austenite housing and matching the acoustic impedance. No buckling of the membrane greater than 10 μm was observed, neither at a room temperature, nor at 800°C up

to 2.5-fold of the working pressure. The LiNbO₃ single crystal as well as the LNN ceramics transducer of the 20 mm diameter was coupled to the membrane by means of the glass solder NaPoLi. The damping body of the glass coated porous ceramic with the trade name "Kager 9900" was soldered to the crystal with the same glass solder. The damping body had concentric cuts at the back side in order to improve absorption. All probe parts were pressed together with a package of serially chained cup springs of Inconel 600. Alternatively, the ceramic springs demonstrated the high performance and stability of the material. The spring diameter was 46 mm and the high 23 mm. During 257 hours 8 temperature cycles up to 800°C have been performed with a spring tension of 50 N and a spring deflection of 3.5 mm. The transducer crystal was capacitively coupled to the electric circuitry by means of the 50 μm austenitic steel foil with the nickel wire welded to it. The ground connection was also capacitive. The capacitive connection means that the glass solder is not conductive. The transducer housing was laser welded and the high temperature metallic/mineral cable was used. A backwall echo from a 20 mm thick specimen shows that the ultrasonic pulse consists of ~6 cycles. High temperature experiments revealed that the glass solder approach has some problems and the coupling problem is far from the final solution.

The first version of the 1-3 connectivity composite transducer made of LiNbO₃ and high temperature alumina adhesive cement was in an experimental stage and without any housing [15]. Four designs of composite were tested: "fat pillar", "thin pillar", "fat pillar" diced at 45° and platelet. No electromechanical enhancement was found as a result of making a composite of lithium niobate. However, platelet or pillar designs operating in a thickness mode could both be used to obtain better mechanical and thermal resistance properties than the monolithic lithium niobate. The electrodes were thick film gold tracks on a ceramic alumina "array plate". The electrodes were connected either in groups or used separately. Using long wires, the array could be excited remotely. This is important for the future developments in array applications. In the experiments presented all transducer pillars were excited simultaneously. Unfortunately, the bonding problem to the test block was not solved yet. More results concerning a piezoelectric linear array structure designed to operate at temperatures up to 400°C for non-destructive testing of steel components of a hot industrial plant are provided in [64]. These arrays are used in pairs: the transmitter is a phased array producing a variable angle steered beam, and the second array is used as a receiver. The prototype devices have 64 elements. The results show the accuracy better than 1mm in finding the location of crack tips. Two cuts of lithium niobate (Y/36° rotated and Z-cut) and bismuth titanate were investigated. Good results were obtained for Y/36° cut; the coupling factor k_t values were correspondingly 0.49, 0.17 and 0.12. For high temperature tests array connection pins were clipped on with the wires and fixed with high temperature cement. As a final solution a more permanent and robust method of attachment for use at high temperatures was proposed. It could be a spot welding of the connection pins to the

mineral insulated leads and later to the head resistant coaxial cables. It is not clear if this method was realized in practice. A suitable method of array coupling to the test block is vitally important for high temperature operation. It was found that high temperature commercial couplants did not survive for long periods. The tested couplants burned or dried out while being heated to 400°C over several hours, although they were rated for from 400°C to 500°C. Some success was recorded only up to 250°C. Metal alloy brazes (e.g., gold-indium, the melting point 637°C) have been shown to transmit sound well, but braze bonds would be difficult to prepare. The authors found that the most practical couplant to reach a test block temperature of 400°C was Cotronics 989 alumina-based cement. In the first experiments they had disbonding problems [15]. After the technology improvement in the longest test the cement coupled array survived for 3 weeks at high temperature without deterioration in its performance.

For manufacturing of miniature high temperature ultrasonic transducers the lead-free thick bismuth titanate films have been successfully deposited on steel substrates of different shapes by the sol-gel spray technique [65, 66]. A short description of the film fabrication process is presented. The bismuth titanate (BIT) 80 µm powders are dispersed into BIT solution by ball milling method to achieve the gel. The final dimension of the BIT is estimated to be less than 1µm. These films are called BIT/BIT. The center frequencies on steel samples ranged from 5 to 30 MHz, their bandwidth was broad. For example, the 10 MHz ultrasonic transducer had 7 MHz bandwidth at the -6 dB level. The signals generated and received by these ultrasonic transducers had a signal to noise ratio 25 dB at 200°C in a pulse/echo mode. This transducer can be as small as 1mm in the diameter. Similar BIT/PZT films operated up to more than 400°C [67]. A top electrode was formed using silver paste thus replacing the vacuum sputtering. The electrodes performed well at high temperatures. The frequency responses of echoes at 24°C and 200°C in a steel substrate are presented. The signals practically are identical. This sample has been thermally cycled many times in a furnace, and no deterioration of the film quality has been observed. By comparing the data it was found [67], that the strength of the signal generated by the BIT/BIT thick film ultrasonic transducer on this flat steel plate was 20 dB weaker than by the BIT/PZT thick film.

Techniques for generation of ultrasound for extended periods at high temperatures are reviewed in [68]. Some transducer materials and various types of adhesive bonds were considered. Measurements of insertion losses, the transducer coupling factor and the capacitance indicated that the most successful transducer-bond combinations were PZT-5A with a solder bond for use up to 250°C, and lithium niobate with either ceramic or gold-indium bond for use up to 400°C. These three combinations all proved relatively stable for periods in excess of 400 days at high temperatures.

An ultrasonic sensor was developed, manufactured and tested for high temperature materials processing [21]. It is capable of emitting and receiving ultrasonic energy at

temperatures exceeding 900°C and pressures above 150MPa. The sensor demonstrated the capability of measuring workpiece deformation during hot isostatic pressing. Details of the sensor design, performance and coupling of the ultrasound to the workpiece are described. It is emphasized, that commercially available ultrasonic transducers are limited because of transducer materials. Electromagnetic acoustic transducer (EMAT) requires that the target material would be electrically conductive and they are very inefficient sources of ultrasonic energy. The piezoelectric material in this new transducer is aluminum nitride (AlN) film, deposited on a substrate (tungsten carbide or graphite), serving as a damping body, which is conductive and at the same time serves as a "hot" electrode. A nickel-based superalloy was selected as the housing material because of its high temperature stability and ease of machining characteristics. The rhenium spring and Inconel push rod keep the substrate pushed against the front of the precisely machined housing. Since the thermal expansion of the housing is matched by the expansion of the push rod, the spring force is constant regardless of the temperature. Achieving of electrical connections to the AlN film ("ground" electrode) required considerable research and development efforts. The film was plated with platinum, and the platinum-covered surface was mechanically forced against the inside of the sensor housing at the narrow edges, the rest front face had a hole. The sensor cable was metallic. The inner wire was welded to the push rod fitted into a hole in the substrate. To produce an ultrasonic energy, a several hundreds volts were applied across the AlN film. Two approaches were taken to produce an efficient ultrasonic coupling between the sensor and the workpiece. The first approach used the gas at elevated pressures as the coupling medium, while the second used metals or glasses as liquid couplants that melted at 700-800°C. Preliminary tests conducted at low temperatures (<100°C) over a pressure range from atmosphere to 20MPa demonstrated that ultrasound in the frequency range 15-25 MHz may propagate through the gas (e.g. Ar) at pressures above 20 MPa. Propagation distances of 100 mm were easily achieved. Test results show that the peak-to-peak signal amplitude is varying only ±25 % in the temperature range 20-800°C, but the signal to noise ratio is very poor. Liquid coupling results are not provided.

We have developed some prototype ultrasonic sensors for operation in a heavy liquid metal. The sensors are high temperature, γ and neutron radiation resistant, they can operate continuously in a liquid Pb/Bi alloy up to a temperature of 450°C [1-3]. Taking into account the design requirements it was necessary to solve the following main problems: to select suitable piezoelectric materials, to optimize the sensor design, ensure reliable acoustic coupling of a piezoelement to the protector (and the backing) in the temperature range 150- 450°C, to get a durable and stable long-term acoustic coupling of an ultrasonic sensor to the liquid metal. Most of these requirements to particular elements contradict each other and therefore some trade-off between them must be found. Some sensor types were investigated: transducers with a buffer rod (with a measurement cell), with a thin (~λ/2) or

thick ($\gg \lambda$) metallic membrane, with or without damping body. Different transducer types were optimal for solution of different tasks: ultrasound velocity measurements in a liquid Pb/Bi in a wide temperature interval, evaluation of signal losses at long distances, irradiation tests, long-lasting wetting experiments, transducer design optimization, etc. Five piezoelectric materials were investigated, including irradiation influence on a transducer operation: PZT, lithium niobate, bismuth titanate, gallium orthophosphate, aluminium nitride. The bismuth titanate transducers demonstrated best high temperature performance and radiation resistance. Dry coupling with a high pressure was used in buffer rod transducers up to 450°C. In transducers with a thin or thick protector high temperature soldering ($<300^\circ\text{C}$) or finally gold-to-gold diffusion bonding method was applied ($\pm 450^\circ\text{C}$). However, at higher temperatures soldering and diffusion bonding of piezoelements becomes problematic due to the local stresses and the possible cracks in piezoceramics leading to the separation of a thin layer of ceramics under the whole surface of the piezoelectric element together with an electrode. Thermosonic approach solved these problems and acoustically good bonds are achieved. The stainless steel housing is laser welded, the polished front face of the thick membrane is DLC coated for improvement of acoustic coupling to the liquid metal. A high temperature cable is laser welded as well. The inner "hot" connections are made by means of a narrow strip of 20 μm gold foil connected by a diffusion bonding to the gold electrode of the piezoelement and pressed to the gold coated tip of the cable.

In high temperature ultrasonic transducers mineral insulated signal transmission cables must be used, e.g. THERMOCOAX [69]. Thanks to their exceptional properties, these cables can be used in most aggressive media such as corrosive liquids or gases, under high pressure, vacuum, vibrations, in nuclear industry. They resist to a temperature up to 1200°C and can easily be welded or brazed onto any piece. They also can carry relatively high currents as well as very low signals at high frequencies. The outer diameter of the cables varies from (1 ± 0.03) mm to (6 ± 0.05) mm. The sheath may be copper plated 304 L stainless steel, stainless steel, iron-copper-stainless steel, etc. The core is made of copper, stainless steel, zirconium copper coated, etc. The insulant is a mineral powder with a low dielectric constancy, magnesia $> 94\%$, alumina 99.6%. The characteristic impedance is (50 ± 3) ohms. The line capacity depends on the insulant and the diameter of the cable and may be (110 - 350) pF/m. The line resistance is (0.13-16) Ohms/m. Attenuation in the cable is very low- only 0.5 dB/m up to 20 GHz (for $\varnothing 3$ mm cable).

Diffusion bonding was used in making piezoelectric actuators [70]. Fabrication of the actuators involves the stacking and diffusion bonding of multiple thin piezoelectric layers coated with silver electrodes. The stacked piezoelectric layers are placed in a press for the diffusion bonding process. The stack is pressed and heated at "a specified curing temperature and pressure for a specified curing time" [70]. The pressure, the temperature

and the time depend on the piezoelectric material selected. No technological details are disclosed.

The difficulties in high temperature packaging lie not only in finding materials that can survive at temperatures exceeding 300°C, but in finding compatibility between materials and assembly techniques also [71]. At high temperatures incompatibilities between materials become exaggerated. Factors such as a thermal conductivity, expansion coefficients, oxidation and diffusion become critical as operating temperatures increase and play influential roles in the selection of packaging materials. Nickel was chosen as the circuit conductor because it possesses the best balance of properties for high temperature electrical performance. The conductivity of nickel is not as high as desired, but it is adequate. Plus, it has the best resistance to oxidation. Reliable interconnection at high temperatures is very important, and wire bonding is the preferred method. The gold bonding is most suitable, so nickel conductor in the area of bond must be gold plated to avoid diffusion between dissimilar metals at high temperatures.

Modern interconnection methods developed for millimeter range wave high power Gunn devices [72] may be useful in high temperature ultrasonic transducer design. They are packed with a metal block for efficient heatsinking using a thermosonic bonding technique for a reliable assembly. The solid gold-to-gold diffusion can occur at a relatively safe temperature ($<200^\circ\text{C}$), but the bond survives and improves at higher temperatures.

Conclusions

After the analysis of the existing solutions of high temperature waveguide and direct contact ultrasonic transducers, including the achievements of the Kaunas University of Technology Ultrasound Institute, the following conclusions can be made.

1. For operation at high temperature the most suitable are piezoelectric elements made of the following materials: bismuth titanate $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ (BIT); modified BIT; lead metaniobate; BIT/PZT film (sol-gel technology).
2. For bonding of the piezoelectric elements to the protector the thermosonic gold-to-gold or silver-to-silver diffusion bonding method may be proposed. This process must be still optimized. In the BIT/PZT film approach the bonding procedure is avoided at all.
3. Design of high temperature ultrasonic transducers depends on the piezoelement and the bonding method selected. The transducer may be direct contact type, placed into Inconel-600 or stainless steel housing, with a thicker protector, thus the backing may not be necessary (protector damps the piezoelement). Electrical connection with the transducer is provided by a high temperature metallic cable, which is by a laser welded to the housing.
4. Electrical connection of the piezoelement to the cable and other inner connections may be provided by means of the soldered (e.g. solders

Au88Ge12, Au82In18) or with gold-to-gold bonded or pressed gold wires or foil strips.

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Aukštatemperatūrių ultragarsinių keitiklių apžvalga

Reziumė

Aukštatemperatūriai ultragarsiniai keitikliai šiuolaikiniame moksle ir pramonėje užima vis svarbesnę vietą. Jų reikiamų parametru įvairovė yra didžiulė ir priklauso nuo taikymo srities. Pagrindinis keitiklio elementas yra aukštatemperatūris pjezoelektrikas, skirtas darbui net iki 700 °C temperatūroje. Straipsnyje apžvelgiama per 10 pjezoelektrikų ir pateikiami svarbiausieji jų parametrai.

Esminė problema - kaip užtikrinti akustinį kontaktą tarp pjezoelektriko ir metalinio protektoriaus plačiame temperatūrų intervale. Vienas perspektyviausių metodų iš daugybės kitų yra difuzinis sujungimas aukso pagrindu, naudojant didelį slėgį, temperatūrą ir ultragarso energiją. Šis metodas taikomas ir straipsnio autorių. Aptariami autorių pasiekimai pasauliniame kontekste.

Nagrinėjamos įvairiausios tarptautinėje literatūroje skelbtos mokslininkų sukurtos keitiklių konstrukcijos: tiesioginio kontakto, bangolaidiniai, integruotieji, kompozitiniai ir kt. Parodoma, kad komerciniai žinomų pasaulinių firmų keitikliai yra ribotų charakteristikų ir dažnai netenkina eksperimentuotojų. Išvados pateikiama siūlymų dėl aukštatemperatūrių keitiklių konstrukcijos.

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