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Encapsulation of ISFET sensor chips

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Abstract

The encapsulation of ISFET sensor chips is a challenge to sensor technology and materials. Recently developed new packaging technologies enable reliable and cost effective chip encapsulation. By this, one of the serious problems, which had hindered the large-scale production and industrial application of ISFET sensors over three decades could be overcome now to a large extent. The paper reports both on ISFET encapsulation methods on laboratory level and advanced industrial production technologies. The influence of the packaging design on special applications and on the dynamic behaviour of the sensor is illustrated. Furthermore, sensor materials are characterised and various testing methods are described.

Keywords: ISFET; Chemical sensor; Encapsulation; Packaging; Steam sterilisation; Response behaviour

1. Introduction

The introduction of the ISFET principle by Bergveld around 1970 [1,2] stimulated a boom of research and development activities in this field. A considerable number of papers and theses appeared and many patents were issued since that time, dealing with theory, technology, encapsulation, instrumentation, the reference electrode and applications of ISFET sensors. In a retrospective view, Bergveld summarised what happened in the past 30 years, and he could draw the conclusion that within this period ISFET research and development made continuous progress [3]. But despite these results and their undoubted advantages, up to now ISFET sensors have been manufactured and applied in numbers hardly worth mentioning in comparison to those of the now as before dominating conventional pH glass electrodes. That can be attributed mainly to the following reasons:

- Early ISFET sensors were light sensitive, were prone to short and long-term drift and were sub-Nernstian in pH response namely, performance was not up to the glass electrode benchmark.
- Lack of equivalent all solid-state reference electrodes.
- Packaging integrity difficulties with reliable encapsulation of sensor chips.

The first problem can be regarded as satisfactorily solved. Nowadays instead of Si_3N_4, which was used preferably as membrane material in the past, some leading ISFET producers use the more suitable Ta_2O_5 along with Al_2O_3 and have succeeded in optimising semiconductor processing to such a level, that those ISFETs are comparable with glass electrodes regarding their overall performance.

In contrast to this, multiple attempts to create a long-term stable all solid-state reference electrode have not been successful. Neither on-chip preparations of miniaturised planar Ag/AgCl reference systems nor numerous suggestions to modify ISFETs to so-called REFETs [4–8] have resulted in devices with stability and performance parameters approximately comparable to those of conventional reference electrodes. So, the miniaturisation of Ag/AgCl-electrodes mainly leads to a decrease in lifetime, whereas the surface potentials of pH-insensitive and inert polymeric layers may depend on the general electrolytic constitution of the solution and the adsorption of ions onto the surface [9]. Because

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the reference electrode is an indispensable component of each potentiometric or amperometric electrochemical sensor, and as important as the ISFET indicator electrode itself, the lack of all solid-state reference electrodes has prevented large-scale introduction of ISFETs into industrial and consumer applications as a substitute for conventional pH sensors.

Concerning the encapsulation of ISFET chips, a considerable progress can be stated now. Some leading producers of electrochemical sensors have developed new ISFET fabrication technologies allowing reliable sensor packaging with reasonable technological expenditure at the mass production scale. This is an important step in the direction to widespread installation of ISFETs also in industrial plants for process control.

The present work aims to give an overview of various technologies for encapsulation and contacting of ISFETs at laboratory and industrial scale and to illustrate the complex interdependence between the encapsulation and the sensor behaviour. Furthermore, selected examples for special encapsulation-related applications of ISFETs and methods for testing the encapsulant durability under different conditions with a variety of chemical, electrochemical and physical methods are described.

2. Encapsulation technologies and materials

The design and technology for contacting and encapsulating ISFET sensors are not only crucial for a low cost production of high numbers but also for important sensor parameters such as long-term stability, response behaviour and lifetime. Well-established usual techniques for encapsulating micro electronic devices are not suited for ISFETs. The substantial difference consists in the fact that the chemically sensitive area of the sensor chip must be in direct contact with the medium to be measured whereas the chip surface in its immediate vicinity as well as the electrical connections must be protected reliably against corrosive attack from the more or less corrosive sensor environment. As indicated in Fig. 1, the sensor can be damaged by dissolution of the sensitive layer of the ISFET as well as by absorption and penetration of moisture through the encapsulation of the ISFET sensor chip which results in corrosion of the chip edges, the bonding pads and wires or of other components of the sensor.

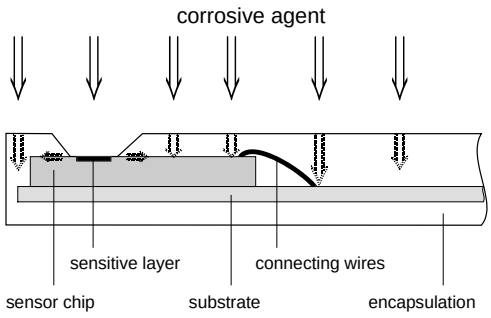

Fig. 1. Absorption and penetration of moisture through the encapsulation of an ISFET sensor chip.

Whereas the wafer preparation of ISFET sensor chips is a well defined, economical, large volume process, the encapsulation and device packaging involves usually a series of time consuming assembly steps sometimes accompanied by a high rate of failure. Therefore it is not surprising that most of the modifications of ISFET designs were directed towards a solution for the encapsulation problems [10]. Modern concepts for the encapsulation of ISFETs must at least pay regard to the following requirements:

- maximum adhesion between the encapsulant and the conductive parts of the sensor chip [11] and the membrane materials to be fixed on the gate;
- low absorption potential of the encapsulation material for the species to be measured;
- good chemical, electrical and thermal stability of the encapsulant;
- acceptable materials or design to accommodate for mechanical thermal expansion mismatch;
- defined shape of the window for the sensitive area; and
- compatibility with mass production techniques preferably at the wafer stage [12].

Because having a packaged ISFET chip is not sufficient to speak about a sensor, additional encapsulation demands toward the sensor system are formulated:

- integration of a reference system (electrode or REFET);
- biocompatibility of all sensor materials; and
- the possibility of periodical sterilisation for biological, pharmaceutical or medical applications [13].

According to these requirements a variety of different materials have been used as encapsulants for ISFETS: glass [14], epoxy resins [15,16], polyimide [10] and silicone [17]. Furthermore, sensor chips are protected by passivation layers and electrical insulation provided by the CMOS technology [18]. Obviously none of these materials can completely meet all the mentioned requirements. Regardless of the employed encapsulant material a window above the chemically sensitive area of the semiconductor chip must be provided for the technology of encapsulation. This can be achieved by means of the following methods with increasing suitability for mass production:

- encapsulation with prefabricated housings,
- embedding of chips by means of a male mould [19,20],
- photolithographic structuring or curing [21],
- capillary fill [12],
- sealing around the chemically sensitive gate area with elastomeric materials.

For electrical contacting of semiconductor chips different technologies are available within the semiconductor device industry. Most of them are highly automated and destined for the large-scale production of electronic components. Nowa-

days semiconductor-based chemical sensors must be fabricated only in low to medium numbers. For this purpose in most cases only the well-approved, very flexible ultrasonic wire bonding technique will be practicable. With regard to the encapsulation and some specific applications it is more favourable, if the contacts are on the reverse side of the sensor chip [18,22,23], but this technology requires a greater manufacturing expenditure. Alternatively a technology of epoxy multi-chip modules employing thin-film interconnections between chips on suitable substrates has been proposed [24].

2.1. Chip encapsulation at laboratory level

2.1.1. Encapsulation of standard ISFET sensor chips

At the Meinsberg Kurt-Schwabe Research Institute a number of special technologies have been developed, allowing reliable and economical contacting and encapsulation of low numbers of chemical sensor chips at laboratory level. These technologies were successfully used to manufacture prototypes of ISFET sensors for basic investigations and selected applications in medical and foodstuff research. Based on sensor chips with Si_3N_4 membranes these prototypes are commercially not available.

Fig. 2 illustrates in detail the sequence of an encapsulation technology, where the sensor chip is embedded in hot-curing epoxy resin by using prefabricated plastic moulds:

(a) The moulds for casting the sensor chips consist of a methylpentene copolymer (TPX). They are produced with injection moulding tools, cleaned and dried 1 h at 100 °C before the first casting. The ISFET connection cable consists of 14 lines of 0.14 mm Cu wires, which are glued together at the bonding side by the covering stoving lacquer. The moulds are combined in clamping devises for the preparation of 10 sensors in one passage.
(b) The sensor body is manufactured by casting of hot-curing epoxy resin Araldit CW 2122 with hardener HY 2123 (Ciba Geigy), cured 3 h at 80 °C and 2 h at 110 °C.
(c) The sensor chip is glued with epoxy resin into a cavity inside the sensor body.
(d) For electrical contacting the bonding wires are drawn directly from the bonding pads on the sensor chip to the connecting leads.
(e) The lower part of the mould is completed by another upper part, which contains a stamp for keeping the gate window free of epoxy. The contour of the stamp has to be designed carefully to prevent any undermining of the low viscous epoxy under the stamp.
(f) The second casting step completes the encapsulation of the sensor chip. The epoxy is cured under the conditions of step (b).
(g)–(h) After the mould release of the sensor the connection cable is insulated with a tube, which is glued with the sensor body resulting in a watertight connection.

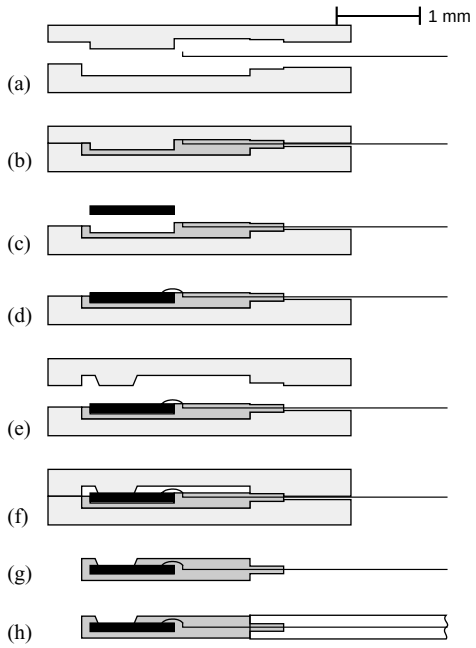

Fig. 2. Technology sequence for encapsulation of ISFET chips in laboratory scale (KSI Meinsberg).

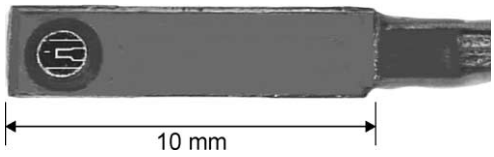

Fig. 3. Photograph of an ISFET sensor encapsulated according to Fig. 2 (KSI Meinsberg).

Fig. 3 shows a photograph of an ISFET sensor, which had been manufactured according to this technology. The outer dimensions of the sensor are essentially determined by the encapsulation. The sensitive gate area of the sensor chip is placed inside the small circular cavity in the encapsulation, the diameter of which is 1.1 mm. Fig. 4 shows a micrograph across the measuring cavity. It illustrates the stable adhesion

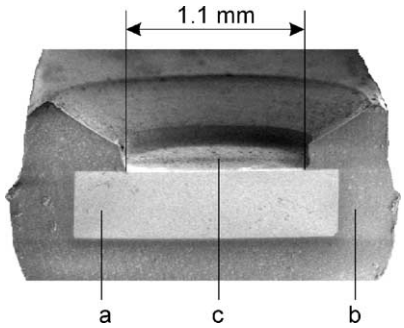

Fig. 4. Micrograph of an ISFET sensor across the measuring cavity: (a) ISFET chip, (b) encapsulation, (c) measuring cavity.

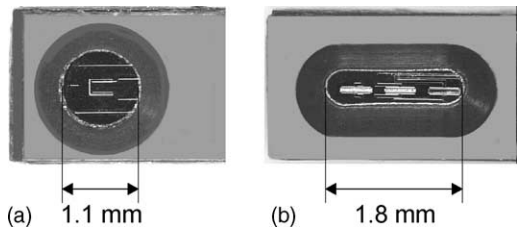

Fig. 5. Different shapes and dimensions of the measuring cavities in the encapsulation according to Fig. 3.

between the polymeric encapsulant and the surface of the sensor chip and the exact boundary lines of the cavity.

The stamp technology enables not only the manufacturing of circular but also of oval gate windows, illustrated in Fig. 5(b), which are necessary for encapsulating chips with more than one ISFET structure. The cavities, manufactured this way, have a depth of 300 μm. Due to their defined contours they can be used as miniaturised measuring cells for investigations in very small liquid volumes.

One example of such an application is shown in Fig. 6. For investigations of crevice corrosion cylindrical probes of different steels were ground obliquely at the front end and positioned in the oval cavity of the ISFET sensor, given in Fig. 5(b). The chip contains three identical ISFET pH structures. For the measurements ISFET 1 and ISFET 3 were used which are arranged on the chip at the distance 1.1 mm from each other; the width of the crevice between the steel probe and both ISFETs differs by 0.2 mm. The results, which have been published elsewhere in detail [25], show that the pH value of the electrolyte solution can differ within the crevice at the length of about 1 mm by several pH units. This example illustrates the outstanding possibilities to use ISFETs for the solution of uncommon measuring problems in many fields of research.

As compared with pH glass electrodes the advantages of ISFET sensors like their small dimensions, mechanical stability and insensitivity to overhead positions become particularly effective in medical applications. A survey of the possibilities of non-invasive pH measurements with ISFET sensors in the fields of ophthalmology, dermatology, gynaecology and dentistry is presented in [26–28]. Fig. 7

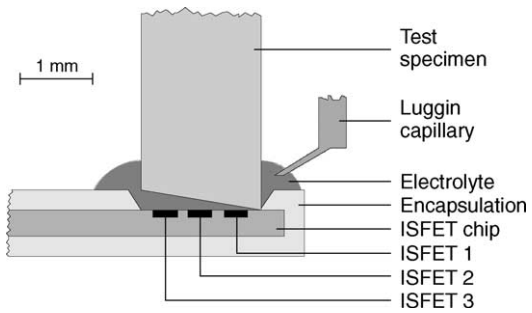

Fig. 6. Investigation of crevice corrosion by means of a multi-ISFET sensor chip with three uniform ISFET structures.

Fig. 7. Examples of ISFET sensors for special medical applications: (a) probe for measurements in dentistry, (b) probe for measurements in gynaecology.

shows two probes, developed for applications in dentistry and gynaecology. An ISFET pH sensor with the encapsulation illustrated in Fig. 3 is integrated in the dental probe. Measurements of local pH differences at various locations inside the oral cavity and of temporal pH changes in the saliva after rinsing the oral cavity with acid rinsing solutions can be performed easily with this ISFET probe.

The integration of an ISFET chip into a probe for the in vivo measurement of the pH value in the vaginal mucus is given in Fig. 8. Certain pathological changes like ascending infections or other complications in the vaginal environment are indicated by an increase of the pH value in the vagina, which amounts normally to pH = 4.5. It was shown in several publications, that ISFET sensors are more suited for these applications than any other measuring method [29–32].

2.1.2. *Encapsulation of tongue shaped ISFET sensor chips*

The encapsulation of needle or tongue shaped ISFET chips with insulated edges is comparatively simple and offers additional advantages. This technology, which was suggested in 1978 by Esashi and Matsuo [33], is based on the etching of needles or tongues out of the silicon wafer. The resulting silicon edges are insulated completely by a following thermal oxidation, guaranteeing a perfect insulation between the liquid to be measured and the electrical contact area of the sensor. By the elimination of leakage currents, the reliability and lifetime of sensors can be improved and the effort and costs of the encapsulation can be reduced sig-

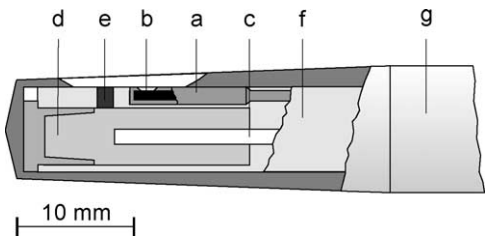

Fig. 8. Encapsulation of the ISFET chip in the sensor for in vivo measurements in gynaecology: (a) chip encapsulation, (b) ISFET chip, (c) Ag/AgCl reference electrode, (d) reference electrolyte, (e) ceramic diaphragm, (f) prefabricated moulded thermoplastic part, (g) sensor shaft (stainless steel).

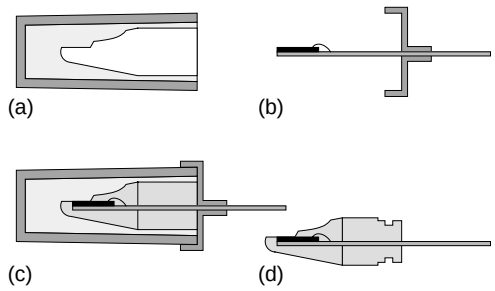

Fig. 9. Technology steps for encapsulation of tongue shaped ISFET chips in laboratory scale (KSI Meinsberg).

nificantly. On the other hand, the chip preparation requires much more expenditure.

A very simple encapsulation technology for such ISFET tongue chips with Si_3N_4 membrane, which had been prepared at the Centre for Micro Technologies of the Chemnitz University of Technology, is demonstrated in Fig. 9:

(a) Moulds are made from a thermoplastic elastomer (Thermolast, Gummiwerk Kraiburg, Germany).
(b) The chip is glued onto a circuit board and bonded. The board is fixed in the cover of the mould.
(c) The chip carrier is positioned in the mould, cast and hot-cured with epoxy at the conditions given in Fig. 2.
(d) Only minor finishing work is necessary after the mould release.

Fig. 10a shows a photograph of the sensor element. Primary purposes of the encapsulation of the tongue formed chips with wafer stage insulation are the protection of the bond connections and the mechanical stabilisation of the sensor. The chip surface in the vicinity of the gate structure is not covered. The decisive advantage of this encapsulation is the avoidance of the disturbing cavity of other designs as the surfaces of the sensor chip and the encapsulation

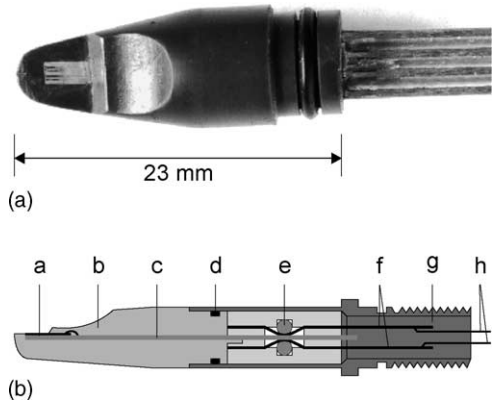

Fig. 10. (a) Photograph of an ISFET sensor element encapsulated according to Fig. 9 (KSI Meinsberg). (b) Inserting and contacting of the ISFET sensor element in the sensor shaft: a, tongue shaped ISFET chip; b, hot-curing epoxy encapsulation; c, printed circuit board; d, O-ring sealing; e, elastic contacting elements; f, flexible printed circuit boards; g, sensor shaft (stainless steel); h, connecting wires.

are at the same level. This results in shorter response times and simpler cleaning procedures of the sensitive surface. According to Fig 10b the completed ISFET sensor consists of such a sensor element and a sensor shaft. The O-ring seal guarantees a watertight plug in connection. Direct board connectors with gold plated contacts give electrical contact. A high flexibility of the sensor with regard to the design and measures of the shaft is achieved by this easily exchangeable sensor element [34] in which additionally temperature sensors or other components can be integrated. The outer diameter of the bar formed ISFET sensor amounts to 8 mm and is smaller than that of conventional pH glass electrodes ($d = 12$ mm). Further reduction of the diameter is possible but not needed for many applications.

The wafer stage insulation of the chip edges of the tongue formed ISFET opens up other possibilities for the encapsulation, which furthermore enhance the advantages of ISFET sensors as small dimensions and short response times. A probe with a sensor chip, covered with encapsulation material only in the area of the bond pads, is given in Fig. 11. The semiconductor chip contains two identical ISFET structures arranged parallel to each other at the distance 1.2 mm. This patented sensor was applied successfully for measurements of the current density distribution in galvanic baths [35]. The defined distance, the temperature equilibrium and the negligible pH differences between both structures with nearly identical response curves enabled measurements of the potential differences proportional to the current in the electrolyte with high precision. In [36] it is shown, that this probe can determine both the amplitude and the direction of the potential lines.

The outstanding suitability of ISFET sensors for measurements of fast changing local pH values or potentials, emphasised by Bergveld [37], can be used especially for measurements of pulse currents. In this case, the transient function has to be characterised by a leap of the current in the electrolyte and not by a change of the pH value. Measurements showed response times below 1 ms for this transient function. ISFET probes for potential measurements are therefore well suited for applications in galvanic cells with pulse current conditions using steps in the millisecond range.

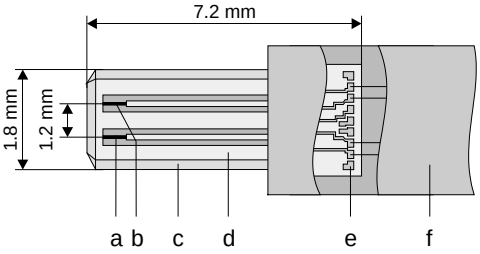

Fig. 11. ISFET sensor with a free standing tongue shaped ISFET chip: ((a) and (b)) two identical ISFET structures; (c) thermally oxidised chip edges; (d) ISFET chip; (e) bonding pads; (f) hot-curing epoxy encapsulation.

2.2. Industrial encapsulation technologies

2.2.1. ISFET sensors for laboratory and field applications

The requirements for encapsulation of laboratory-use ISFET sensors can be met more easily than requirements for industrial process applications, described in Section 2.2.2. This is due to the smaller temperature gradients, the narrower bands of application temperatures and pH values as well as smaller flow velocities and the limitation to aqueous media. Furthermore, the often discontinuous measurements in lab applications enable more frequent calibrations and require weaker standards of long-term stability and response behaviour in comparison to process applications. On the other hand these sensors can be marketed successfully only at lower prices.

Manual encapsulation with hot-cured epoxy has become a widely used technology for the production of ISFET sensors for lab and field applications. One example of the currently developed technologies, which is used for the ISFET sensor production at the company Sentron Europe BV, The Netherlands, is given in Fig. 12:

(a) The tip of the probe is equipped with a cavity for the ISFET chip, a channel for the bonding wires and the diaphragm for the reference electrode (not illustrated).
(b) The ISFET chip is glued into the cavity with epoxy glue after threading the bonding wires through the channel.
(c) Then the cavity is filled automatically with a defined amount of epoxy up to the upper level of the ISFET chip, which is equipped at the wafer stage with an adhesion layer.
(d) A hot-curing epoxy is applied manually during the second casting step. The shape of the encapsulation window is controlled by the amount of epoxy, the surface tension of the epoxy at the adhesion layer and the viscosity. The reproducible encapsulation finish by this complex step requires a particular skill.

One advantage of this technology is the prevention of mechanical damage by a mould compound or covering of the sensitive gate surface with epoxy. The oval encapsulation window in the cavity is about 1 mm long, 0.4 mm wide and 0.3 mm deep. The depth of the cavity and the contact angle of the epoxy at the chip surface influence the response behaviour of the sensor and the effectiveness of cleaning procedures. The outer shape of the encapsulation with a continuous curvature supports the formation of intensive flows inside the cavity in flow-through applications.

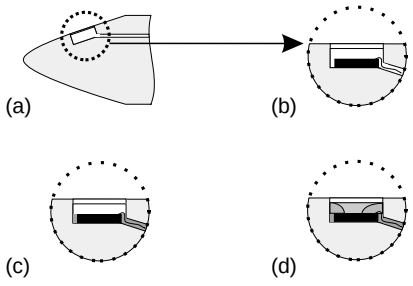

Fig. 12. Encapsulation steps of a Sentron ISFET probe for laboratory applications.

Fig. 13. Photograph of the tip of a Sentron ConeFET probe.

These Sentron ISFET probes are available in different probe tip designs for various applications. Fig. 13 shows a photograph of the tip of a so-called ConeFET, which is ideal for measuring pH in thick, dirty or sticky samples up to 105 °C. The diaphragm of the reference electrode is on the other side of the tip, just opposite to the ISFET chip. In the SurFET probe the ISFET pH sensor and reference junction are mounted on the distal end of the probe making direct surface measurements quick and easy. The pointed stainless steel tip of the LanceFET probe allows easy penetration in dense, very viscous samples and semi-solids like meats or cheeses.

2.2.2. ISFET sensors for industrial process applications

Nowadays, the most advantageous process applications of ISFET sensors are coming with the production of foods and cosmetics due to the glass free construction of these sensors. Some of these applications require frequent cleaning and sterilisation and are characterised by fast temperature changes, elevated pressures and high flow velocities. Sensors that are applicable with a long-term stability of several years have to complete a number of harsh test routines (see Section 3). The encapsulation of these sensors must assure package integrity even at elevated temperature gradients. Intensive research and development efforts of a number of ISFET suppliers led to the realisation, that this requirement cannot be met completely by epoxy on silicon but exclusively by tightening the chip with elastomer gaskets. This concept is used for example by the companies Endress+Hauser, Germany, Honeywell, USA, and Mettler Toledo, Switzerland, for the production of ISFET sensors for measurements in foods [38,39].

The cross section through the encapsulation of a sensor (Fig. 14), produced by Endress+Hauser, shows this elastomer gasket in form of a miniaturised quadring made of EPDM (ethylene propylene diene monomer), which is tightened by pressing a spring onto the reverse side of the chip. The front-side contacts of the chip are encapsulated by epoxy to improve the mechanical stability during the production process and the adhesion between the gasket, the chip and the sensor body at the non-process side of the gasket.

Fig. 15 shows the sensor tip with 12 mm diameter. Within the PEEK body and the EPDM seal, the cavity is formed

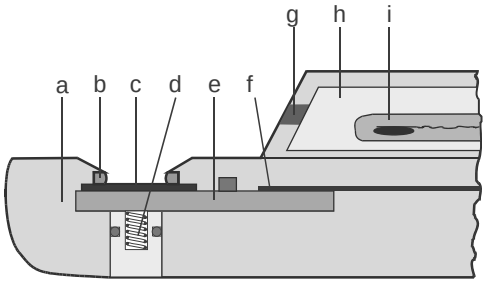

Fig. 14. Cross section through an Endress+Hauser ISFET probe for process applications: (a) sensor tip with PEEK body; (b) EPDM quadring seal; (c) ISFET chip; (d) reverse side pressure spring; (e) chip carrier; (f) connection cable; (g) ceramic diaphragm; (h) first reference electrolyte chamber filled with highly viscous gel; (i) second chamber filled with low viscous gel and Ag/AgCl-reference electrode.

with the depth of 1.4 mm and the bottom diameter of 1.8 mm. Made of PEEK (polyether ether ketone) this sensor meets the requirements of the US Food and Drug Administration (FDA) and is certified by the European Hygienic Engineering & Design Group (EHEDG) as one of the preconditions necessary for applications in foodstuffs.

The materials for the sensor body (PEEK) and the elastomer (EPDM or Kalrez®) are chemically resistive in a wide variety of media and provide the necessary stability in the required temperature range between −15 and +135 °C. According to the elevated corrosion rate of the sensitive Ta_2O_5 gate layer in media with pH > 12 at ϑ > 60 °C ISFET sensors cannot be used for instance during cleaning in-place (CIP) processes working with hot bases.

The design of the sensor, made by Honeywell, employs an open window in a moulded thermoplastic tube housing (Fig. 16), which is made of glass reinforced polyphenylene sulfide (PPS) for industrial use and polysulfone for sanitary food, dairy and beverage applications. Sanitary electrode materials and design are both FDA compliant and meet the requirements of the International Association of Milk, Food and Environmental Sanitarians, Inc., commonly known as 3-A. Within the sensor window, between the ISFET chip and the housing, an elastomer seal of EPDM or Viton® provides, similar to the E+H sensor, an effective seal between the ISFET chip and the measurement liquid by compression. A multi-layer conductive elastomer is used to connect source, drain and substrate of the back side contacted chip with

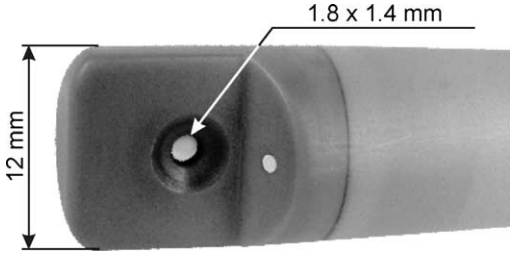

Fig. 15. Photograph of the tip of a Endress+Hauser ISFET probe for process applications.

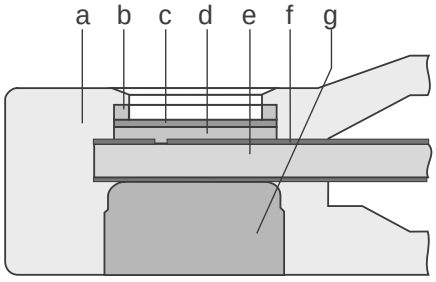

Fig. 16. Cross section through a Honeywell ISFET probe for process applications: (a) sensor tip with PPS body; (b) EPDM seal; (c) ISFET chip; (d) reverse side conductive seal; (e) printed wiring board; (f) connecting wires; (g) conductive plug (counter electrode).

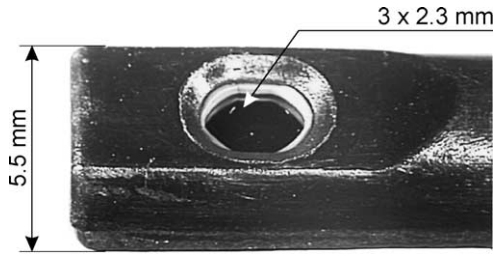

Fig. 17. Photograph of the tip of a Honeywell ISFET probe for process applications.

a printed wiring board (PWB). The design also employs a conductive plug at the back side of the tube, to control the ISFET drain current and voltage as explained elsewhere [40]. Electrical continuity to this electrode is developed through the physical contact with the underside of the PWB. The conductive plug is heat sealed into the tube. The sensor, shown in Fig. 17, has the tip diameter 5.5 mm.

In Fig. 18, the Mettler Toledo ISFET encapsulation is presented. Here the sealing of the sensor housing is made using ultrasonic welding. The design uses two EPDM rings on both sides of the ISFET. Like E+H, the ISFET has front-side contacts. By using PEEK parts of accurate dimensions, one can compress the ISFET between the elastomeric rings with a constant, definite force, thereby allowing highly reproducible packaging conditions for the ISFET. Also, in this version the ISFET orientation along the main sensor axis can be freely chosen, enhancing the cleanability of the sensor tip. In fact, this design has been approved by EHEDG

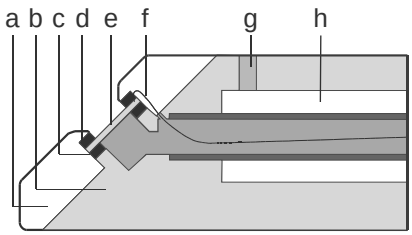

Fig. 18. Cross section through a Mettler Toledo ISFET probe for process applications: ((a) and (b)) PEEK body, ultrasonically welded; (c) back side EPDM seal; (d) front-side EPDM seal; (e) ISFET chip; (f) ISFET contacts; (g) ceramic diaphragm; (h) reference electrolyte.

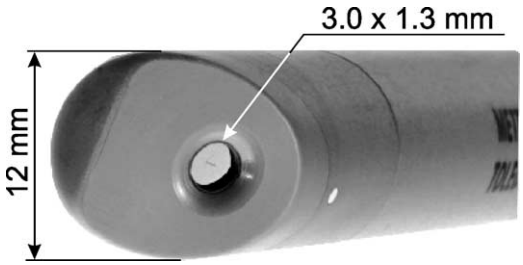

Fig. 19. Photograph of the tip of a Mettler Toledo ISFET probe for process applications.

for in-place cleanability. Fig. 19 shows a photograph of the tip and the dimensions of the circular measuring cavity of a Mettler Toledo ISFET probe for process applications.

One of the major differences between the designs of Honeywell and Endress+Hauser or Mettler Toledo is the die contacting method. Based on Baxter's work [23], in the Honeywell design the backside contacting method is employed, whereas in the Endress+Hauser and Mettler Toledo designs, front-side wire bonds make electrical contact.

All sensor designs have in common that the sensor chip is located at the bottom of a cavity in the encapsulation. Despite of this, measurements in low viscous media show very short response times of all these sensors. On the other hand, when measuring in highly viscous media the cavity can cause elevated response times as discussed in Section 4.2.

3. Properties and testing of the encapsulation materials

3.1. Main requirements to the encapsulation materials

It stands to reason that packaging materials for chemical sensors should be highly resistant to chemical attack. Good processibility, mechanical stability, low electrical conductivity, a suitable thermal expansion coefficient and biocompatibility are some general further demands, between which a compromise must be found. In the special case considered here, the adhesion behaviour on the sensor chip and moisture absorption are among the most important parameters of the encapsulation material. When being exposed to more or less corrosive measuring liquids, ISFET sensors may be damaged both by undermining the interface chip/encapsulation as well as by penetration of the encapsulation. Consequently, the most crucial requirements for good quality and long-term durability of the encapsulation are

- very strong and stable adhesion between the polymeric encapsulant and the surface of the sensor chip, and
- low penetration of moisture into and through the bulk of the encapsulant.

These two requirements overlay each other with nearly the same results, and it is not easy to experimentally distinguish which is the dominating one.

The first aspect was discussed extensively in [11]. As a result of the theoretical and practical studies, it was shown that a suitable coupling agent may improve the adhesion between the encapsulation material and the surface of the chip and therefore the packaging reliability. However, voids can be formed at the interface, which can cause a breakdown of the packaging. Another problem comes with the fact that the surface of the sensor chip is not of a uniform material, e.g. Si may be covered with layers from different materials like SiO_2, Si_3N_4, Ta_2O_5, Al_2O_3 or electrodes made from metals like Pt, Ag or Al.

3.2. Testing of the encapsulation of ISFETs for laboratory and field applications

With respect to the second aspect, at the Kurt-Schwabe Institute the penetration of water and a number of other liquids into different epoxy resins was determined by means of standardised gravimetric methods as well as with a more practical measuring method. This method starts with careful cleaning and drying of a sample from the material to be tested which is then inserted for 20 min into a buffer solution pH = 4.61. After that the sample is carefully cleaned and dried again and then inserted into a well-defined volume of a test solution with low buffer capacity (0.1 M KCl solution). Due to the release of the buffer solution, which had been absorbed in the test sample, the pH of the test solution is shifted to lower values. From this pH shift the absorption power of the respective encapsulation material may be estimated. A result of our measurements and some producer data are indicated in Table 1. Obviously, the water absorption increases considerably at higher temperatures. In numerous other liquids like 0.1 M HCl, 0.1 M NaOH, denaturised alcohol or formaldehyde, similar values of the absorption power have been observed. The stability of ISFETs in different commercial disinfectants has been tested, too. In [41] it was shown, that even exposure over several days in the disinfectants had no disturbing or damaging influence on the epoxy encapsulation and function of the ISFET sensor. On the other hand, dioxan, acetone or tetrahydrofuran attack and dissolute the epoxy resin encapsulation. In concentrated

Table 1
Water adsorption of epoxy resin according to ISO 62

	Duration of exposure	Temperature (°C)	Water adsorption (%)
Producer specification	10 days 30 min	23, 100	0.23, 0.16
Our own measurement	4 days	50	0.20

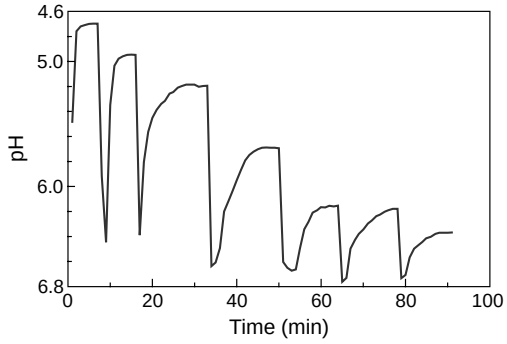

Fig. 20. Changes of the pH value of 0.001 M KCl solution in a small measuring cavity after calibration with buffer solution (pH = 4.6).

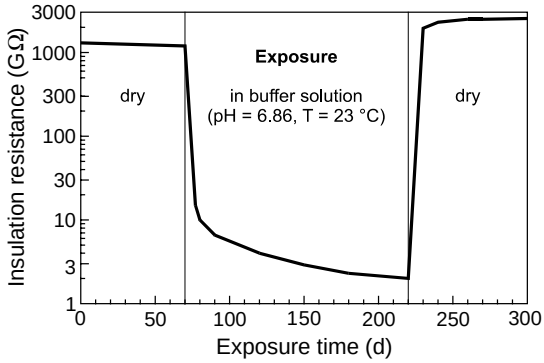

Fig. 21. Long-term changes of the resistance of the epoxy insulation when the ISFET sensor is exposed continuously to buffer solution (pH = 6.86, $T = 23\,^{\circ}C$).

HF epoxy resin is pretty stable, whereas highly concentrated NaOH, especially at elevated temperatures, severely attacks the resin. It should be noted that cold-curing epoxy is not suitable for reliable ISFET packaging due to the considerably higher penetration of water into this material.

In principle, due to their small dimensions, ISFET sensors are favourable for pH measurements in micro samples. However, at such applications the moisture absorption and desorption by the encapsulation material must be carefully taken into consideration. This effect is especially troublesome if the sensor, after having been calibrated in a buffer solution, is used for measuring pH values of very small amounts of solutions with low buffer capacity and pH values around the neutral range. Fig. 20 shows the result of a model experiment to demonstrate this source of error. At first the ISFET sensor according to Fig. 3 had been calibrated over a period of 15 min in a buffer solution pH = 4.61. After that minor amounts of the neutral, weakly buffered test solution (0.001 M KCl solution, pH = 6.8) were repeatedly filled into the small measuring cavity of the sensor (measuring volume 2 μl), and the changes of the pH value were measured with the ISFET sensor in each case. As can be seen, the pH of the test solution is tending continuously to lower values even after many measuring cycles as a result of the excretion of the buffer solution, which is still stored in the encapsulation, into the test solution. As a consequence, it is recommended to rinse the measuring vessel with special care if dealing with micro samples.

Also, at higher temperatures, the electrical resistance of dry hot-curing epoxy resin is very high ($>10^{15}\,\Omega$ cm at 20 °C, $>10^{13}\,\Omega$ cm at 100 °C), but it is reduced as a result of moisture absorption. Fig. 21 shows that the resistance of the ISFET encapsulation decreases during storing of the sensor in buffer solution (pH = 6.86) over a period of 5 months by three orders of magnitude. Nevertheless, the resistance remains in the GΩ range in which the normal function of the sensor is guaranteed. Essential is the fact that the resistance at dry storage after short time increases again to the original high value. This indicates that the decrease of the resistance is not caused by undermining but by absorption of moisture in the encapsulation.

A variety of methods can be applied for characterisation and testing of the ISFET encapsulation reliability [42,43]. Apart from resistance measurements according to Fig. 21 the quality and long-term durability of the encapsulation may be tested by means of measurements of electrode potentials or leakage currents, by capacitance–voltage (*C–V*) measurements, impedance measurements as well as measurements of the electrochemical noise. Furthermore, CO_2 and NH_3 response measurements have been proposed to show the existence of condensed water at the interface [11].

For leakage current or potential measurements e.g. an additional aluminium indicator electrode may be arranged on the ISFET chip around the sensitive area. The undermining of the encapsulation results in significant changes of the electrode potential or the leakage current, respectively, which can be measured between the test electrode and an external reference electrode. In normal potentiometric operation, by suitable interface circuit means, these electronics control the potential difference between the gate electrode and the source. The effective gate electrode may be the reference electrode or a chemically inert counter electrode [39,40]. Electrical leakage between the gate electrode and FET internals, including the source, drain and substrate, is an excellent measure of package integrity. During packaging designs' evaluation and for production qualification of ISFET products, Honeywell measures counter electrode leakage currents over operating temperature to assure package integrity. Electrochemical impedance measurements have been applied already for a long time for monitoring of ISFET encapsulation ageing [44]. Fig. 22 shows temporal changes of the impedance due to gradual undermining of improper encapsulation of an ISFET sensor (curves 1 and 2). As measuring electrodes the Al bonding pads on the ISFET chip were used. If chloride ions are added to the test solution, corrosion processes at the Al electrodes cause considerable potential fluctuations resulting in curve 3 in Fig. 22.

Besides these already established methods the measurement of the electrochemical noise offers an additional possibility for assessment of penetration or undermining of the encapsulation [45,46]. The beginning of local corro-

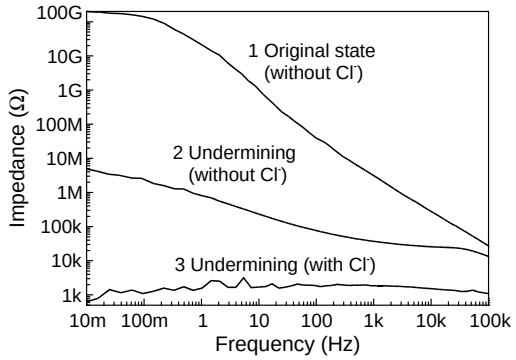

Fig. 22. Temporal changes of the impedance due to undermining of the encapsulation of the ISFET sensor, measured at an Al electrode on the ISFET chip. Curves 1 and 2: measurement in borate buffer solution (pH = 6.4); curve 3: measurement in borate buffer solution with 0.01 M HCl.

sion phenomena under coatings can be detected in situ at a very early stage by means of this highly sensitive method. Fig. 23 shows the result of an electrochemical noise experiment, which had been performed contemporaneously to the impedance measurements described above in Fig. 22. In the original state and even at undermining by the buffer solution the noise amplitudes are very low. Adding of chloride ions to the test solution causes corrosion of the Al electrodes, which is indicated immediately by heavy, irregular voltage fluctuations, which are typical for electrochemical noise.

3.3. Testing of ISFET sensors for process applications

The harsh conditions of a variety of process applications require additional tests before the sensor commercialisation. The thermal-shock resistance can be proofed by periodic, fast temperature changes between 5 and 95 °C. According to the quality standards of Endress+Hauser the sensor must remain stable during a minimum of 3000 temperature cycles with the duration of 5 min per cycle. Another test proofs the durability by keeping the sensor at 100 °C and 16 bar over several days and characterising sensitivity and base line periodically. A third test concerns the stability during steam sterilisation cycles at 145 °C over 30 min. This test indicates another advantage of the ISFET sensor compared with the pH glass electrode, which shows significantly higher changes of the sensitivity and base line during a single sterilisation cycle. This recommends the application of ISFET sensors for controlling of biotechnological processes with improved accuracy. It should be noted that the epoxy encapsulated ISFET sensors according to Fig. 3 indeed withstand temperatures above 100 °C under dry conditions, but they do not endure steam sterilisation procedures [47].

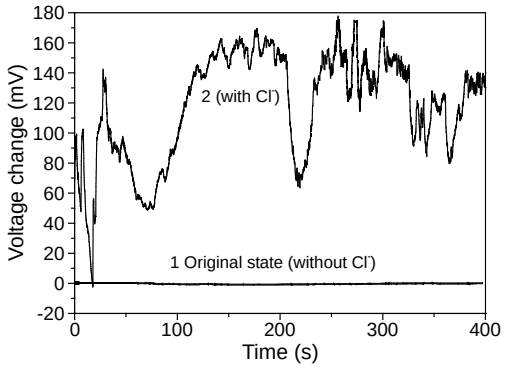

Fig. 23. Voltage noise, measured at an Al electrode on the ISFET chip under the epoxy encapsulation. Curve 1: measurement in borate buffer solution (pH = 6.4); curve 2: measurement in borate buffer solution with 0.01 M HCl.

Honeywell tests its ISFET sensors with the Durafet® II design extensively as operating pH sensors. Automated temperature cycling over 0–90 °C of powered ISFETs in pH buffers has been routine over the past 15 years. Each production ISFET is tested through two 0–80 °C temperature cycles before it is qualified for customer shipment. Testing is not only used for evaluation of package integrity and die performance, but also to allow for proper temperature compensation as explained elsewhere [48].

Mettler Toledo checks the quality of each ISFET encapsulation routinely by using firstly a Helium leak tester to detect badly ultrasonically welded sensor tips during manufacturing; and secondly it also tests extensively the sensor properties at the end of manufacturing such as drift and leakage current. With this procedure, Mettler Toledo can guarantee that the leakage currents of the delivered ISFET sensors are negligibly low.

3.4. ISFET-inspection under operating conditions

Sensor check systems are now commercially available for the E+H ISFET sensors. This feature allows the customer to inspect automatically the sensor operation during use. ISFET sensors are operated within a control loop. The control loop consists of the ISFET gate, ISFET chip, electronic circuitry, reference or solution ground connection and the analyte. Any leakage of the housing or damage of the gate will result in a significant increase of the leakage current, measured within this loop. Typical current values for a proper housing are on the order of picoamps and in the range of nano- to micro-amps for leaks in the housing.

4. Influence of the encapsulation on the dynamic response behaviour of ISFET sensors

4.1. Response behaviour in low viscous media

Apart from their small size and their inherent safety against breaking, the rapid response of ISFET sensors is considered to be among their chief advantages. Especially prepared ISFET sensors show response times $t_{(5–95\%)}$ of about 5 ms in high velocity fluid jets (velocity greater than 100 cm/s) in a special measuring set-up [49].

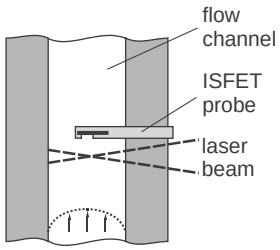

Fig. 24. Frontal impingement of an ISFET probe in a flow channel as part of a heatable flow circuit for measuring flow patterns in the cavity with laser Doppler velocimetry (two separate beams) and particle image velocimetry (one two-dimensional light sheet).

However, the flow rate in the bulk of the chemically sensitive gate surface of the ISFET is much lower in most practical applications using continuous flow cells. This is partly because the sensor itself disrupts the flow in its vicinity, but also because of the gate position at the bottom of a cavity in the encapsulation where the liquid exchange is partly obstructed.

To quantify this phenomenon the flow patterns in the cavity near the sensitive surface were studied by laser Doppler velocimetry (LDV) and particle image velocimetry (PIV) in different arrangements between the ISFET and the flow (Fig. 24).

The results of these optical investigations, given elsewhere [50], show that the liquid exchange is delayed significantly when the main flow direction is changed from frontal to reverse side impingement of the sensor. This tendency was also found for the response time of the ISFET sensor in Fig. 3, which was positioned in a flow-through channel. Frontal impingement of the channel flow onto the cavity leads to a response time of about 0.3 s after switching over between buffer solutions with pH = 6.86 and pH = 4.62 (Fig. 25) while reverse side impingement leads to significantly longer response times.

As compared to the short response time of the gate surface itself (see [49]) the encapsulation prolongs the response time of an ISFET sensor to an extend, where it is comparable

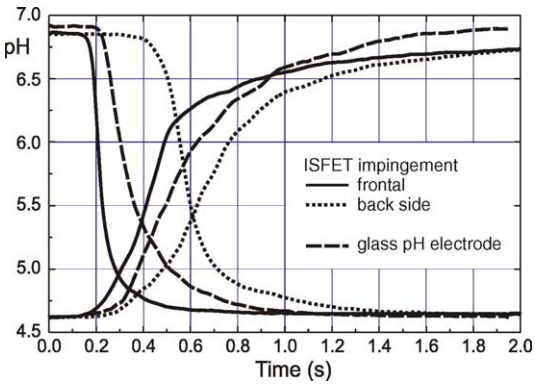

Fig. 25. Transient functions of the ISFET sensor in Fig. 3 at frontal and back side Impingement of the channel flow in comparison to a miniaturised glass pH electrode with a flat membrane (4 mm diameter).

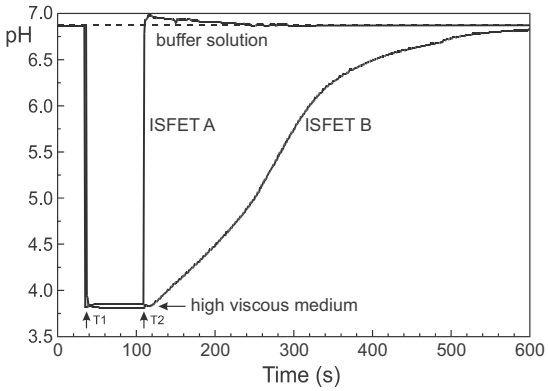

Fig. 26. Response behaviour of two ISFET sensors with different encapsulations in tomato ketchup, ISFET A see Fig. 10, ISFET B see Fig. 3.

with that of a miniaturised pH glass electrode, tested under the same conditions (dashed lines in Fig. 25).

4.2. Response behaviour in highly viscous media

Applications of ISFET sensors for the control of production processes of foods or cosmetics require online measurements with short response times also in highly viscous media. With increasing viscosity of the media to be measured, the exchange is obstructed more and more if the sensitive surface is positioned in a cavity. To quantify this effect, two ISFET sensors with different encapsulations were tested in tomato ketchup [51]. Fig. 26 shows the results. The sensors were rinsed with buffer solution (pH = 6.86) and dipped into the ketchup at time T1. At time T2 they were rinsed and cleaned with the same intensity and stored in the buffer solution again. The signal of the sensor A (see Fig. 9) with the sensitive gate area at the encapsulation level indicates the pH value of the buffer solution immediately after T2, while sensor B with the cavity (see Fig. 3) shows an elevated time delay.

The short response time of ISFET sensors in low viscous media is essential for the installation of fast control circuits. To ensure short response times also in highly viscous media, important for a variety of food and cosmetic production processes, the sensitive gate surface has to be positioned at the same level as the surrounding encapsulation.

5. Conclusions

So far ISFET sensors have been manufactured in the laboratory scale or in short run manufacturing. The encapsulation of the sensor chips was made predominantly by embedding in epoxy resin, whereby the shape of the sensor was adapted to the intended application to a large extent. Their specific advantages—small dimensions, planar surface, mechanical stability, and short response times—could be used for the solution of special measuring problems, for which glass electrodes are less suitable.

The recently achieved improvements of long-term stability and lifetime of the functional layer of the ISFET by using Ta_2O_5 or Al_2O_3 and advanced semiconductor processing techniques made them increasingly more attractive for industrial production and application as an alternative to conventional pH glass electrodes. Consequently, advanced packaging technologies have been developed, allowing economical mass production of ISFET sensors. By carefully choosing packaging materials, which meet the stringent requirements of the 3-A, EHEDG and FDA standards, an important precondition for widespread application in foodstuff, pharmaceutical and cosmetics industries, biotechnology and process control is fulfilled.

Summarising, it can be ascertained that significant progress in design and encapsulation technology and gate surface advances have resulted in the successful introduction of ISFET sensors in some important applications, where the classic pH glass electrode cannot be used at all or with function or survival disadvantages. Modern commercially available ISFET sensors are designed for applications at home, in the laboratory, in the field and also for the control of industrial processes where they are used successfully because of their unbreakable glass free design, their fast response and their outstanding stability during steam sterilisation.

Looking at the outstanding developments in encapsulation technology made during the last decade, Prof. Bergveld's question "Why has it not been discovered earlier?" invites all to accept the challenge of innovation.

Acknowledgements

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