Thermoelectric hydrogen gas sensor

— Technology to secure safety in hydrogen usage and international standardization of hydrogen gas sensor —

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A thermoelectric hydrogen gas sensor developed for leak detection in hydrogen stations has shown good hydrogen selectivity and wide hydrogen detection concentration ranging from 0.5 ppm to 5 %. We have demonstrated high sensitivity and reliability of the sensor exceeding conventional technology through a field test of one year. We could optimize the following three constituent technology elements to meet the social needs, i.e. new principle integrating catalytic combustion and thermoelectric conversion technology, micro-fabrication technology to realize the principle and high performance ceramic catalyst for gas combustion. In addition, the sensor performance evaluation technology established during the development has been proposed for ISO standardization.

Keywords : hydrogen sensor, hydrogen station, field test, thermoelectric, catalyst combustor

1 Introduction

It was only 10 years ago in 2000 that we began the research and development of a new hydrogen sensor of thermoelectric principle presented in this paper. With the reorganization of the research environment into the National Institute of Advanced Industrial Science and Technology (AIST), we, the researchers working on material science and engineering explored the future of new materials research. The R&D level of leading industrial manufacturers or private sectors was already high, especially on devices, and also the process facilities and analysis equipment were highly advanced. We have tried to find the answer to differentiate ourselves from the basic studies of the materials in the university laboratories and we attempted to use new functional materials in the production of sensor elements or devices.

At the same time, a hydrogen-energy technology boom occurred, led by hydrogen fuel cells. For the goal of establishing a better leak detection sensor technology, which is the most important for the safe use of hydrogen, a novel sensor of a new working principle was developed in our laboratory. It is a “fusion of knowledge” of catalytic combustion gas using a catalyst which is the operating principle of a flammable gas, and a thermoelectric material being researched at that time. With a new principle of thermoelectric materials converting the local temperature changes caused by internal heating from burning gas to voltage, we jumped into the field of hydrogen sensors where so much had been developed up to that point.

Not limited to hydrogen sensors, a variety of performances is required with gas sensors. First of all, there are the detected concentration range, detection limit concentration, and performance such as selective gas detection. Most of the reports at related academic societies are concerned with materials research and device research and present arguments based on these detection performances. However, for sensors to be used in a real society, economy and reliability are required in addition to the detection performances. In particular, reliability is a subject that is most severely questioned when applied to a real sensor system.

We have developed a novel hydrogen sensor by continuously providing solutions to these challenges, the results showing the best performance in hydrogen leak detection applications. In terms of social receptivity of actual application of this sensor, we also have proposed international standards for hydrogen detectors.

2 Social demands for performance of hydrogen sensors

To ensure clean, safe to use hydrogen without the danger of its explosion, safety warranty technology is essential. At hydrogen-related facilities that require a high level of safety management such as hydrogen stations, hydrogen sensors are required to monitor changes in the concentration of hydrogen leaks continuously. In the hydrogen stations, large quantities of hydrogen are stored and frequently transferred to hydrogen cars, and the new technology of safe management of the stations is a challenge for a clean energy society. It is time to review the weaknesses of the current technology of safety sensors described below.

Most of the commercially available hydrogen sensors are

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semiconductor types or catalytic combustion types. In principle these sensors detect only a narrow range of concentration of gas, and hydrogen sensor technology of selective gas detection has not yet been achieved. The semiconductor sensors have been most extensively studied up to date because of their ultra-sensitive gas detection performance, and the majority of papers on gas sensors contribute to this technology. When improving the sensitivity of semiconductor sensors, the reliability problems always occur because the sensor is also very sensitive to the environmental noise, such as humidity. In addition, the accuracy of the sensor signals has another problem of signal drift (variation of the indication) [1], showing lack of reliability.

Catalytic combustion type sensors are widely used because they are robust with excellent stability. With this type of sensor, a slight rise in sensor temperature due to catalytic combustion of hydrogen gas increases the resistance of the platinum-coil heater, and this enables the detection of the hydrogen in the atmosphere. Catalytic combustion is a simple chemical reaction, and wrong signal outputs even in the presence of some environmental noise are extremely few. However, to achieve both reliability and sensitivity is difficult. The sensitivity of the detection signals of the catalytic combustion sensors which are the resistance rates of the sensor is significantly reduced in low concentration, and the practical range of concentration is in the detection range of a few percent from 1000 ppm[2].

Figure 1 shows the concept of a multi-level security system required in the hydrogen stations. In hydrogen-related facilities, if a system is established which can detect hydrogen concentrations before a specified concentration of hydrogen or fraction of flammable limit is reached, it will allow for single and/or multilevel safety operations, such as nitrogen purging or ventilation and/or ways to avoid shut-downs. Furthermore, in the stations, the system should have a sensor element which is robust against poisoning, which is caused by any interferential gas that permanently affects the sensitivity of a sensor, and is hydrogen selective against other flammable gases. In actual hydrogen stations, catalytic combustion type sensors are used for high gas concentrations as an alarm sensor, and semiconductor type sensors are also used for low concentrations as a monitoring sensor, respectively.

3 Elemental technologies for the gas sensor synthesis

3.1 Three elemental technologies for the three performances of the gas sensor

We have proposed a completely new principle of hydrogen gas sensor combining the catalytic combustion and thermoelectric conversion technology. This sensor uses the heat generated by combustion of gases within the catalyst in the same manner as conventional catalytic combustion type sensors. Rather than using a resistance change in temperature of the entire device, the changes in the local temperature inside the device are converted directly into voltage signals based on a thermoelectric conversion principle. We named this the thermoelectric sensor. Figure 2 shows three performances required for gas sensors or the 3S, correlating them with the elemental technologies: gas selectivity, high sensitivity, long-term stability.

1) new principle, of “integration of knowledge” - The thermoelectric conversion principle is the most fundamental idea which enables the wide-range selective gas detection, which is not possible with conventional technology[3].
2) microfabrication technology - For minimization of the heat capacity of the sensing element to catch a very small amount of heat for the detection of lower gas concentration, microfabrication techniques such as anisotropic etching of silicon was used. The thermoelectric sensor is a micro calorimeter with a catalyst combustor.
3) catalyst technology - Mounting a high-performance ceramic catalyst combustor on a specific position of the pattern on the micro thermoelectric devices has been accomplished.

By integrating these elemental technologies, a prototype of excellent hydrogen selectivity, of wide range detection up to 5 % hydrogen concentration from 0.5 ppm was realized and a year long stability field test proved that the same stability was realized as the current technology[4]. Typical characteristic of a high-sensitivity sensor is a trade-off between selectivity and stability, but thermoelectric gas sensor is able to satisfy both performances. Considering that the lower limit of catalytic combustion type sensor is about 500 ppm, the ppm-level detection performance of thermoelectric sensor is innovative. For the stability of the sensor, we have clarified with a scientific approach the factors causing degradation in the sensor response, and improved the stability of the sensor

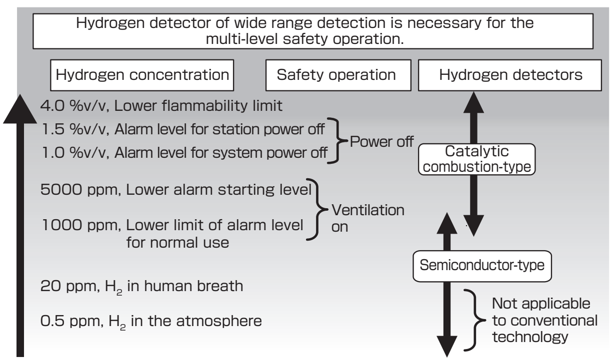

Fig. 1 Multi-level or multiple safety system for the operation of hydrogen facilities
Current safety technology of hydrogen leak detection lacks lower level stability and hydrogen selectivity.

by controlling the composition and thickness of the catalyst[5].

In the case of new born sensors, it is generally difficult to be accepted as a reliable technology for practical use. In order to demonstrate the reliability of the sensor, our thermoelectric sensors have been installed and tested for a year at Ariake hydrogen station in Tokyo. This field tests has validated the stable sensitive system-level safety operation, showing response to 100 ppm hydrogen in air[6].

3.2 The other elemental technology:technology of evaluation

Assembling each elemental technology, a sensor device is created. Then the method analyzing or checking the "workmanship" of the final product of the device also becomes an important issue. The device performance may satisfy the manufacturer, but it should be evaluated or tested for characteristics which are determined by social demands, customer needs or specifications. In this study, the following three testing methods were utilized as shown in Fig. 3:

・new test method to observe the surface temperature changes on the sensor device in the sensor operation using IR camera,
・standard test methods for the response speed (Appendix ISO CD26142),
・system technology to test the gas sensing performance for serial products.

As noted above, the thermoelectric gas sensor consists of two components of the ceramic catalyst and the thermoelectric device. The first test method is to evaluate the two different elemental technologies of the sensor separately to get a clear balance between the individual components. The second test method is a simple test method not using any special or complicated equipment so that anyone may evaluate the performance of sensor response in the same way. It is as simple as just filling the gas to be detected into a chamber with a capacity of 30 liters. This method is adopted as an appendix in the international standards issued in the year 2010 (ISO) as an evaluation method for the testing of sensor response time.

The third one is the test method for mass production. How much is the manufacturing cost of every single gas sensor element? Fabrication of small sensor elements costs little but testing of sensors costs much. The testing equipment for the materials properties is commercially available but the evaluation device for a “product” such as sensors is not commercially available. Sometimes the testing machine of the products is the most important secret or know-how for the manufacturers. So we started from an early stage of research and development efforts to develop a high-efficiency testing system as a top priority of mass production technology of the device.

3.3 The first step is to set a scenario

The integration of the elemental technologies mentioned so far is part of how the research progressed, aiming at solving various technical problems in order to meet the social demands. Figure 4 shows a chronological scenario of the thermoelectric hydrogen sensor development. We received a three-year research fund for the development from fiscal year (FY) 2000, and the achievement was the “knowledge” of fusing catalytic combustion and thermoelectric conversion as an elemental technology. The idea was very fragile, and as soon as we started, we found that even if the sensor was based on an excellent principle, to bring about satisfactory

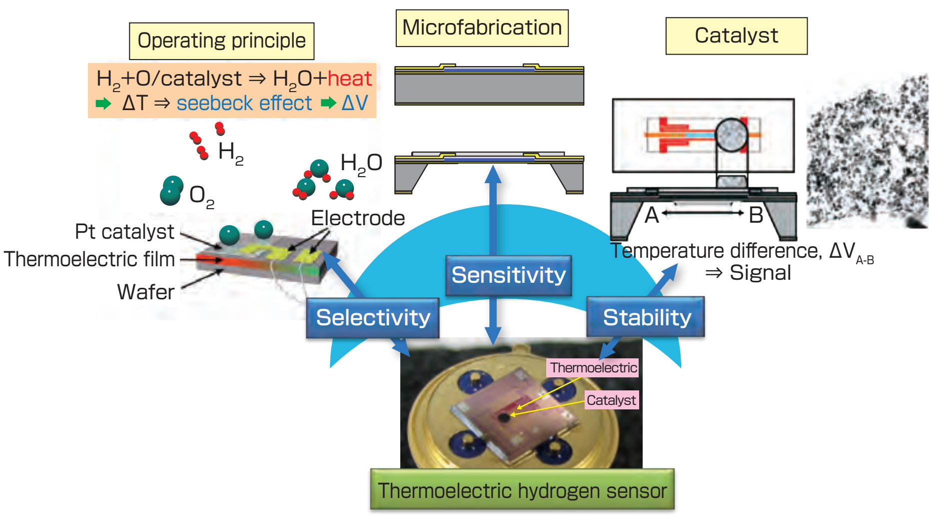

Fig. 2 Correlation between the sensor performances and elemental technologies
Three performance requirements, selectivity, sensitivity, and stability are of trade-off relations. Novel working principle of thermoelectric conversion of local temperature differences in sensor device induced by catalytic combustion can bring about the selective, stable, and sensitive detection.

performance, microfabrication was essential.

A five-year scenario of microfabrication of the sensor was drawn in 2002. One study cycle consisted of fiscal years of 2003-07. By integrating the elemental components, the necessary sensor performances were achieved, and as a result, we could harvest the output of research, such as papers, patents, transfer of technology one after another, and start a new cycle. The cycle of the development can be divided into four steps as follows, among which the third is the synthesis:

1) idea - to discover new ideas or to search social demands,
2) integration of knowledge - to quantify it in such experiments that embody it,
3) synthesis - to set the necessary properties (goals), to further development,
4) completion - summarize research results leading to the following research.

It takes a long time to organize the different elemental technologies. Based on a new idea for sensor performance improvement, new batch fabrication is processed, and then the performance of the manufactured sensor device is investigated, in relation to the improvement of the idea. By searching for new methods of development from the results obtained in this process, the leader of the research team could direct engineers who were responsible for each elemental technology to new directions. This feedback sometimes took a few months or one year. It is very similar to the practice of an orchestra. The most highly agile methodology practices in the development of thermoelectric sensors are the following two:

・laboratory design aimed at real application from the beginning (the full-automated process equipment and sensor testing systems)
・sharing of the whole development scenario and discussions within the laboratory with all the engineers.

In the process of the integration of knowledge, it is important to speed up the feedback of the fabrication and of the results of sensor tests. Our strategy was the improvement of both the process and analysis tools with a compact lab design. Though all the details of tools or lab layout cannot be described in this paper, we can explain a unique idea of our laboratory for manufacturing micro-sensor devices. We have succeeded in minimizing the clean room space very efficiently, and to integrate most of the process equipment within 5 meters radius. The sensor test was carried out next door. This high quick fabrication and tests realized the commercialization of the sensor after the 5 years research period.

Introducing full-automatic process equipment has brought good results in this research. Typically, especially at an early research stage, people tend to introduce an experimental facility which is designed specially for scientific researches. We, however, were able to develop a practical-looking production system by introducing a semi-generic one. Such equipment does not require a high professional staff with knowledge and is easy to maintain, and this led to a significant reduction in development costs.

The people who put out the results by running the facility are the members of the team. It is important to position the members in the right places, supplying an environment not

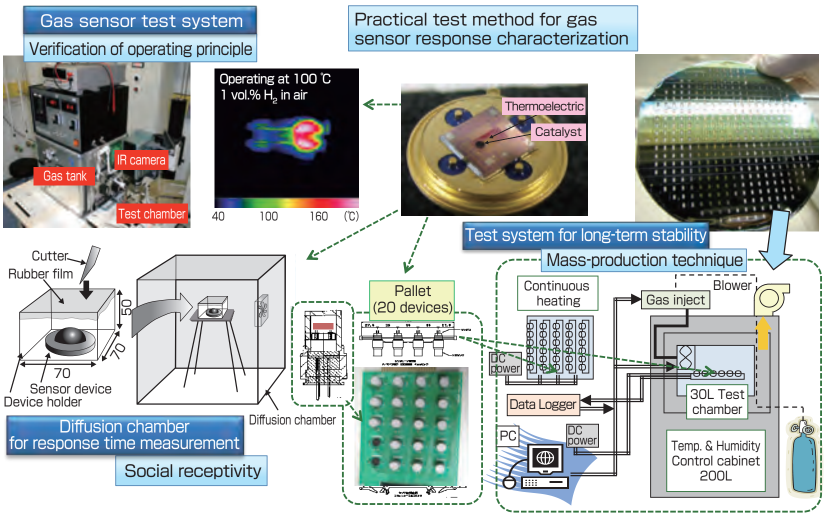

Fig. 3 Three test methods for sensor element
They are developed for checking the working principle, social receptivity and mass production, respectively.

bound by their expertise, encouraging the members to propel their corresponding special jobs. To direct them to a single goal keeping every core technology of each member is not easy, but it is possible to align their vector if the scenario is shared. In developing thermoelectric sensors, with all the members understanding the technological domains of others while holding firmly their own technological specialties, the organic collaboration between the members was achieved.

There is a concept called agile type often used in software development. As the waterfall type development is considered to be difficult to have communication among the staff, the agile type is currently widely used from the point of emphasizing communication among the members. Our research progress was close to the agile type but was not the same. A negative aspect of the agile type is the risk that the progress becomes slow dragged by too much discussion among members. We, however, built up the scenario at an early stage, and the whole image of development could be shared by the members.

For example, the supply and installation of the equipment, process and test facilities for the overall research period were carried out following the plan written in the proposal at the start. The leader of the project continuously showed the clear vision of the results after five years and also the five-year scenario to all the members. His work is similar to an orchestra conductor. The essential part is to share all the possible information and visions together.

4 How to make synthesis

4.1 Elements and boundary conditions

A simple combination of elemental technologies cannot make synthesis. Figure 5 shows the schematic plan of synthetic integration of each element. Natural phenomena and engineering components are listed on the left, and the observations and analysis are related tools to quantify their characteristics. It is important to bring out the full features of each element, resulting in performance which has an engineering or scientific value. When one integrates them into a new synthesis, specific and detailed boundary conditions are necessary. The boundary conditions are the various social demands.

For example, if you configure a catalyst and thermoelectric material for the applications to a generator and to a gas sensor, the conditions of configuration are largely different. For synthesis of the generator, it is important to obtain large electric voltage and current by high-calorie combustion. A catalytic material which can endure high temperature combustion needs to be selected and the device needs a complex flow channel for efficient fuel combustion. A large thermal capacity of the whole system is desired. Therefore, it is necessary to design a thermoelectric device that allows large electric current flow.

For the synthesis of a gas sensor detecting a wide range of gas from a few ppm to a few percent, ensuring reliable, linear sensor output, extremely high catalytic activity of the combustor is required which enables combustion even with low concentration gas. In order to detect slight temperature changes, a thermoelectric device which generates high voltage is required with a sensor structure of easy fast gas diffusion and sensor elements of minimized heat capacity for fast response.

In our sensor development, we configured and integrated the elemental technologies with the boundary conditions based on social demands as illustrated in Fig. 5. The conditions on the right such as high sensitivity, stability,

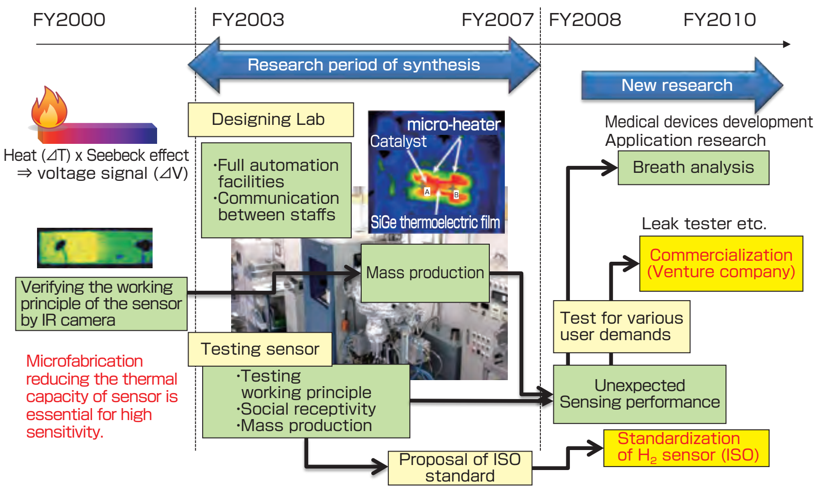

Fig. 4 Scenario of thermoelectric hydrogen sensor development
In FY2000, the operation of the new sensor was started with the NEDO grant, and the full development of hydrogen sensor technology was carried out under the national project of developing hydrogen infrastructure, FY2003-07.

and fast response could be determined by specific numeric target values requested from the user's needs. A prototype sensor is fabricated and its performance is evaluated, verifying the achievement against the targets. During this process, the elemental components are selected and integrated for the synthesis.

When we select the elemental technology by considering the boundary conditions, the knowledge of the past is sometimes insufficient, and it is necessary to confirm and to recognize the details by fabricating and testing the actual prototype device. At the start of the development of the sensors, we had a catalyst problem. The composition of the catalyst combustor followed the previously reported data in the papers, which said that a few nanometer thick catalyst film was selected for higher activity. However, after trial and error, thick film catalyst of much higher platinum content was found to be better by our experiment. This thick film catalyst made hydrogen combustion possible at room temperature[7].

The researchers in the field of catalyst science have wondered and were surprised by the very high amount of 20 ~ 40 wt% of noble metal, platinum, in the catalysts of our sensor devices. This was because their common knowledge of the catalyst was that several wt% of metal content was better to prohibit unwanted sintering of the metal during the operation. However, the catalyst of our design was found to exhibit a high activity and stability to detect the hydrogen gas in air[8]. Unlike the typical catalytic reactions, the catalyst for the sensors is required to be able to burn gas at extremely low concentrations of flammable gases, so that thick film type of high metal contents was integrated into the sensor. As a result, boundary conditions of gas sensor application stimulated the elemental technology of the catalyst to be something of a totally different quality.

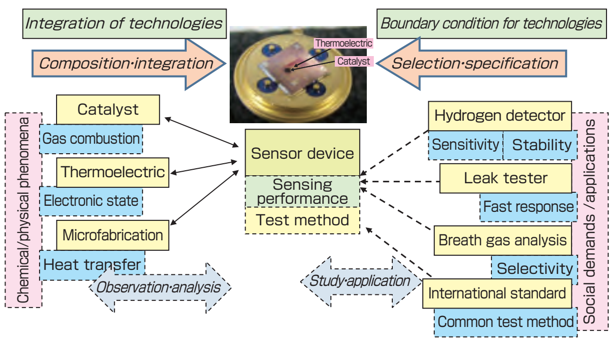

Fig. 5 In the synthesis of thermoelectric gas sensor, the elemental technologies have been configured and integrated with the boundary conditions of social demands.

4.2 Scenario for commercialization

After investment for research such as capital, facilities, and human resources, papers and patents are always desired as results of research. Of course commercialization of the new technology is an ultimate goal to complete the R&D. Especially for AIST, a research institute without any commercial product vending department, to complete R&D is to transfer technology obtained in the development to private companies as intellectual property such as in the form of patents.

In mass production technology of our prototype, it was easy to scale up the fabrication of sensors, as a result of the strategy described above. It was necessary to make technology transfer possible even with some companies that do not have their own semiconductor process facilities. We performed 4 inches or 6 inches wafer processes not only in the laboratory prototype manufacturing but also in a commercial foundry. In 2007, the foundry services in Japan still had little business experience, including the issue of acceptance of service and other contractual issues of technical problems, and it was very difficult to work out our first batch. The experience of the difficulty of the foundry process, and confirming the process yield was a big step toward commercialization. Fortunately, the Seebeck coefficient of the SiGe thermoelectric film pattern in our device which is the most important parameter of the sensor, was relatively process independent. Therefore, we established a manufacturing technology sensor device with reduced discrepancies in sensor performance.

4.3 Results over the scenario

Unexpected sensor performance triggered a start of new development which was not drawn in the scenario. A leak detector is one example in our research. The very promising thermoelectric hydrogen sensor capability detecting the gas leak of several-ppm level was over-specification in the application of safety sensors. However, this performance is necessary in new industrial application of leak detection, which is the technology to check the air tightness or sealing of the products, such as in fuel tanks, battery packs, water proof housings, etc. In this gas leakage test, currently helium gas is used, but hydrogen is now also used in place of helium because of recent instability of He import from the US. Recent testing equipment with 95 % nitrogen 5 % hydrogen gas as a gas alternative is now widely used, and with the high cost of helium, there is a strong need for cheap, sensitive, and selective hydrogen sensors.

Another development is a medical device to support human life, which aims to expand the application of the sensor which can measure hydrogen concentration in the breath[9]. Current technology used in medical centers is expensive, costs around 3 million yen, and it combines a semiconductor type gas sensor and a gas chromatograph. The thermoelectric sensor can detect ppm level hydrogen concentration, and can be a simple alternative solution, which does not need gas chromatograph, reducing the size, cost, and analysis time significantly. Currently, a prototype has been commercialized as an AIST venture, and is expected to be applied to various fields as well as to hydrogen-energy facilities such as hydrogen stations.

5 International standard making use of research output

The state of the market of hydrogen sensor strongly correlated to the spread of the hydrogen energy use and the social receptivity of hydrogen are also important issues for commercialization. However, the situation of the current market is inactive and conservative. The sensors of the old technology are already adopted in the present hydrogen stations operated in Japan, and the new technology cannot be accepted easily. Even though there is new demand for a wide range of hydrogen detection, the thermoelectric hydrogen sensor could just end up as a development study. To promote the application of our new sensor technology, we also carried out a study for the standardization of the gas sensor, making a new proposal for hydrogen sensors. The standardization work has been proceeded in parallel with the technology development of the thermoelectric sensor, propelled by the policy of the Ministry (METI).

We prepared a proposal based on the performance of our newly developed "thermoelectric hydrogen sensor with a wide range of hydrogen concentrations with hydrogen selectivity", and submitted it as a new proposal (NWIP) to the committee of ISO/TC197 (Hydrogen technologies) in 2005. This proposal was accepted and WG13 (Hydrogen Detectors, host country is Japan) started to discuss a new international

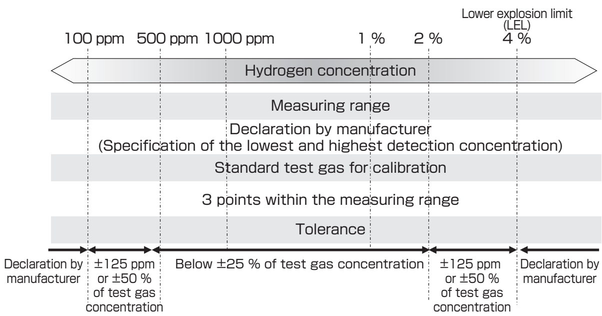

Fig. 6 Measuring range and calibration
It was extremely difficult to write down the measuring range of the hydrogen detectors to satisfy all the members in the ISO working group for hydrogen detectors. This figure shows the meeting material suggested and used in the final stage of the committee meeting, showing a range from 500 ppm to 2 %, which was the result of the previous meeting. This was finally rejected and the measuring range of the detector was decided to be declared by each sensor manufacturer.

standard of hydrogen detectors. This international standard was issued in June 2010. This was one example of proposed international standards development efforts utilizing the technology, and the best detection technology that satisfied many of the contents of the proposed standard was the thermoelectric hydrogen sensor developed by us. However, the draft did not pass as it was, and the final draft was edited and changed significantly from the 2005 draft. This International Standard set the performance requirements for hydrogen detectors as follows:

・ measuring range, concentration calibration and alarm set points,
・stability (short and long term),
・time of response and recovery, selectivity, poisoning,
・temperature, pressure, humidity (standard test conditions),
・ operation above the measuring range, power supply variation and interruptions.

The most discussed issue was the measuring range of the detector as shown in Fig. 6. In the related IEC standard on the inflammable gas detectors, only the upper limit of gas concentration is specified and this is declared to be the range. However, the tolerance required in the calibration is defined as 5 % of the upper limit or 10 % of the indication, and the error becomes seriously large in the low gas concentration range.

We have explained the importance of low-concentration detection for the multi-level safety operation, and proposed the standard of detectors covering a wide range of hydrogen concentration. This claim of our Japanese delegate was accepted and a committee draft containing the measuring range from 500 ppm to 2 % as shown in Fig. 6 was drawn. However, several countries did not agree to this, because their technology works well at high gas concentration. In the end, as the international standard is not intended to exclude any specific technologies that meet the performance requirements, the measuring range of the detector including the tolerance of the detection was decided to be declared by each sensor manufacturer.

6 Summary

We have invented a novel thermoelectric gas sensor, integrating the elemental technologies of catalytic combustion of hydrogen and thermoelectric conversion from thermal gradient to voltage, for gas-leak detection systems in hydrogen stations. By integrating various elemental technologies, completely new performances such as robustness, hydrogen-selectivenes, wide-range hydrogen detection from 0.5 ppm to 5 % in air, fast and linear response are realized, which are impossible by the current sensor technology. Furthermore, taking advantage of this new technology, we have proposed and published a new ISO international standard in 2010.

We are carrying out several application researches of the thermoelectric hydrogen sensors for various practical uses, expecting future spread of the related technologies and international cooperation including hydrogen energy use.

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Received his BS and MS degrees in polymer chemistry from Osaka University in 1985 and 1987, respectively, and his doctorate of science in inorganic and physical chemistry from Osaka University in 1994. He is the director of Research Planning Office for Nanotechnology, Materials and Manufacturing, AIST, and the convener of the ISO/TC197/WG13 (hydrogen detection apparatus) and TC146/WG16 (Test method of VOC detector). His research interests include functional oxide materials, organic-inorganic hybrid materials, and gas sensors. In this paper he discussed the part of international standard working group for hydrogen detectors.

Discussions with Reviewers

1 Investigating social demands

Question (Norimitsu Murayama, Advanced Manufacturing Research Institute, AIST)

This report points out the importance of catching the clear social demands in the beginning of the research. What is your opinion on the method of grasping the social demands?

Answer (Woosuck Shin)

We first collected information from the domestic and oversea R&D reports and technology road maps, our team discussed the future social demands using the obtained information, and we finally made an analytical evaluation of the social demands we found.

2 Selection of elemental technologies.

Question (Hisao Ichijo, Tsukuba Center, Inc.)

The assembling and integration of the elemental technologies are clearly written in this paper. How do you make a choice?

Answer (Woosuck Shin)

Though it is simple and clear to choose each elemental technology from the boundary condition of the social demands, to select in detail and the way of integration is rather complicated. By fabricating and testing the sensors, we carried out the integration.

3 Originality of the research

Question (Norimitsu Murayama)

The originality of this research is the combination of catalytic combustion and thermoelectric conversion. How did you come about this idea?

Answer (Woosuck Shin)

Before the sensor research, I researched papers to take advantage of the thermoelectric research we had developed for the sensor application. I found a paper of 1985 reporting a bulk oxide gas sensor with Pt electrode on one side. Both the materials and designs of the sensors of the paper are developed and advanced in this research.

4 Research of commercialization

Question (Norimitsu Murayama)

In this research, mass production facilities are equipped from the beginning, which made easy commercialization. I wonder if this method can be applied to other researches or if it is more beneficial to specific research types.

Answer (Woosuck Shin)

The materials research is a fundamental issue and the research of the catalyst material in this research was also carried out in the usual chemistry laboratory. The most important role of the process and test facility foreseeing mass production is to reduce the barrier to real application. I think these mass production facilities could be applied to most research and will become more and more important in speeding up the research and development.

5 Publicity activities

Question (Norimitsu Murayama)

At first the application of the sensor was for the hydrogen stations, but it was changed and expanded to others such as leakage detection of air-tight tests and breath gas tests. To explore these unexpected applications, publicity activities need to be performed effectively. Which activity worked well in your case?

Answer (Woosuck Shin)

The press release and the material transfer were very effective to draw attention of society, and to lead to new applications.